

# Modification of Screen Printed Carbon Electrode-Gold Nanoparticles (SPCE-AuNPs) for Mercury Detection in Cosmetics

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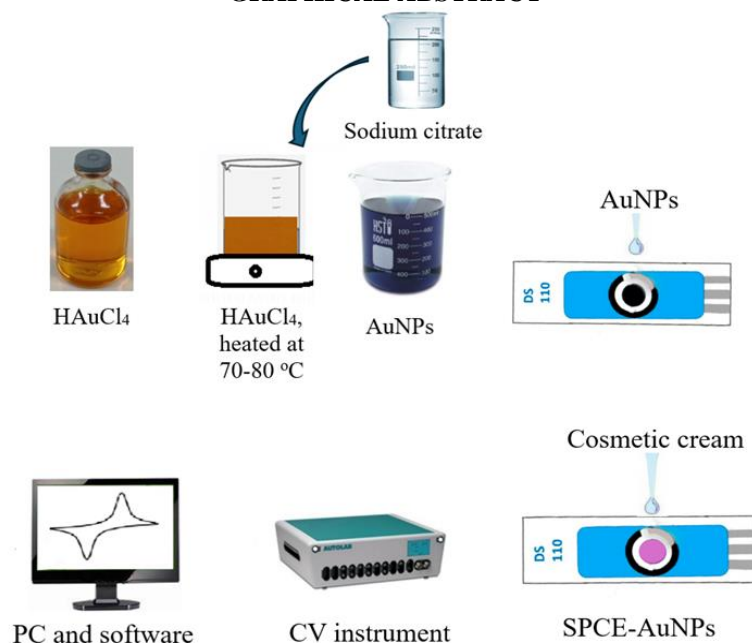
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## GRAPHICAL ABSTRACT



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## ABSTRACT

The cosmetics industry in Indonesia has experienced a significant increase of 12.6% from 2022 to June 2023, with 1025 industries positively impacting the Indonesian economy. It has also triggered several naughty manufacturers to use dangerous chemicals in their cosmetic products, such as mercury (Hg). Therefore, developing a fast and sensitive method for detecting mercury in cosmetics using SPCE-AuNPs is vital. This research was carried out by synthesizing gold nanoparticles (AuNPs) using the reducing agent sodium citrate ( $\text{Na}_3\text{H}_5\text{C}_6\text{O}_7$ ) with varying concentrations and characterization of several chemical instruments. Furthermore, SPCE was modified by AuNPs, and its performance and

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application were tested to detect mercury in cosmetic cream using cyclic voltammetry (CV). SPCE-AuNPs provided an electrochemical response with higher oxidation and reduction peaks than bare SPCE, with an oxidation peak at a potential of 0.1 V to 0.3 V and a reduction peak at -0.05 V to 0.05 V at a scan rate of 0.01 V/s to 0.03 V/s. This demonstrates a robust electrochemical response, indicating that SPCE-AuNPs are suitable for detecting mercury in cosmetic samples due to their high sensitivity, reproducibility, and overall performance, so they can be used for application to detection of mercury in cosmetic samples. The SPCE modified using AuNPs can potentially be used as a sensor for mercury detection in cosmetics

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## 1. INTRODUCTION

Currently, the cosmetics industry in Indonesia has experienced a significant increase of 12.6% in 2023, with 1025 industries [1]. This has positively impacted the Indonesian economy but has triggered several rogue manufacturers to use dangerous chemicals in their cosmetic products. Mercury (Hg) is a chemical derivative of a heavy metal carcinogenic and toxic. In cosmetic products, mercury is usually used as a skin whitener, because it can inhibit the production of melanin, making the skin whiter than before [2]. However, the government has prohibited mercury-based use on the Indonesian Minister of Health Regulation No. 445/Menkes/PER/VI/1998 [3]. Various analytical methods have been developed to analyze mercury, such as atomic absorption spectroscopy (SSA), gas chromatography, colorimetric tests [4], inductively coupled plasma mass spectrometry (ICPMS) [5], and fluorescence spectrometry [6]. However, these methods have several disadvantages, such as being expensive, requiring expert practitioners, and taking a long time to obtain results. So, we need a method with a good level of selectivity, relatively high sensitivity, cost-effectiveness, and fast detection, such as electrochemical sensors, which in recent years have been widely applied [7].

