Nano Titanium(IV) Oxide Modified Carbon Paste Electrode for Analysis of Butylated Hydroxytoluene Concentration in Lotion by Cyclic Voltammetry

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Abstract: *This research focuses on the manufacture and application of TiO_2 NPs modified carbon paste electrodes (TiO_2 NPs-CPE) to analyze butylated hydroxytoluene (BHT) concentration in lotions by cyclic voltammetry. This study aimed to determine the best composition of TiO_2 NPs-CPE, optimal addition of phosphate buffer pH, application of deposition time, and optimal scan rate by cyclic voltammetry. The BHT standard solution was used to determine the electrode with the best conditions so that an optimal voltammogram was obtained at a composition of 3:5:2 electrode (carbon:TiO_2 NPs:paraffin oil), phosphate buffer pH 8, deposition time of 25 s, and scan rate of 100 mV s^{-1}. Obtaining BHT levels of samples A, B, and C with TiO_2 NPs-CPE by cyclic voltammetry were 0.4686, 0.48029, and 0.39044%, while the concentration of samples in HPLC were 0.46873, 0.48111, and 0.39191%. A significance value of 0.985 (> 0.05) was obtained, meaning there was no significant difference in the results of measuring the concentration of BHT in the lotion samples.*

Keywords: *CPE; TiO_2 NPs; butylated hydroxytoluene; cyclic voltammetry*

■ INTRODUCTION

Lotion has become one of the types of cosmetics that moisturize to protect the skin from sun exposure. In lotions, antioxidants are often added as active ingredients or stabilizers [1]. One kind of antioxidant that is widely used is butylated hydroxytoluene (BHT) because it is inexpensive to produce [2]. However, BHT has a toxic effect on the lungs and has the potential to cause tumor effects [3]. It is even known that the addition of BHT in doses of 250 and 500 mg/kg in rats has a toxic effect on motor and neurobehavioral activity, as well as histopathological changes in the brain, heart, and lungs [4]. Therefore, the European Committee for Science and Consumer Safety has decided that the range of permitted BHT levels in cosmetics is 0.0002 to 0.8000%, in mouthwashes a maximum of 0.001%, and in toothpastes a maximum of 0.1000% [5].

Nowadays, there are several instruments available for detecting BHT, such as high-performance liquid chromatography (HPLC) and gas chromatography (GC) [6]. However, these analysis methods have disadvantages. HPLC may take a long time and use hazardous solvents [7-8]. GC has limitations of low resolution and low mass accuracy [9]. So, another method is needed to analyze BHT. Cyclic voltammetry is a method based on current and voltage measurements for quantitative analysis in terms of oxidation and reduction reaction processes, adsorption processes on electrode surfaces, and chemical electron transfer mechanisms [10]. The cyclic voltammetry analysis method also has other advantages, including efficiency, high sensitivity, relatively low cost, simplicity, and easy application [11]. Carbon paste is an electrode that is widely applied in the field of electroanalysis, such as voltammetry. The advantages of carbon paste electrodes are that it has a wide potential range, are stable, inexpensive, inert, environmentally friendly, easy to obtain, uncomplicated in the manufacturing process, have a porous surface, produce low residual current, and can be used for various sensors [12-13].

The performance of the carbon paste working electrode can be maximized by modification using other

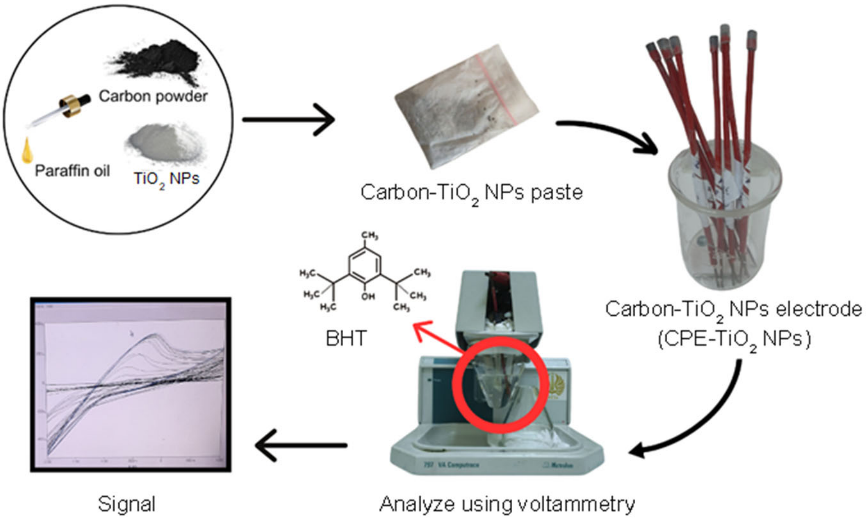


Fig 1. BHT analysis scheme using cyclic voltammetry with TiO_2 NPs-CPE

materials [14] because it affects the selectivity and kinetics of an electrochemical reaction, and sometimes the product [15-16]. This modification aims to obtain a working electrode that is more sensitive and produces an optimal current response, thus facilitating the analysis process in voltammetry. One of the modifiers used for carbon paste electrodes is the addition of anatase TiO_2 nanoparticles (NPs). TiO_2 is an inorganic material that is inert, non-toxic, and has the potential as adsorbent in electrode [17-18]. The crystal structure of anatase has a wider activated area than rutile. The use of nano-sized modifiers in electrodes can also improve redox reaction reversibility, sensitivity, and limit of detection due to their wider surface area [19].

