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Electrochemistry Response of Platinum Powder Composite as Electrode for Sensor Capsaicin

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ABSTRACT. So far, the concentration of capsaicin in food has been determined using the tongue. Several analytical methods were developed to determine the concentration of capsaicin, but they have many drawbacks such as being expensive, long analysis time, requiring hazardous chemicals, and being difficult to operate. This research aims to prepare a material that has high stability and excellent electrocatalytic activity to determine capsaicin concentration. The research was conducted by preparing powdered platinum electrodes mixed with polyvinyl chloride (PVC). The mixture of the two materials is added with tetrahydrofuran (THF), dried, and pressed so that it becomes a solid electrode which is a platinum powder composite (PPC). These electrodes are used as working electrodes for the analysis of capsaicin in food sauce. The results showed that the NaOH electrolyte was the best electrolyte for capsaicin analysis using platinum powder composite electrodes. The PPC electrode showed good test method validation results, namely recovery of 108.69%, LOD, and LOQ of 5.9 x 10^{-1} mM and 19.9 x 10^{-1} mM, respectively. The capsaicin concentration in the food sauce was 0.029 M in a 0.5 g sample.

Keywords: Capsaicin, composite, cyclic voltammetry, platinum powder, sensor

INTRODUCTION

The capsaicin compound in chili peppers can add a spicy sensation to food and kill food-borne bacteria because plant species such as chili peppers have antimicrobial properties, improve blood circulation, strengthen the heart, arteries, and nerves, prevent colds and fever, raise spirits in the body without narcotic effects, and make consumers release endogenous opioids and cause addiction (Xiang et al., 2022; Spence 2018). The spicy sensation in chilies is caused by the presence of capsaicin compounds ($C_{18}H_{27}NO_3$) and dihydrocapsaicin with a percentage of 69% and 22%, respectively (Nolden and Hayes 2017; Zavala et al., 2018). The chilies as raw material for spicy sauce and structure molecule of capsaicin is shown in **Figure 1.**

In addition to causing a spicy sensation, capsaicin also has biological activities such as anti-carcinogenic, antimicrobial, antioxidant, and analgesic properties so capsaicin appears as an alternative treatment for rhinitis, cancer, inflammation of the salivary glands, pain reliever, and weight loss (Ziyatdinova et al., 2020). Capsaicin is included in the Capsaicinoids class, which is a group of lipophilic alkaloids that cause a spicy taste in food. The capsaicin compound has high biological activity so it can be used as a medicine for several diseases. So far, capsaicin analysis uses expensive instruments such as HPLC and UV-Visible spectrophotometers. Because of that, a fast and accurate capsaicin analysis method is needed.

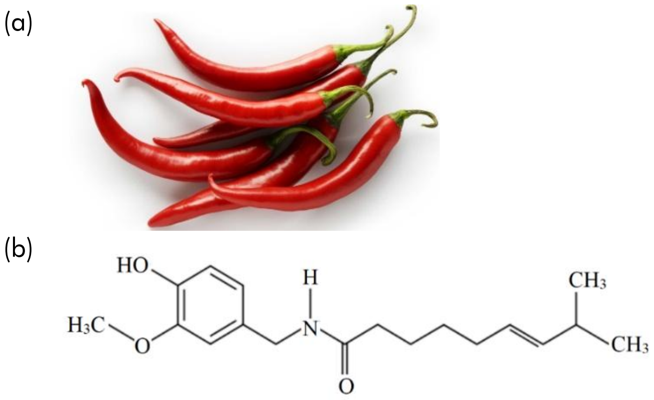


Figure 1. Chilies as raw material for spicy sauce (**a**) and structure molecule of capsaicin (**b**)

Chilies contain compounds from the capsaicinoids group. The compounds of this group give a spicy taste. This group contains 11 compounds with the main ingredients being capsaicin and dihydrocapsaicin. Capsaicinoids compounds have biological activities such as anticarcinogenic, antimicrobial, antioxidant, and analgesic. Chilies have long been used as a mixture for making food and drinks and spices which is widely consumed by humans. The use of chilies as a food additive continues to increase. The content of capsaicinoids in chili peppers depends on environmental conditions, soil quality, plant variety, growing season, geographic location, and degree of maturity. Therefore, a sensitive, selective, and fast method is needed to determine the capsaicin content in food (Buczkowska et al., 2013).