Electrochemical sensors are a type of sensor with electrodes as transducer elements for analyte concentrations based on chemical reactions, where a potential is applied and a measurable current is obtained [8]. In previous studies, glassy carbon electrodes were one of the working electrodes commonly used in electrochemical sensors. However, the measurement requires a huge analyte solution (mL), and the resulting voltage is unstable, so an electrode with a small LOD (limit of detection) capability is needed and is more stable in measuring the analyte solution ( $\mu\text{L}$ ). Therefore, screen-printed carbon electrodes (SPCE) have recently become an alternative electrode to overcome this problem [9]. Current approaches for mercury detection in cosmetics often need to be revised to avoid limitations such as high costs, lengthy analysis times, and insufficient sensitivity for trace amounts of mercury. The effectiveness of electrochemical sensor performance can be increased through modification of metal-based nanoparticles because nanoparticles are sensitive, selective, unique, and good catalysts in electrochemical reactions [10]. One of the metal nanoparticles used in electrochemical sensors is gold nanoparticles (AuNPs). AuNPs were chosen because they have Surface Plasmon Resonance (SPR) in amplifying signals in optical-electrochemical detection techniques and have high surface area and conductivity, which allows the detection of low concentrations of analytes [11]. A new research innovation is proposed: the Screen-Printed Carbon Electrode (SPCE) electrochemical sensor method based on gold nanoparticles to detect mercury in cosmetic products. Through this latest research, it is hoped that the detection of mercury ions in cosmetic samples will be more selective and high sensitivity.

## 2. EXPERIMENTAL METHOD

### 2.1. Materials and Instrumentation

The materials used were cosmetic samples obtained from the commercial market, 1% Sodium Citrate (Merck), pure gold bars (Antam), 37% HCl (Merck), 67% HNO<sub>3</sub> (Merck), Screen Printing Carbon Electrode (Metrohm). All of the chemicals used in this research are analytical grade. The instruments used were Hitachi UH 5300 ultraviolet (UV)-Vis Spectrophotometer and Fourier-

Transform Infrared Spectroscopy (FT-IR) from Perkin Elmer Spectrum Version 10.5. A Horiba SZ-100 pore size analyzer (PSA), Japan and PGSTAT 100 N/250 mV (Metrohm Autolab).

## 2.2 Synthesis of AuNPs

AuNPs were synthesized by adding the reducing agent sodium citrate ( $\text{Na}_3\text{H}_5\text{C}_6\text{O}_7$ ) with varying concentrations. This stage begins with making a stock solution of chloroauric acid ( $\text{HAuCl}_4$ ) 50 mM by dissolving 0.5 g of gold (99.999%, Antam) into a 50 mL mixture of concentrated  $\text{HNO}_3$  (65%) and concentrated  $\text{HCl}$  (37%) (1:3 (v/v)). Next, a 1 mM  $\text{HAuCl}_4$  solution was made from a 50 mM  $\text{HAuCl}_4$  stock solution of 2 mL of 50 mM  $\text{HAuCl}_4$  solution in 100 mL of distilled water. Synthesis of AuNPs was carried out by heating 1 mL of 1 mM  $\text{HAuCl}_4$  solution at a temperature of 70-80 °C for 1-2 minutes and adding 7 mL of sodium citrate solution with varying concentrations of 0; 0.5; 1; 1.5; and 2% (1:7 (v/v)), accompanied by stirring at 4 rpm for 30 minutes until a wine red-bluish purple color was produced, indicating that the AuNPs were successfully synthesized. After that, several material instruments were characterized, including a UV-Vis Spectrophotometer, Particle Size Analyzer (PSA), Fourier Transform Infrared (FTIR), and Transmission Electron Microscopy (TEM). Next, AuNPs modified SPCE. The synthesis procedure of AuNPs is shown in Figure 1a.