In addition, the increase in electrode performance can be influenced by adding pH buffer, deposition time, and electrode scan rate. The addition of buffer is adjusted to the range of stable conditions of the object compound to be identified to prevent changes in pH when a redox reaction occurs. It is known that BHT can be stable with the addition of a pH buffer, except in conditions of high pH or equal to 9 [20]. Deposition time and scan rate have an important influence on improving electrode performance when viewed from the current response. The higher the deposition time and scan rate, the greater the current response of the resulting voltammogram [21]. Based on the explanation of this background, there was urgency in optimizing the performance of nano TiO_2-modified carbon paste electrodes (TiO_2 NPs-CPE) regarding the variation of the best electrode composition, addition of pH buffer, deposition time, and electrode scan rate. Thus, TiO_2 NPs-CPE can be used as an environmentally friendly alternative electrode with high sensitivity to analyze BHT levels by cyclic voltammetry (Fig. 1).

■ EXPERIMENTAL SECTION

Materials

The materials used in this study included copper wire, TiO_2 nanopowder (99.99%, Hongwu new material), paraffin oil, BHT (99%), Whatman 41 filter paper, methanol (96%), distilled water, activated carbon powder, potassium chloride (KCl), hydrochloric acid (HCl, 37%), sodium hydroxide (NaOH), $NaH_2PO_4{\cdot}2H_2O$, and $Na_2HPO_4{\cdot}12H_2O$ from Merck.

Instrumentation

The tools used in this study included 797·VA Computrace voltammetry equipped with Pt auxiliary electrode, Ag/AgCl (saturated KCl) reference electrode, HPLC type HP 1050, pH meter, analytical balance, spatula, hard-plastic cylinder, sandpaper, magnetic stirrer, beaker, volumetric flask, measuring pipette, dark glass bottle, and bulb pipette.

Procedure

BHT concentration was analyzed using a TiO_2 NPs-CPE with the best composition, which was obtained by adding phosphate buffer pH, deposition time, and optimal scan rate by cyclic voltammetry.

Preparation of sample and standard solution

The body lotion from three different brands was weighed (1 g) and dissolved using 96% ethanol in a 50 mL volumetric flask. The solution was then filtered using Whatman 41 filter paper, and the filtrate was stored in a dark glass bottle. A 0.25 g of BHT powder was weighed and dissolved in a 250 mL volumetric flask with 96% ethanol to form a 1000 ppm BHT. In this study, BHT standard solutions used 20, 40, 60, 80, and 100 ppm concentrations. Dilution was carried out from high to low concentration.

Electrode body manufacturing

Copper wire 1 mm in diameter is cut into an 11 cm length. The tip of the cable was peeled 2 mm until it showed only the copper, while at the base, it was peeled 1.5 cm long. The exposed copper wire is then sanded down. Furthermore, the tip of the cable is mounted on a hard-plastic cylinder 7 mm long, with 4 mm of its part glued to the tip of the electrode skin and the other 3 mm left as space to fill the electrode composite.

TiO_2 NPs-CPE assembly

The working electrode composition of TiO_2 NPs-CPE consists of graphite powder, nano TiO_2 powder, and liquid paraffin with various compositions of 5:0:5, 3:5:2, 3:4:3, 3:3:4, and 3:2:5 (w/w). At a composition of 3:5:2, 0.3 g of graphite powder, 0.5 g of nano TiO_2, and 0.2 g of liquid paraffin were weighed. The three components are stirred until evenly mixed to form a paste. The paste, which has been thoroughly mixed, is inserted into the electrode body using a spatula until it is solid and fills the space. The same thing was done for the second paste composition, and so on.

Determination of the best TiO_2 NPs-CPE composition

The working electrode in various compositions of carbon paste modified by nano TiO_2, i.e., 5:0:5, 3:5:2, 3:4:3, 3:3:4, and 3:2:5 (w/w), was determined in 10 mL of 80 ppm BHT solution given the addition of 5 mL of phosphate buffer pH 7 and 10 mL of KCl. The concentration of KCl solution is 50–100 times that of the sample [19]. The solution was tested using the cyclic voltammetry method with a potential of −2 to 2 V, a deposition time of 10 s, and a scan rate of 0.05 V/s. The best electrode is selected from the one that produces the current with the highest peak.

Optimum phosphate buffer pH addition determination

Phosphate buffer pH 7.0, 7.5, 8.0, 8.5, and 9.0 were added to 10 mL of 80 ppm BHT solution and 10 mL of KCl, respectively. The mixture was then tested using the best TiO_2 NPs-CPE by cyclic voltammetry at a potential of −2 to 2 V, a deposition time of 10 s, and a scan rate of 0.05 V/s.