Capsaicin compounds contain phenolic groups which are easily oxidized so that they can be analyzed by electrochemical methods (Reyes-Escogido et al., 2011). The electrochemical method has high sensitivity, fast response, efficient, easy, and cheap, being an alternative method of capsaicin analysis besides the Scoville method which is considered subjective, and High-Performance Liquid Chromatography (HPLC) which is considered expensive and requires a long analysis time (Zavala et al., 2018). The choice of material for the working electrode in the electrochemical method is very important because it affects its sensitivity and selectivity. Several previous studies have developed various working electrode materials for electrochemical analysis of capsaicin, for example developing carbon paste electrodes (Díaz de León Zavala et al., 2017), carbon black (Deroco et al., 2020), graphene nanocomposite (Smith et al., 2019), and carbon dot-grafted electrodes (Supchocksoonthorn et al., 2021).

Pt metal is a stable metal and has excellent electrocatalytic performance. Pt metal contains d orbitals that are not filled with electrons, and has good electrochemical catalytic capabilities, especially for oxygen release reactions (Contreras et al., 2007). Platinum (Pt) has strong catalytic properties for various electrochemical reactions. Some organic compounds can be absorbed on the surface of the platinum electrode through hydrogen adsorption, and this is the first step in the electrochemical catalytic process. Pt metal is a good material to adsorb organic molecules and break their intermolecular bonds (Iwasita 2022). Most metal Pt electrodes are used to make fuel cells in acidic solutions such as H_2SO_4 and $HClO_4$ (Camara & Iwasita 2005; Lamy et al., 2004; Lamy et al., 2001; Liao et al., 2004). Pt metal in solid or powder form allows OH^- adsorption to occur on the electrode (Xu et al., 2005).

In this paper, the results of research on the preparation of platinum electrodes in the form of powder are presented. Powder-shaped platinum electrodes have the advantage of having a larger surface area than solid electrodes. Powder-shaped electrodes require an adhesive, namely polyvinyl chloride (PVC). The platinum powder is pressed with a pressure of 10 tons/cm^2 to make a working electrode. The prepared electrodes are applied for the analysis of capsaicin in food sauces.

EXPERIMENTAL SECTION

Reagents and Instruments

The materials used in this study were capsaicin ($C_{18}H_{27}NO_3 \geq 95$ %, Sigma Aldrich), PVC (Polyvinyl Chloride) powder, THF (Tetrahydrofuran), NaOH, H_2SO_4, KNO_3, KH_2PO_4, ethanol with pro analysis grade from Merck. The instrumentation used in this study was cyclic voltammetry (CV) from Metrohm, Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) of the Phenom Desktop ProXL.

Preparation of Platinum Powder Composite

Platinum metal powder with a purity of 99.99%, particle size < 2-micron as much as 95% is mixed with 5% polyvinylchloride (PVC), with a ratio of Pt powder to PVC (95:5), the total weight of the electrode is 1.5 g and stirred until it is homogeneous for 3 h. The mixture was then added with 4 mL of THF to dissolve the PVC and dried in an oven at 100 °C for 3 h. The obtained powder is put into a tray to be made in the form of pellets with a diameter of 1 cm and pressed with a pressure of 10 tons/cm^2. The resulting pellets are then made into electrodes. The schematic of the preparation of platinum powder composite (PPC) and detection of capsaicin using cyclic voltammetry can be seen in **Figure 2.**

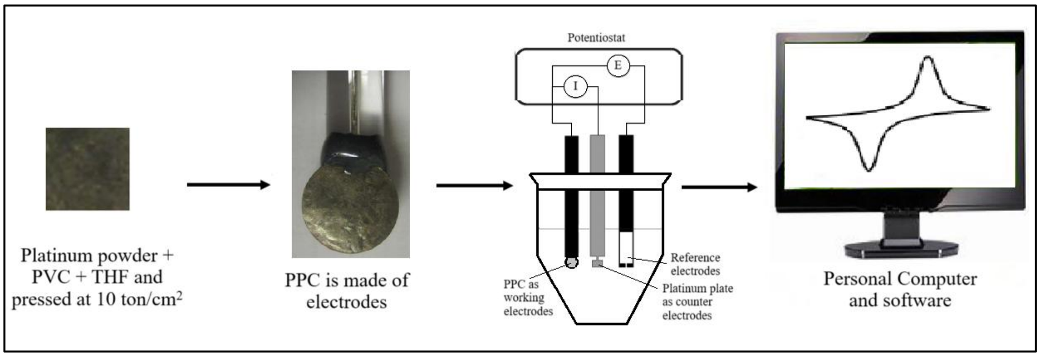


Figure 2. The schematic for the preparation of the PPC and detection of capsaicin using CV.