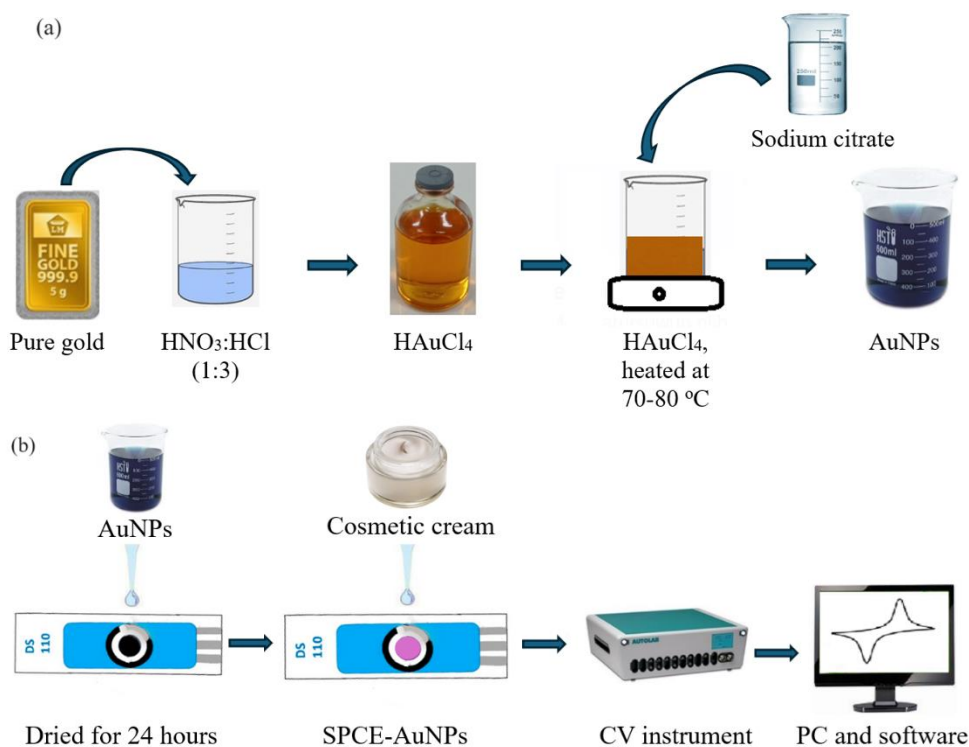


Figure 1. (a) AuNPs synthesis procedure and (b) SPCE-AuNPs synthesis and application for mercury analysis in cosmetic cream using CV.

## 2.3 Modified Screen-Printed Carbon Electrode-gold nanoparticles (SPCE-AuNPs)

Modification of SPCE-AuNPs was carried out using the one drop method (5  $\mu\text{L}$ ) by dropping 5  $\mu\text{L}$  of AuNPs solution on the SPCE working electrode, leaving it for 24 hours and then oven at 50 °C for 5 minutes until a clear layer formed on the SPCE electrode. The SPCE modified by AuNPs was evaluated using CV in the ferricyanide system. The solution used is a solution of  $\text{K}_3[\text{Fe}(\text{CN})_6]/\text{K}_4[\text{Fe}(\text{CN})_6]$  10 mM (1:1 molar ratio) containing 1 M  $\text{KCl}$ . The potential range used in this test was -0.05 to 0.55 V, and the scan rate varied from 0.01 to 0.03 V/s. The synthesis procedure of SPCE-AuNPs and its application for the analysis of mercury in cosmetic creams are shown in Figure 1b.

## 2.4 Test the performance of the electrochemical response of SPCE-AuNPs

Electrochemical measurements of the  $\text{HgCl}_2$  working solution were carried out through SPCE-AuNPs with varying concentrations and continued using cyclic voltammetry. The working solution variation was 0, 1, 2, 3, 4, and 5 ppm in 25 mL. Next, using Cyclic Voltammetry (CV), electrochemical measurements were carried out with a mixture of varying concentrations of  $\text{HgCl}_2$  and 30 mM HCl (4:1 (v/v)) with a potential range of  $-0.4$  to  $0.4$  V and a scan rate of  $0.03$  V/s. In this case, Cyclic Voltammetry (CV) was used to determine the peak current and concentration of mercury to be detected. Cyclic Voltammetry (CV) allows the determination of kinetic parameters such as reaction rate constants and diffusion coefficients commonly used to estimate the concentration of compounds in solution.

The preparation of cosmetic cream samples for analysis was a thorough process, ensuring the accuracy of the results. It involved wet digestion of 1 gram of sample with 10 mL of aquaregia solution  $\text{HClO}_4$  and  $\text{HNO}_3$  (1:1) for 15 minutes, followed by heating at  $80$  °C, filtration, and dilution to 50 mL. The cosmetic cream samples to be measured were made in duplicate, where 4 mL of the sample was added with 1 mL of 30 mM HCl solution. The performance and application test for detecting mercury in cosmetic cream samples using Cyclic Voltammetry (CV) was carried out in duplicate (two repetitions) with a potential range of  $-0.4$  to  $0.4$  V and a scan rate of  $0.03$  V/s.