Optimum deposition time determination

The best TiO_2 NPs-CPE with optimal buffer addition was tested by cyclic voltammetry in 10 mL of 80 ppm BHT solution and 10 mL of KCl at a potential of −2 to 2 V, a scan rate of 0.05 V/s, and a deposition time of 5, 10, 15, 20, and 25 s.

Optimum scan rate determination

The best TiO_2 NPs-CPE with optimal buffer addition and optimal deposition time was tested in 10 mL of 80 ppm BHT solution and 10 mL of KCl at potential −2 to 2 V, and it was determined in various scan rates in 0.05, 0.075, 0.1, 0.125, and 0.15 V/s using cyclic voltammetry.

■ RESULTS AND DISCUSSION

The research results obtained include the best electrode composition of TiO_2 modified carbon paste, optimization of electrode performance under conditions of buffer addition, deposition time, and scan rate, the LOD and LOQ values, and the results of sample concentration in lotions by cyclic voltammetry and HPLC.

XRD Characterization

This XRD characterization aims to determine the particle size and type of TiO_2 crystal structure prior to application to the carbon paste electrode. This analysis uses the wavelength at 0.15406 nm and the Scherrer constant of 0.9 for Eq. (1).

$$D = \frac{k\lambda}{\beta\cos\theta} \tag{1}$$

Based on the results of the XRD analysis, the diffractogram of the TiO_2 powder sample was obtained as Fig. 2 and explained in Table 1. The diffractogram data above shows the appearance of 5 peaks with the highest intensity of 2θ at 25.2272°, which means that the crystal structure of TiO_2 is anatase. Furthermore, the particle size of TiO_2 NPs anatase was analyzed based on the Debye-Scherrer equation, so the average particle size of TiO_2 NPs anatase was 48.66 nm. This shows that the TiO_2 powder fulfills the requirements as a nano-sized particle with a good anatase crystal structure.

FTIR Characterization

This study used FTIR characterization to analyze the TiO_2 functional group as a carbon paste electrode modifier. FTIR characterization results can also indicate the presence of other functional groups, in addition to being useful for identifying the TiO_2 anatase functional group. Fig. 3 shows the results of the characterization of anatase TiO_2 powder, it is known that the spectrum shows five peaks, of which two have the lowest transmittance. Low transmittance means that the infrared radiation transmitted to the detector is very small, or, in other words, more infrared is absorbed in the sample. The following data on transmittance and functional groups are presented in Table 2. The low transmittance also indicates the dominance of functional groups at certain wavenumbers. The signal at 402 cm^{-1} is characteristic of the titanium functional group in the that in the TiO_2 sample, anatase absorbs a lot of water

Table 1. Diffractogram peak of TiO_2 NPs anatase

2θ	Intensity (a.u.)	FWHM
25.2272	312.27	0.1673
31.6595	12.42	0.1004
47.8872	102.05	0.4684
53.7377	39.76	0.8029
54.9365	57.91	0.8029

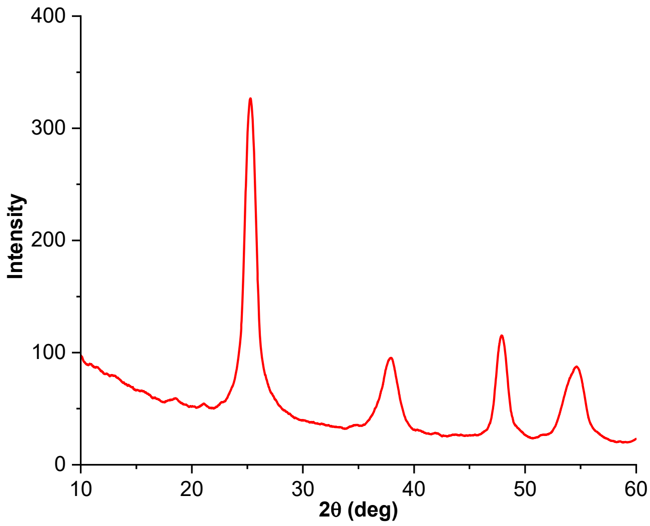


Fig 2. TiO_2 NPs anatase diffractogram

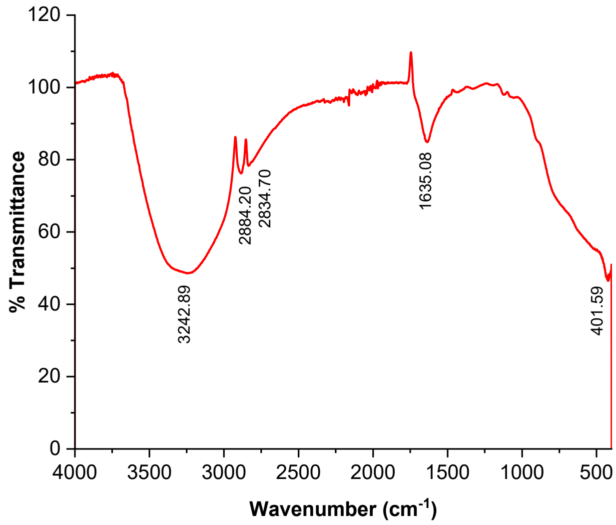


Fig 3. FTIR spectrum of TiO_2 NPs anatase

Table 2. FTIR data of TiO_2 NPs anatase

Wavenumber (cm^{-1})	Transmittance (%)	Functional group
3243	48.57	O–H stretching
2884	76.20	C–H stretching alkane
2835	78.29	C–H stretching aldehyde
1635	84.80	C=O stretching aldehyde/ketone
402	48.41	Ti–O–Ti stretching (anatase titania)

because it forms bonds between Ti–OH, while the other functional groups are organic residual groups that have not been lost during the synthesis of TiO_2 anatase [22].