Application of PPC for Detection of Capsaicin in Food Sauce

Capsaicin electrochemical testing was carried out using cyclic voltammetry (CV). As much as 20 mL of 0.1 M capsaicin was put into the sample container. Amount of 5 mL of phosphate buffer electrolyte solution pH 7, NaOH, H_2SO_4, and KNO_3 electrolyte solutions were added to the sample container, tested under conditions at an upper potential of 1.0 V, lower potential of -1.0 V, start-stop potential -1.0 V, scan rate 0.1 V/s. PPC material as the working electrode, Ag/AgCl as the reference electrode, and solid Pt as the reference electrode. A total of 0.5 g of food sauce sample was dissolved in 50 mL ethanol, then filtered, and the filtrate was analyzed by CV.

RESULTS AND DISCUSSION

Characterization of PPC Material

Morphological analysis of electrodes PPC was carried out using SEM. **Figure 3a** shows the morphology of the electrode surface and **Figure 3b** crosses a section of the PPC electrode. The surface morphology of the PPC shows a porous or perforated surface. SEM results in the cross-section show that platinum metal is bound by PVC. The PPC has holes that increase the surface area and increase the contact of the analyte with the electrode.

The Effect of Electrolytes on Electrochemistry Responses of Capsaicin.

The electrolyte in cyclic voltammetry analysis has a very big influence. The electrolytes used in this study were phosphate buffer pH 7, NaOH, H_2SO_4, and KNO_3. The CV of the research results is shown in **Figure 4A-4E**. Figure 4A shows a cyclic voltammogram comparison between electrolytes alone (phosphate buffer pH 7) without capsaicin. **Figure 4(A)b** shows the pH 7 phosphate-buffered electrolyte with capsaicin. Cyclic voltammogram shows that capsaicin undergoes oxidation at a potential of 0.4 V. These oxidation and reduction potentials occur in the electrolyte of sulfate buffer, NaOH, H_2SO_4, and KNO_3. The highest capsaicin oxidation peak will be used to determine the best electrolyte. According to Daubinger et al., (2014) electrolytes greatly affect the electrochemical response of platinum electrodes. The electrochemical response can be seen from the difference in the cyclic voltammogram profile and the shift in the oxidation reduction potential. This difference is caused by the activity of protons and hydroxide ions. The platinum electrode is not reactive when using a salt electrolyte and a buffer phosphate (PBS) pH 7, because the number of protons and hydroxide ions is the same.

Figure 4E shows the cyclic voltammogram with electrolyte comparison. The highest capsaicin oxidation peak was seen on the cyclic voltammogram with NaOH electrolyte. NaOH electrolyte is used to determine the concentration of capsaicin in food sauces. The alkaline electrolyte has the advantage that the reaction rate is faster than in an acidic solution (Chen and Schell 2000). In NaOH solution, OH decomposition takes place at a lower potential than in acidic solutions. The electrocatalytic properties of alkaline solutions are due to the OH- anion. OH^- can be adsorbed on the surface of the platinum electrode is the first step in the electrocatalytic process. Photoelectron spectroscopy can be used for detection of OH adsorption will form platinum hydroxide species $Pt(OH)_4$ on the surface of platinum electrodes. Pt metal is a good material to adsorb organic molecules and break their intermolecular bonds (Iwasita 2022).