## 3. RESULTS AND DISCUSSION

### 3.1 Effect of variation of sodium citrate concentration on the AuNPs synthesis

Synthesis was done using the Turkevich method to form small diameter nanoparticles, 10-30 nm [4]. The synthesis was carried out by adding the reducing agent sodium citrate to gold salts in the  $\text{HAuCl}_4$  precursor with a brownish yellow  $\text{Au}^{3+}$  oxidation state, which decreased to  $\text{Au}^0$  forming the AuNPs core [12]. This process is characterized by a change in the color of the solution from yellow to purple, as seen in Figure 2a. The color change of AuNPs is based on the SPR phenomenon, where electrons on the surface of the nanoparticles oscillate when exposed to light, which is influenced by the size and shape of the particles. This process is influenced by the reductant concentration, where 1.5% reductant is considered the optimal concentration, producing stable purple-colored nanoparticles with better size, reaction rate and quality, as in Figure 2b.

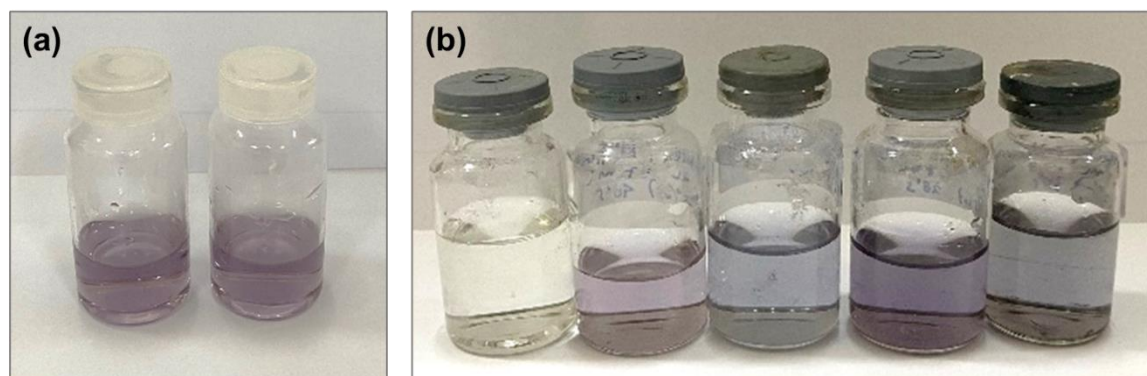


Figure 2. (a) AuNPs solution and (b) visual observation of AuNPs variations of the reducing agent sodium citrate 0; 0.5; 1; 1.5; 2% (1:7 (v/v)).

### 3.2 Characterization of gold nanoparticles (AuNPs)

Characterization was carried out with various chemical instruments. UV-Vis spectrophotometer analysis is used to determine the maximum wavelength of the blank absorption peak and variations in the concentration of the reducing agent sodium citrate, which can be represented in Figure 3a. Based on the research by Oliveira et al., 2023, the data shows that a reducing agent concentration of 1.5% is the best, where AuNPs are formed at a maximum wavelength of 544 nm with an absorbance of 0.219. The results obtained follow the theory of AuNPs formation, which is 500-600 nm [13].

This is based on the SPR phenomenon, generally in the maximum wavelength range ( $\lambda_{max}$ ) 515-560 nm with a color change from yellow to ruby red to purple [14].