Determination of the Best TiO_2 NPs-CPE Composition

TiO_2 NPs-CPE is made by mixing 3 components, including carbon, TiO_2 powder, and paraffin oil, with a composition of 3:5:2, 3:4:3, 3:3:4, 3:2: 5, and 5:0:5. The composition of graphite powder was maintained constantly, while the composition of paraffin and TiO_2 NPs was modified to determine any changes in electrode performance. Paraffin oil was chosen because it could homogenize the mixture of carbon powder and TiO_2 NPs as paste form. In this research, carbon powder from graphite was used because it is inert and a good conductor, while TiO_2 NPs are a modifier because of their semiconductor and have a large surface area, thus increasing the electron transfer activity of the analyte to the electrode surface. The fabrication of the electrode is based on a three-component diagram that is shown in Fig. 4.

The composition of the best TiO_2 NPs-CPE was also determined from the highest BHT voltammogram peak current shown in Fig. 5 and Table 3. It is known that the TiO_2 NPs-CPE with a composition of 3:2:5 produces the smallest current response of 0.996 µA. The composition electrodes were given a lot of paraffin oil addition, while the nano TiO_2 modifier was very little. The addition of excess paraffin oil is known to impact the conductivity of the electrodes. The more paraffin oil is added, the lower the conductivity [23]. This happens because the composite particles in the electrodes are in a tenuous position, which results in a small current response. In addition, TiO_2 NPs-CPE with a composition of 3:5:2 has the highest current response among the other compositions, namely 374.420 µA. This is because in this composition, modifiers are added in large quantities, while paraffin oil in small amounts makes the electrode composite particles close together, thereby increasing the conductivity of the electrode and the current response.

Based on Fig. 5, a voltammogram is obtained for each electrode with a predetermined composition. A good voltammogram has high and sharp peaks to analyze the current response. In addition, the peaks of oxidation or reduction can be used as a benchmark for a current response. However, the voltammogram image above shows an upward peak, which means that the BHT oxidation reaction occurred on the electrode surface. The BHT oxidation reaction occurs when the electrode

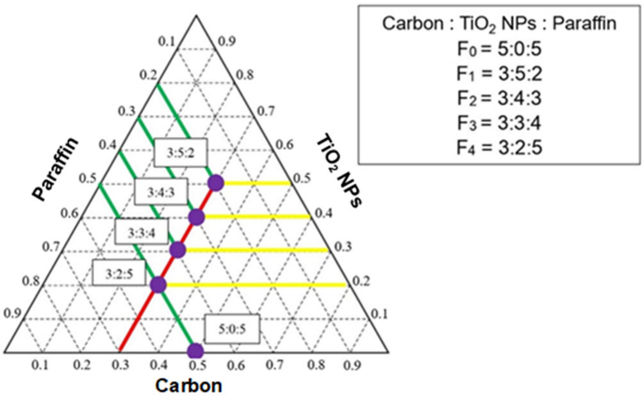


Fig 4. The 3 components diagram

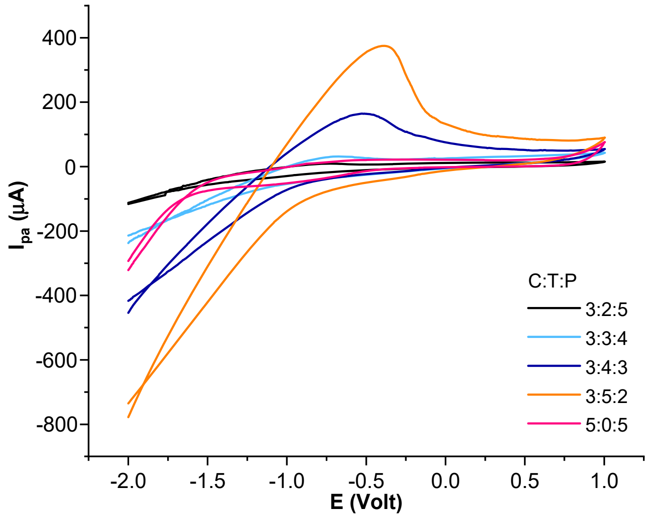


Fig 5. BHT voltammograms with variation TiO_2 NPs-CPE in carbon:TiO_2:paraffin compositions

Table 3. Current response in various TiO_2 NPs-CPE compositions

TiO_2 NPs-CPE composition (carbon:TiO_2:paraffin)	I_{pa} (µA)
3:2:5	0.996
3:3:4	31.749
3:4:3	164.460
3:5:2	374.420
5:0:5	2.066