The Effect of Scan Rates on Electrochemistry Responses of Ferricyanide

Electrochemical response of PPC working electrode was evaluated using CV in the ferricyanide solution. The potential range used during testing was 0.01 to 0.65 V and the scan rate varied from 0.01 to 0.03 V/s. The reduction-oxidation reaction that occurred in the $[Fe(CN)_6]/K_4[Fe(CN)_6]$ solution was the reaction:

$[Fe(CN)_6]^{4-} \rightarrow [Fe(CN)_6]^{3-} + e-$ (oxidation)

$[Fe(CN)_6]^{3-} + e- \rightarrow [Fe(CN)_6]^{4-}$ (reduction)

Figure 5 shows the CV of PPC electrodes in ferricyanide solutions. Oxidation peaks were observed at potential 0.3 V and reduction peaks at 0.2 V. This showed the occurrence of a reversible redox reaction ferricyanide solutions in 0.1 M NaOH. The redox couple $[Fe(CN)_6]^{3-}/[Fe(CN)_6]^{4-}$ is a fast electrochemical reaction with a one electron transfer process. The relationship between the scan rate and the peak oxidation current showed that electron transfer from the ferricyanide solutions to the electrode surface was controlled by diffusion.

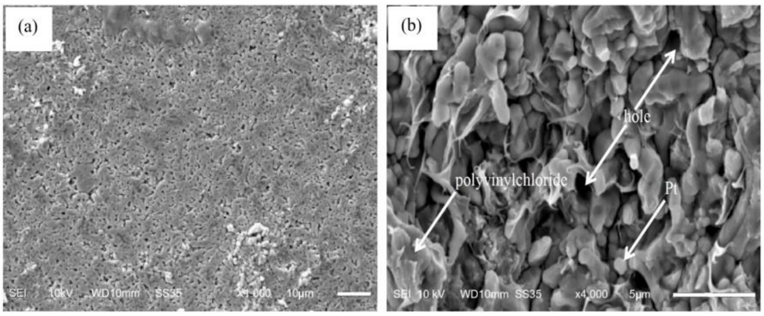


Figure 3. SEM image of the surface electrode with magnification 1000x (**a**) and cross-section with magnification 4000x (**b**)