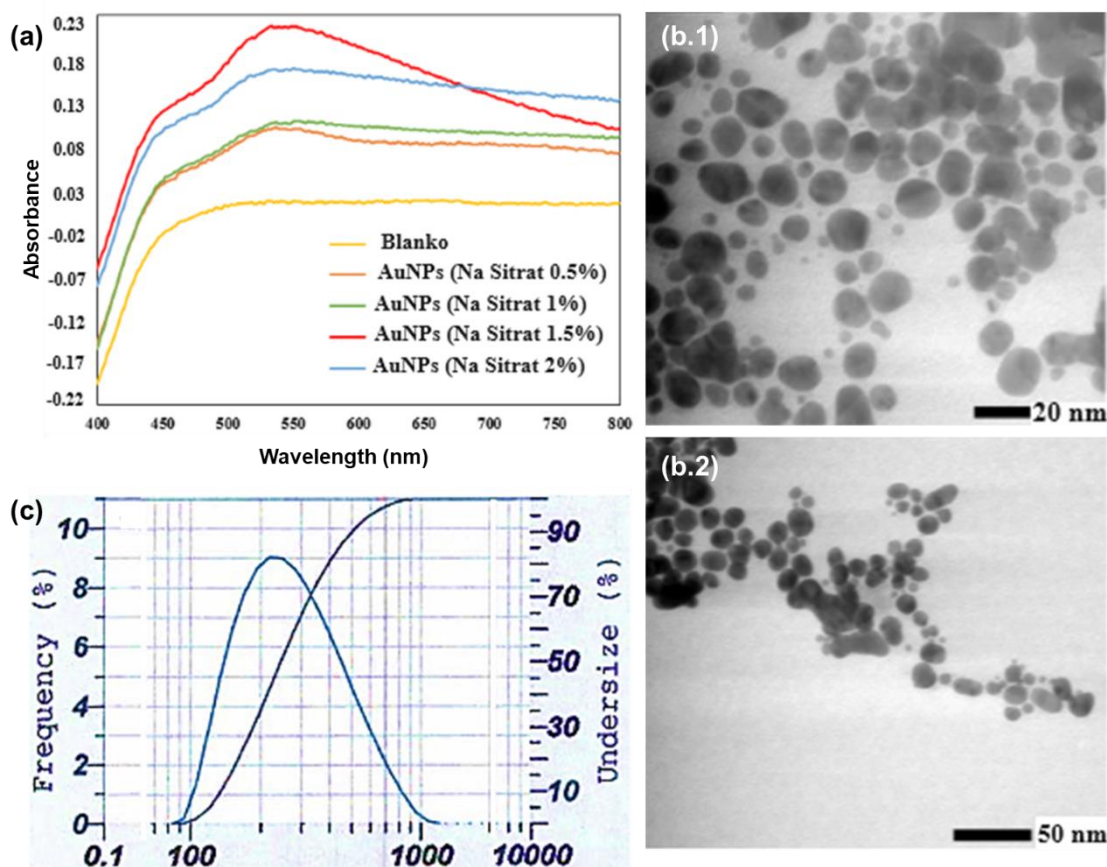


Figure 3. (a) UV-Vis spectra, (b) TEM image and (c) the particle size distribution using PSA of AuNPs.

In transmission Electron Microscopy (TEM), the TEM imaging results of AuNPs are presented in Figure 3b, which shows a spherical shape with a diameter of  $15.06 \pm 3.06$  nm. These results show that gold nanoparticles are tiny or nano-size with a uniform distribution and have good homogeneity [15]. Particle Size Analyzer (PSA) to determine the particle size distribution of AuNPs in a solution sample can be determined through the PSA test, as shown in Figure 3c, where there is a sharp peak at around 199.9 nm with a narrow peak width. This indicates that most particles have an average particle size of 199.9 nm with a narrow size distribution, and the particle size is more uniform. Gold nanoparticles exhibit size differences when characterized using TEM and PSA, with the size measured by PSA being significantly more significant than the results from TEM. This difference occurs because PSA measures the nanoparticle size based on particle diffusion in a medium, capturing the entire particle structure, including the surrounding hydration shell. In contrast, TEM allows for direct measurement of the particle core without considering the hydration shell, resulting in a smaller and more accurate size measurement.

Figure 4 shows that AuNPs with a reducing agent concentration of 1.5% have the sharpest absorption band compared to other reducing agent concentrations. Two prominent peaks at  $3337.82$   $\text{cm}^{-1}$  and  $1634$   $\text{cm}^{-1}$  demonstrate these, with a medium peak at  $1389.81$   $\text{cm}^{-1}$ . The peak at  $3337.82$   $\text{cm}^{-1}$  correlates with the O—H stretching group by intermolecular hydrogen bonds, the sharp peak at  $1634$   $\text{cm}^{-1}$  corresponds to the C=O stretching group and the peak at  $1389.81$   $\text{cm}^{-1}$  corresponds to the C—O stretching group vibration. The presence of C—O groups shows the interaction and stabilization of the nanoparticles formed [16]. Reduction is characterized by the appearance of a significant absorption peak at the C=O wave number (carbonyl group), which indicates the involvement of sodium citrate as a reducing agent in the synthesis of AuNPs [17]. Therefore, this

FTIR analysis confirms that the surface of the formed AuNPs is bounded by citrate groups, which are essential for the stability of the nanoparticles [18].