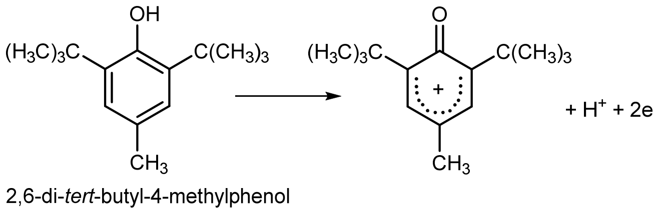


Fig 6. BHT oxidation anodic mechanism [24]

potential is scanned from a negative potential towards a more positive potential, resulting in the release of 2 electrons on the electrode surface accompanied by the formation of phenolic groups to form positively charged BHT ions, which is shown in Fig. 6.

Optimum Phosphate Buffer pH Addition Determination

The addition of a pH buffer to this analyte aims to stabilize the pH of BHT when oxidation or reduction reactions occur with a wide potential application. In addition, proper pH conditioning can increase the current response of the electrode. The addition of buffer is adjusted to the range of stable conditions of the object compound to be identified to prevent drastic changes in pH when a redox reaction occurs [25]. In this research, phosphate buffer is used from the combination of 0.1 M Na_2HPO_4 and NaH_2PO_4.

Based on the voltammograms in Fig. 7(a) and 7(b) and the peak response of the electrode current, it was found that pH 8 was the most optimal BHT pH with a current of 406.480 µA. It is known that BHT can be stable with the addition of a pH buffer, except in conditions of high pH or equal to 9 [26]. The detailed electrode current response is shown in Table 4. The oxidation of BHT in the electrode surface releases hydrogen ions and positively charged BHT ions, so it is unstable at a higher pH. With proper pH, conditioning can improve the current response of the electrode by the reaction activity [27]. The addition of buffer is adjusted to the range of stable conditions of the object compound to be identified to prevent changes in pH when a redox reaction occurs.

Optimum Deposition Time Determination

Determination of the optimal deposition time is intended as a step to determine the response of the highest electrode current due to the long immersion of the electrode in a voltammetry cell so that reduction-oxidation occurs on the surface of the electrode. Based on Fig. 8 and Table 5, the best electrode deposition time occurred at 25 s deposition with a peak of 415.790 µA. The higher the deposition time applied, the higher the resulting current response. This happens because the long

Table 4. Current response in various pH

pH	I_{pa} (µA)
7.0	375.040
7.5	384.210
8.0	406.480
8.5	393.492
9.0	385.399

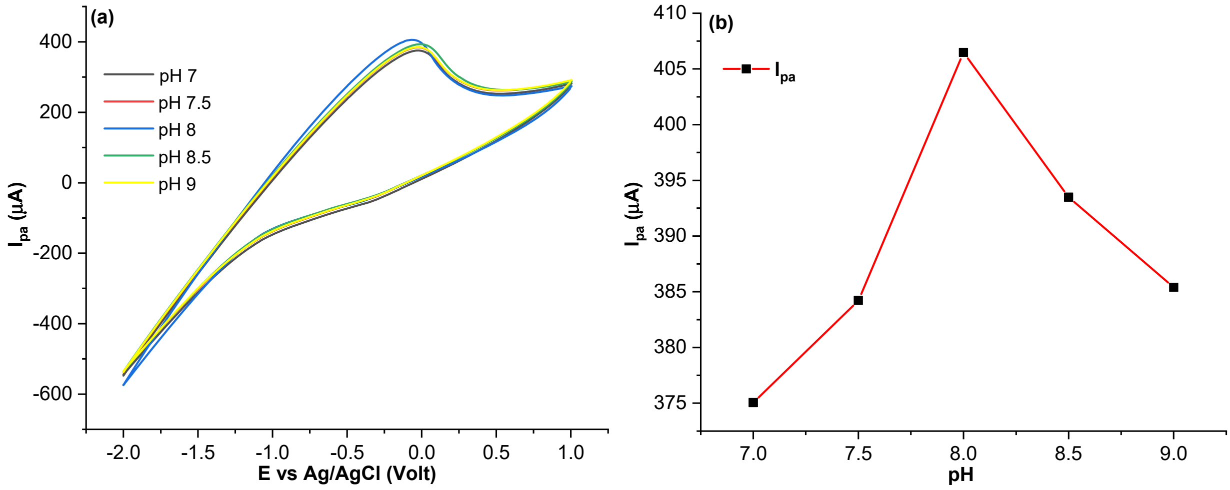


Fig 7. (a) The voltammogram and (b) diagram of BHT in pH 7–9

deposition time affects the oxidation process of BHT in the sample. The longer deposition time applied can maximize the oxidation of BHT into positively charged BHT ions so that the current response is high.