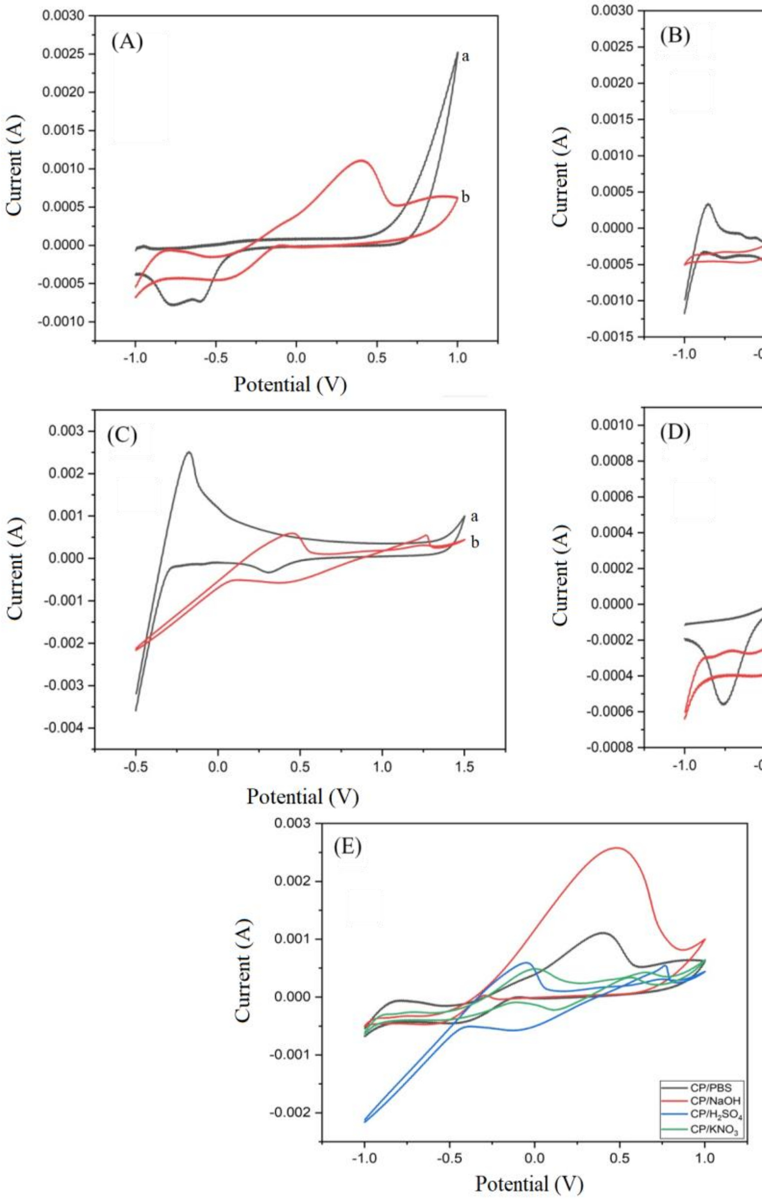


Figure 4. CV of (**A**) 20 mL distillate water + 5 mL buffer phosphate (PBS) pH 7 (a) capsaicin 20 mL 0.1 M + buffer phosphate (PBS) 5 mL pH 7(b); (**B**) 20 mL distillate water + NaOH 5 mL 0.1 M (a) capsaicin 20 mL 0.1 M + NaOH 5 mL 0.1 M (b); (**C**) distillate water 20 mL + H_2SO_4 5 mL 0.1 M (a) capsaicin 20 mL 0.1 M + H_2SO_4 5 mL 0.1 M (b) (**D**) distillate water 20 mL + KNO_3 5 mL 0.1 M (a) capsaicin 20 mL 0.1 M + KNO_3 5 mL 0.1 M (b) (E) comparison of capsaicin in various electrolyte solutions

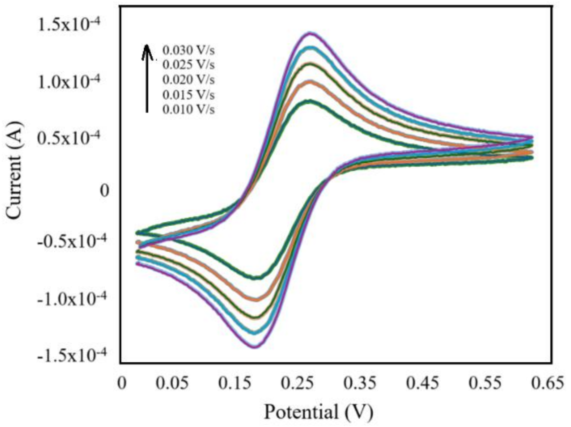


Figure 5. CV of PPC electrode in $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$ solution using 0.1M NaOH electrolyte

The Effect of Scan Rate to Electrochemistry Responses of Capsaicin.

Scan rate is a very important variable in CV analysis. Inappropriate scan rate selection will make it difficult to determine the reduction and oxidation peaks. Because of that scan rate optimization must be done before the sample is analyzed with CV. **Figure 6a** shows the CV performed at a low scan rate and **Figure 6b** CV performed at a high scan rate. CVs performed at a low scan rate produced sharp capsaicin oxidation peaks. The higher the can rate, the higher the oxidation peak and the potential shifts to the right. The higher the scan rate, the less visible the capsaicin oxidation peak. The selected scan rate for capsaicin analysis with PPC electrodes is 0.1 V/s. Variation of scan rate is very important to know the reaction mechanism on the surface of the electrode. The reaction mechanism on the surface of the electrode can be known from the value of R^2, which is the correlation between the scan rate and the peak oxidation current. **Figure 6c** shows the relationship curve between the scan rate and the peak oxidation current with an R^2 of 0.99. This shows the compound on the surface of the electrode is homogeneous (Khalafi et al. 2021).

Scan rate is an important parameter in cyclic voltammetry. Based on the Randles-Sevcik equation (at 25 °C) the equation applies:

$i_p = (2.69 \times 10^5)\, n^{3/2} A\, D^{1/2} C_i\, v^{1/2}$

where ip = current maximum in Amps, n = number of electrons transferred in the redox event (usually 1), A = electrode area in cm^2, D = diffusion coefficient in cm^2/s, C = concentration in mol/cm^3, v = scan rate in V/s. Based on this equation the peak current (i_p) will increase linearly with increasing scan rate (v). ip vs. plot $v^{1/2}$ is useful for electrochemical characterization of redox systems. The value of R^2 or linearity is an indication of the kinetics of electron transfer, or the result of chemical changes that occur because of electron transfer (Khalafi et al. 2021 and Elgrishi et al. 2018).