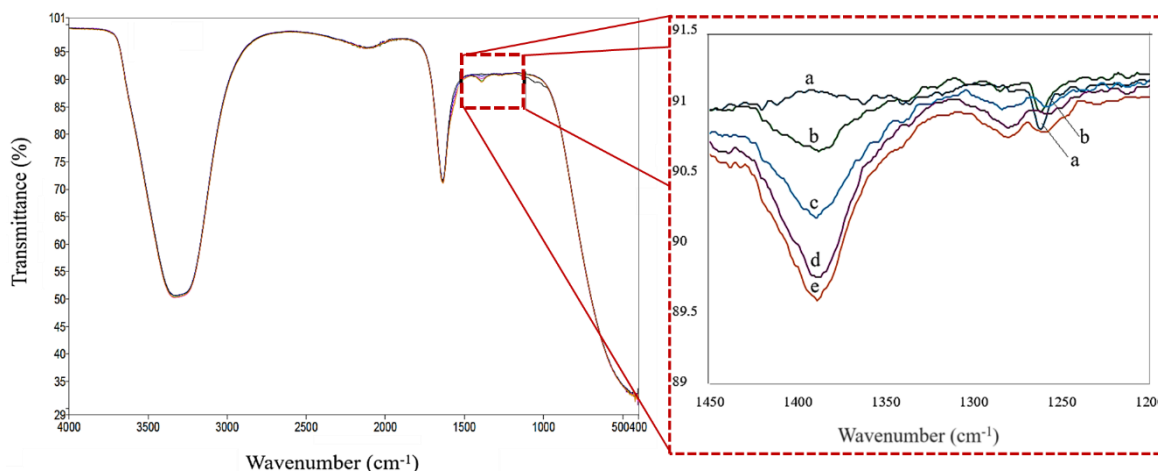


Figure 4. FTIR spectra of the AuNPs with various Na-citrate and FTIR spectra at 1450-1200  $\text{cm}^{-1}$  where: (a):  $\text{HAuCl}_4$ ; b: Na-citrate 1.0%; c: 1.5%; d: 2.0%; e: 0.5%.

### 3.3 Electrochemical performance of SPCE-AuNPs

The prepared SPCE-AuNPs were evaluated for their electrochemical response with CV in the ferricyanide system, namely  $\text{K}_3[\text{Fe}(\text{CN})_6]/\text{K}_4[\text{Fe}(\text{CN})_6]$  with the following reaction:

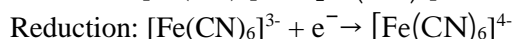
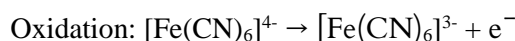


Figure 5a shows that SPCE-AuNPs provides an electrochemical response with higher oxidation and reduction peaks than bare/original SPCE, with an oxidation peak at a potential of 0.1 V to 0.3 V and a reduction peak at -0.05 V to 0.05 V to 0.3 V. 05 V. This is supported by the electrochemical response test of scan rate variations where the performance of SPCE-AuNPs at a scan rate of 0.01 V/s to 0.03 V/s shows that the higher the scan rate, the more fantastic the current read in Figure 5b. This means that the SPCE-AuNPs produced have an excellent electrochemical response, which can be used for mercury standard solutions and cosmetic samples.

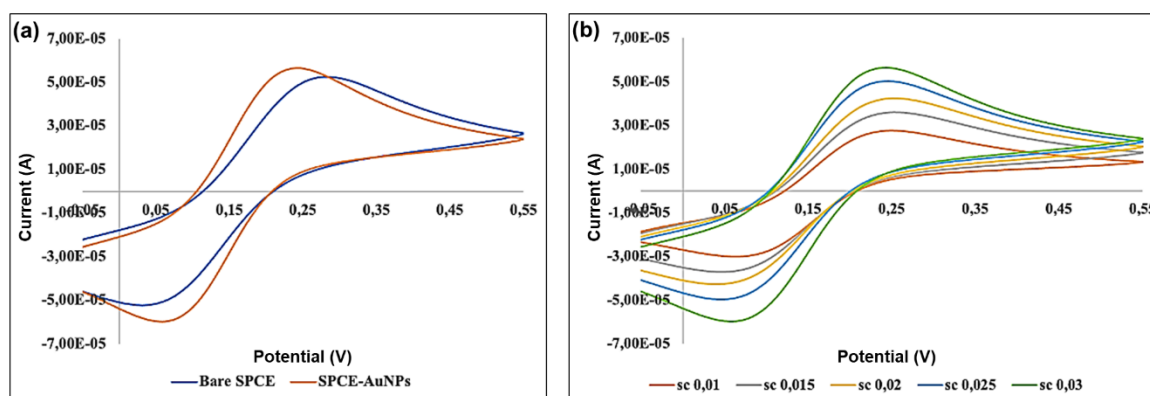
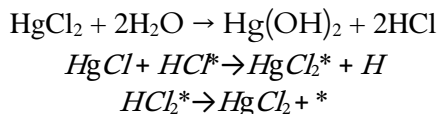


Figure 5. (a) Voltammogram evaluation of the response of SPCE-AuNPs to the  $\text{K}_3[\text{Fe}(\text{CN})_6]/\text{K}_4[\text{Fe}(\text{CN})_6]$  solution system and (b) variation of scan rate 0.01 V/s to 0.03 V/s SPCE-AuNPs.