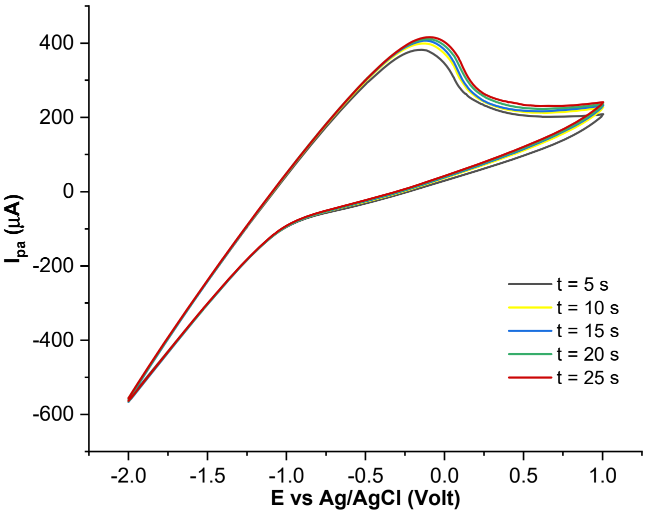


Fig 8. Voltammogram in various deposition time

Table 5. Current response in various deposition time

Deposition time (s)	I_{pa} (μA)
5	382.170
10	399.150
15	406.620
20	410.360
25	415.790

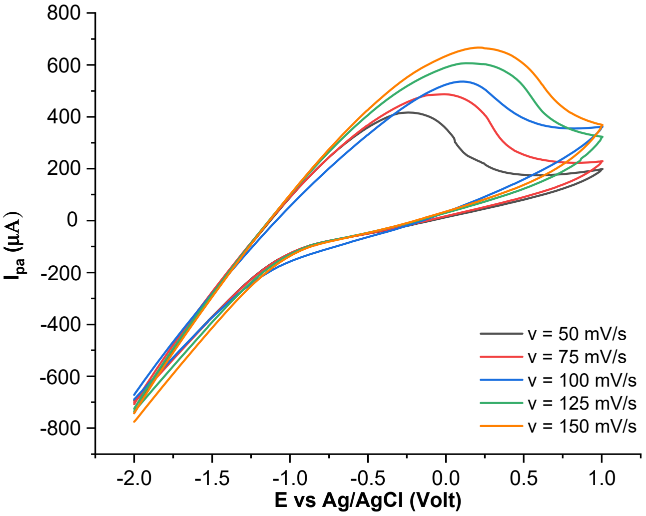


Fig 9. Voltammogram in variations scan rate

Table 6. Current response in various scan rate

Scan rate (mV/s)	I_{pa} (μA)
50	415.650
75	486.360
100	535.910
125	606.620
150	666.750

Optimum Scan Rate Determination

Scan rate is the scanning rate of the electrode in an analyte with a certain potential range. Scan rate optimization aims to determine the optimal current response of an electrode. The current response indicates that the redox reaction of a compound in the analyte can be scanned by an electrode at a certain scanning rate. The following presents the voltammogram data and current response in Fig. 9 and Table 6. The higher the scan rate, the higher the current response [28]. With increasing scan rates, the redox peaks become broadened and distorted, suggesting higher polarization in higher scan rates [29-30]. A scan rate of 100 mV/s produces a current response of 535.910 μA with sharp and clear voltammogram peaks. The shape of the sharp and obvious peaks indicates that the charge transfer at the electrode surface occurs optimally. This means that at a scan rate of 100 mV/s, the diffusion rate is close to or equal to the rate of the BHT oxidation reaction, resulting in optimal charge transfer of the analyte to the electrode surface.

Linear Analysis of Scan Rates

Electrode reaction depends on scan rate, which was studied by varying the scan rate from 50, 75, 100, 125, and 150 mV/s at a concentration of 80 ppm. The plot between current vs scan rate resulted in Fig. 8 ($I_{pa} = 2.49 \times 10^{-3}v + 2.93271 \times 10^{-4}$; $R^2 = 0.99885$), otherwise the plot between anode peak current vs square root of scan rate ($I_{pa} = 4.98 \times 10^{-3}v^{1/2} + 2.9327 \times 10^{-4}$; $R^2 = 0.99885$). The value of log peak current vs log scan rate suggested that the reaction in the electrode surface was diffusion-controlled [31]. The plot of log peak current vs log scan rate is shown in Fig. 10.

Determination of BHT Concentration by Cyclic Voltammetry

Fig. 11 shows the voltammogram data for standard BHT concentration from 20–100 ppm, while Table 7 shows the details of the oxidation current. The result of the voltammogram curve, the standard equation, can be obtained $I_{pa} = 4.9383 \times 10^{-6}C + 1.1986 \times 10^{-4}$. so that the I_{pa} obtained for samples A, B, and C were 613.328, 310.583, and 220.006 µA respectively.