Electrochemical Testing of Samples Containing Capsaicin

Testing of samples containing capsaicin was carried out using food sauce samples dissolved in ethanol. This test was carried out using the standard addition method, where the composition of the test solution consisted of 5 mL of 0.1 M NaOH plus 5 mL of the sauce sample solution, and 3 mL of the capsaicin standard solution with various concentration variations, namely 0-0.005 M. The capsaicin standard solution was added in stages. and stirred with a magnetic stirrer before being measured by cyclic voltammetry. The results of sample measurements using cyclic voltammetry with a scan rate of 0.2 V/s are shown in **Figure 7a**. **Figure 7b** shows the standard addition curve of the relationship between the concentration of standard capsaicin added and the peak oxidation current. The linear regression equation obtained y=0.825x+0.0024 obtained the capsaicin concentration in food sauce of 0.02909 M in 0.5 g sample.

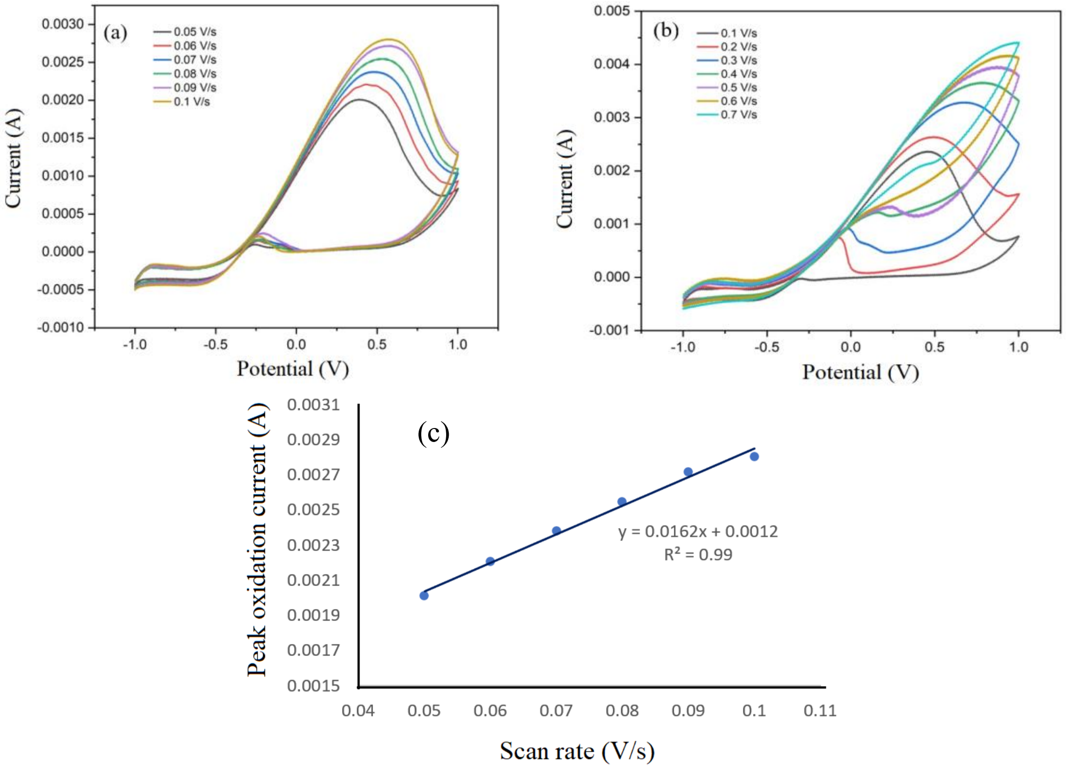


Figure 6. CV of 20 mL capsaicin 0.1 M + 5 mL NaOH 0.1 M with a scan rate variation of 0.05-0.1 V/s (**a**) and a scan rate variation of 0.1-0.7 V/s (**b**) and the correlation curve of scan rate with peak oxidation current (**c**)