Determination of mercury levels electrochemically begins with testing the response to a distilled water/HCl solution as a blank using an AuNPs working electrode (WE) to confirm the peak of

mercury oxidation in the  $\text{HgCl}_2$  working solution. The voltammogram shows the difference between the blank solution and the mercury working solution, where the oxidation peak occurs at a potential of 0.093 V as in Figure 6a and the reaction below:



Bimetallic metal nanoparticle (AuNPs) electrodes, with their good catalytic properties and high conductivity, hold significant potential as electrocatalysts. One of the suitable options for electrode modification is gold nanoparticles. Chloroauric acid would be reduced and deposited on the electrode surface when gold nanoparticles are used to alter the electrode. This process increases the electrode's active surface area and speeds up the rate of electron transmission [19]. The  $\text{HgCl}_2$  concentration variation test follows Ilkovic's Law, where the current is directly proportional to the concentration.

The voltammogram results can be used to make a linear correlation between the  $\text{HgCl}_2$  concentration and the peak current at the same potential, 0.09 V, as shown in Figure 6b. Based on Figure 6b, the linear regression equation is obtained, namely  $y=0.4\text{E}-6x - 0.2\text{E}-6$ ,  $R^2=0.9938$ , which shows that the standard concentration ( $\text{HgCl}_2$  working solution) is directly proportional to the peak oxidation current. According to SNI 06-6989.20-2004 regarding heavy metal testing, the correlation coefficient ( $R^2$ ) that meets the requirements is  $\geq 0.990$ . This means that the analytical method used in this research is included in the outstanding category because the correlation coefficient value is close to the specified correlation coefficient value requirements.

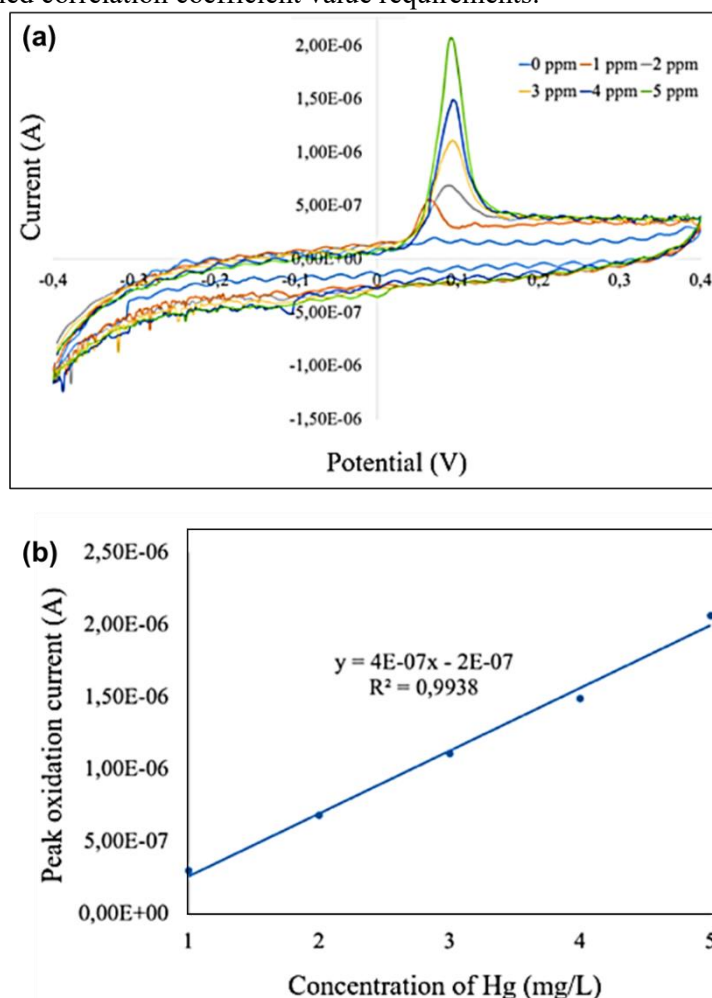


Figure 6. (a) Voltammogram of SPCE-AuNPs testing for varying concentrations of  $\text{HgCl}_2$  working solution 1, 2, 3, 4, and 5 ppm and (b) linearity graph of concentration and peak oxidation current.