Determination of BHT Concentration using HPLC

The separation process of BHT using the C18 column with acetonitrile as eluent. Based on the resulting chromatogram, the standard BHT equation for

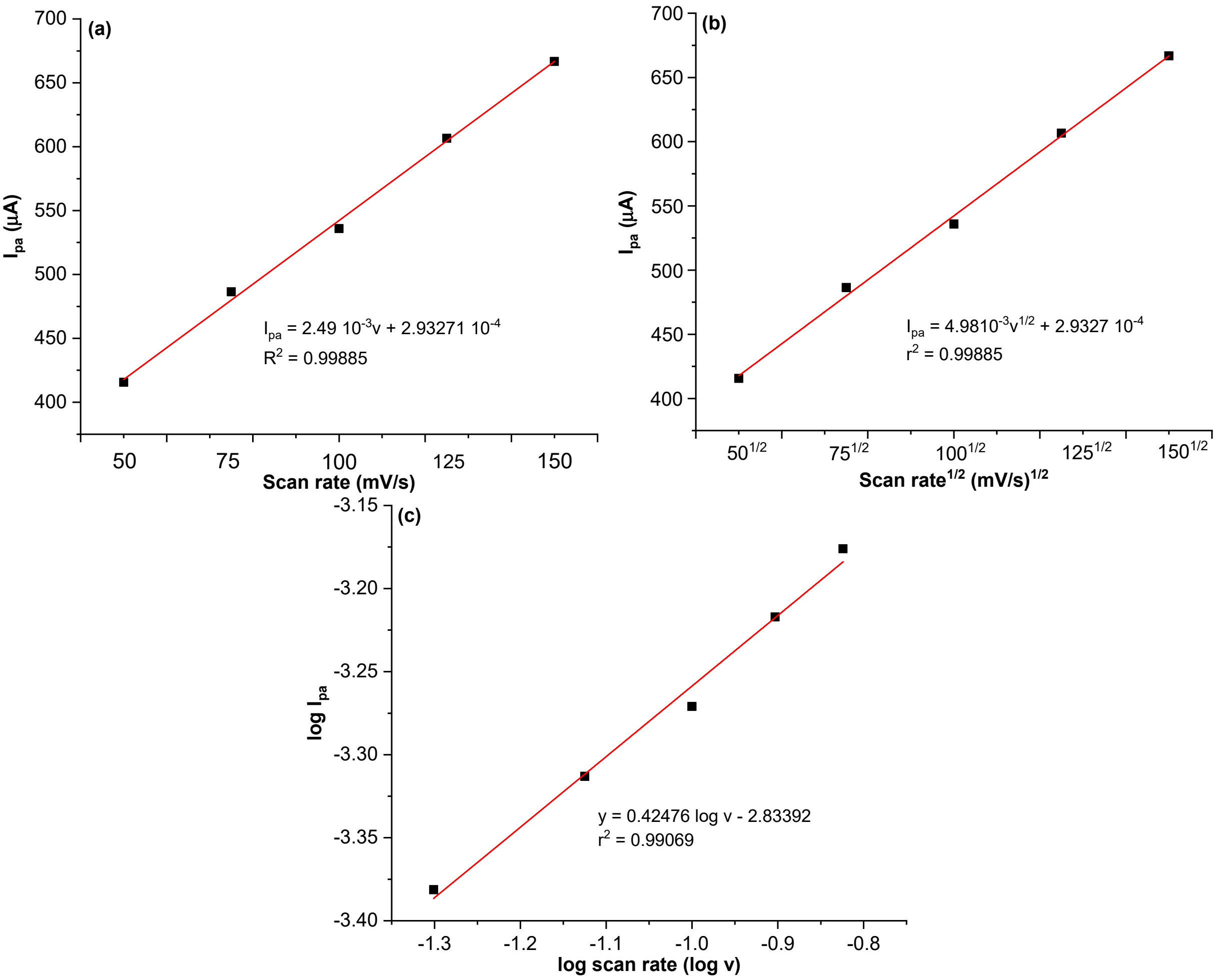
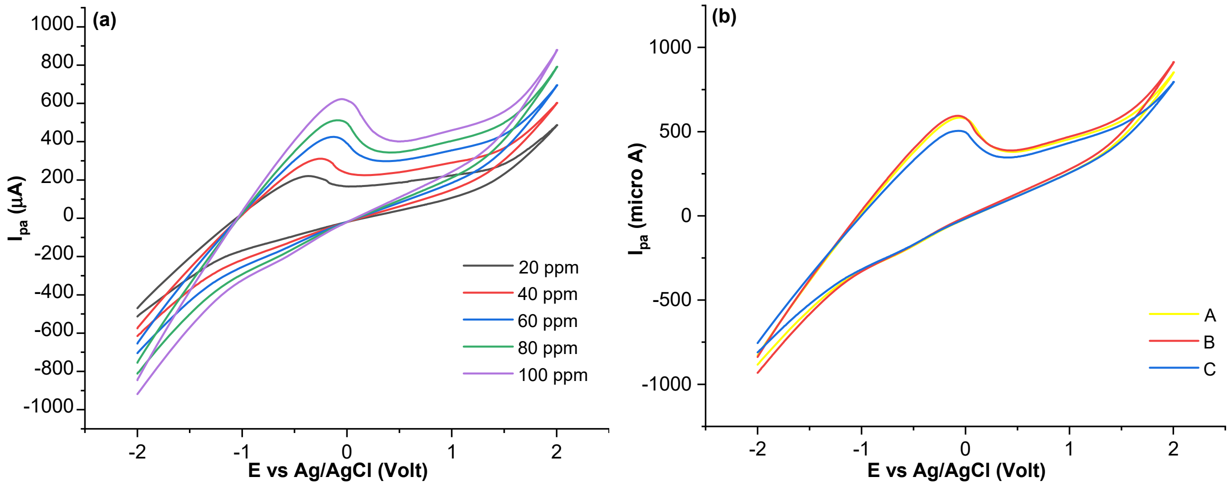