Measurement of the electrochemical response of cosmetic cream sample significant in the potential range of 0.1 V to 0.3 V, where the potential range area is a peak indicating the presence of mercury compounds according to the SPCE-AuNPs performance test in the  $\text{HgCl}_2$  working solution. In the cosmetic cream sample, the oxidation current is significant in the potential range of 0.1 V to 0.3 V, where the potential range area is a peak indicating the presence of mercury compounds following the SPCE-AuNPs performance test in the  $\text{HgCl}_2$  working solution Figure 7a and theory by [20]. The current oxidation peaks obtained in Figure 7a contain two peaks in the potential area of 0 V to 0.3 V, where this occurs due to the presence of interferences/impurities that interact with the electrode, thus changing the ion distribution and electrode potential. As a result, there is a shift in the electrochemical potential at the peak of the mercury oxidation current. The stability value of SPCE-AuNPs was at the 60<sup>th</sup> minute, and it was measured using repeated tests, which can be seen in Figure 7b, so it can be concluded that the SPCE-AuNPs electrode has proven to be sensitive and stable in detecting the presence of mercury compounds.

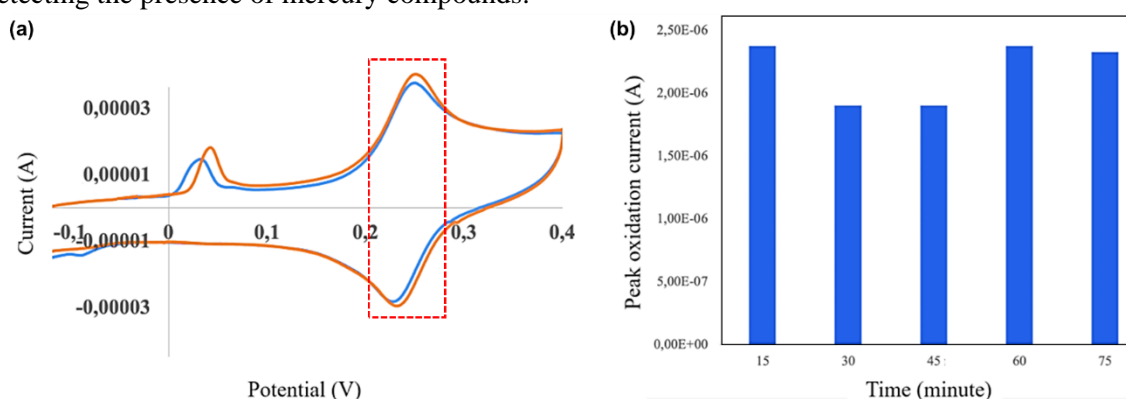


Figure 7. (a) Duplo voltammogram of cosmetic samples and (b) SPCE-AuNPs stability test.

TABLE I. Concentration of mercury contained in samples

No	Sample	Peak oxidation current (A)	Conc. of Hg (mg/kg)
1	Repetition 1	$3.69 \times 10^{-5}$	25
2	Repetition 2	$3.98 \times 10^{-5}$	25

The mercury concentration in cosmetic cream samples is determined by substituting the peak current of the sample into the linear regression equation of the working solution curve to obtain the results as in Table 1. Cosmetic cream sample X was tested in duplicate (two repetitions). The results of repetitions 1 and 2 obtained 25 ppm, while the label for cosmetic product X stated that it did not contain mercury. According to BPOM Number 18 of 2015 concerning Technical Requirements for Cosmetic Ingredients in Indonesia and the United States Food and Drugs Administration (US FDA), the permitted mercury level in cosmetics is 1.0 ppm [21,22]. Thus, according to Table 1, the cosmetic samples analyzed contain mercury that exceeds the usage limits and is therefore unfit.

#### 4. CONCLUSION

Based on the research carried out, the SPCE (Screen Printed Carbon Electrode) sensor modified by AuNPs has the potential to be a sensitive sensor for the detection of mercury in cosmetics. This is indicated by an oxidation peak current of around 0.1 V to 0.3 V and a scan rate of 0.03 V/s, where the mercury concentration in 1 gram of cosmetic sample is 25 ppm.



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