Fig 10. The plot of (a) peak current vs scan rate, (b) peak current vs scan rate$^{1/2}$, and (c) log peak current vs log scan rate



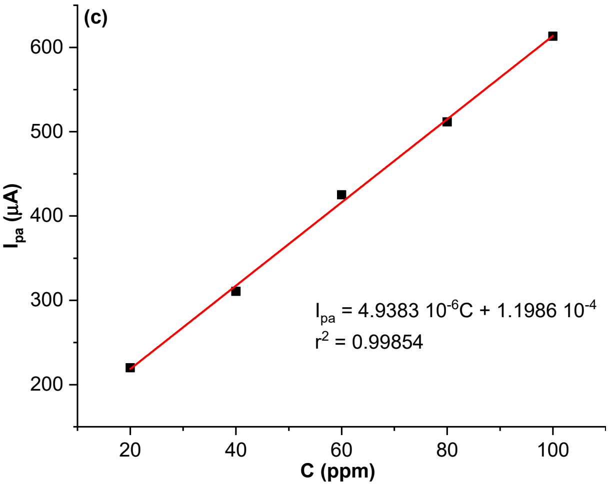


Fig 11. The voltammogram of BHT (a) standard solutions, (b) samples, and (c) linear regression

Table 7. Current response of BHT standard solutions

Concentration (ppm)	I_{pa} (µA)
100	613.328
80	511.602
60	425.297
40	310.583
20	220.006

Table 8. BHT concentration in lotion by cyclic voltammetry

Samples	I_{pa} (µA)	Concentration (ppm)	%
A	613.328	93.720	0.469%
B	310.583	96.058	0.480%
C	220.006	78.089	0.390%

Table 9. BHT concentration in lotion using HPLC

Samples	Height (mAU)	Concentration (ppm)	%
A	38.05263	93.74635	0.46873%
B	38.99894	96.22140	0.48111%
C	32.17827	78.38215	0.39191%

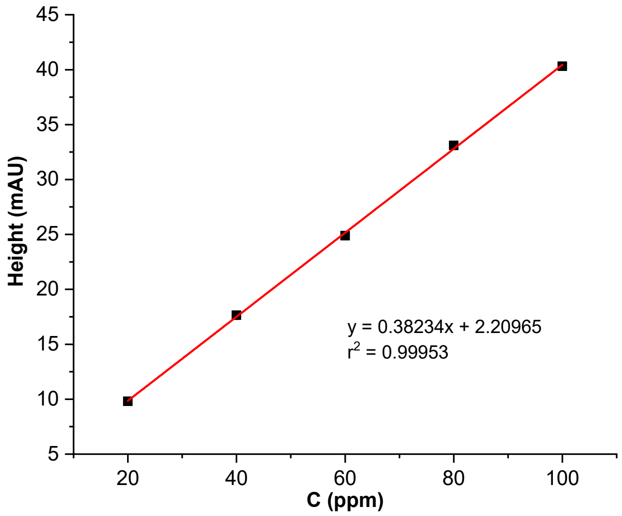


Fig 12. BHT standard curve using HPLC

this HPLC instrument is y = 0.38234x + 2.20965 (Fig. 12). The following data shows the percentage of BHT in samples A, B, and C tested using TiO_2 NPs-CPE working electrodes by cyclic voltammetry and HPLC instruments in Tables 8 and 9.

Based on the statistical analysis of the independent t-test using SPSS, a significance value of 0.985 (> 0.05) was obtained, which means that there was no significant difference in the results of measuring the concentration of BHT in the lotion samples using TiO_2 NPs-CPE by cyclic voltammetry and HPLC instruments. All three brands of lotion still fulfil the threshold limit for BHT content in cosmetics set by the European Committee for Scientific and Consumer Safety, which is 0.0002 to 0.8% or equivalent to 2 to 8,000 ppm.

■ CONCLUSION

The TiO_2 NPs-CPE can be used as an electrode to analyze BHT concentration. The 3:5:2 composition was the best composition for TiO_2 NPs-CPE with the addition of pH 8 phosphate buffer, 25 s deposition time, and 0.1 V/s scan rate. Obtaining BHT levels of samples A, B, and C with TiO_2 NPs-CPE by cyclic voltammetry were 0.4686, 0.48029, and 0.39044%, while the recorded concentration of samples in HPLC were 0.46873, 0.48111, and 0.39191%, respectively. There was no significant difference between the concentrations of BHT and TiO_2 NPs-CPE by cyclic voltammetry and HPLC based on the statistical analysis.

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■ CONFLICT OF INTEREST

The authors do not have a conflict of interest.

■ AUTHOR CONTRIBUTIONS

Nur Anisa Rosyiidah conducted electrode fabrication, optimization, sample measurement, and article writing, while Pirim Setiarso contributed to the recommendation of measurement techniques, data analysis, and article writing.

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