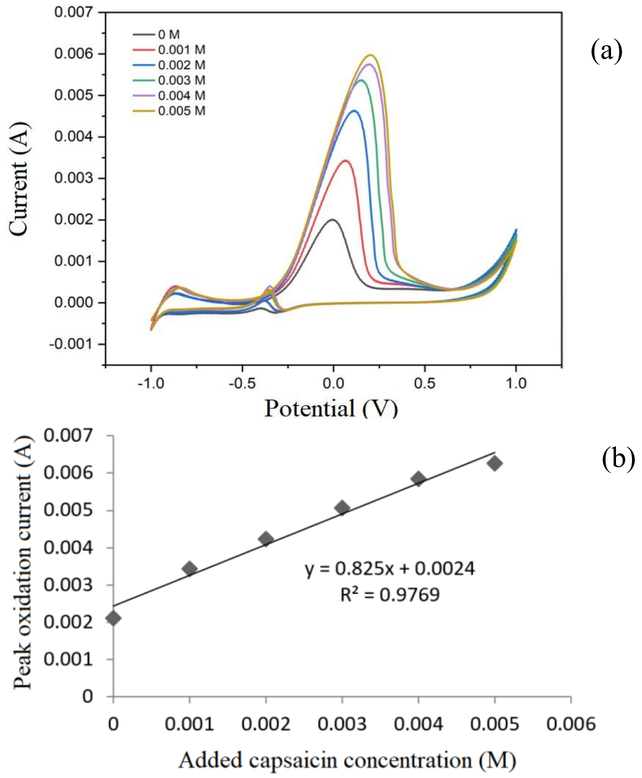


Figure 7. CV of 0.5 g food sauce sample dissolved in 50 mL ethanol, filtered, taken 5 mL + NaOH 5 mL 0.1 M + standard capsaicin 3 mL 0-0.005 M, scan rate 0.2 V/s (**a**) and addition curve standard (**b**)

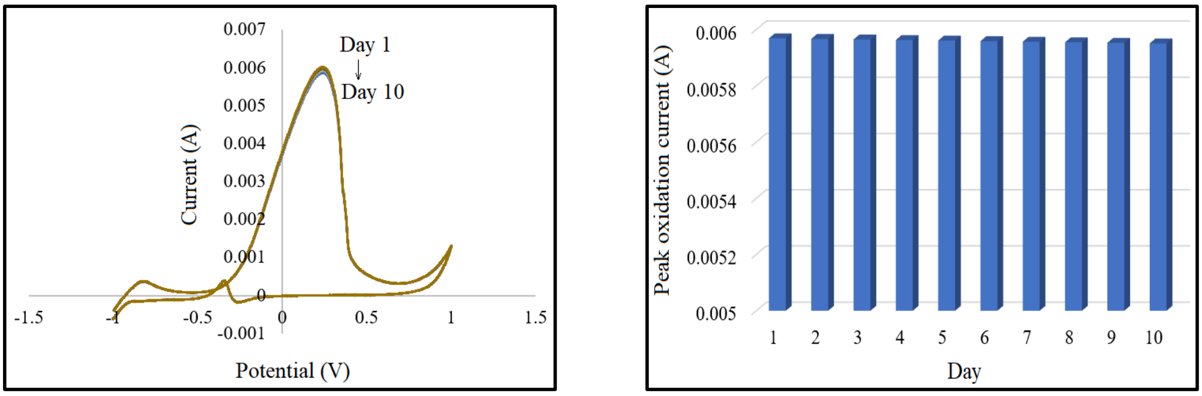
Data for determining recovery is shown in **Table 1**. Recovery is the suitability between the analysis results and acceptable reference values expressed as percent recovery. Percent recovery is a number indicating the amount of standard addition that can be re-identified by a method. The recovery value depends on the sample matrix, sample processing procedure, and concentration of impurities. The recovery value obtained is said to be good if it is in the range of 85-115% (AOAC 2000). Based on the results obtained, the average recovery value of 108.7% indicates good recovery. The test method has a LOD and LOQ of 5.9 x 10^{-1} and 19.9 x 10^{-1} mM.

Figure 8a shows the cyclic voltammogram using platinum powder composite for capsaicin analysis. The analysis was carried out on different days for 10 days. This analysis aims to test the stability of the electrode. **Figure 8b** shows that the PPC electrode has very good stability. PPC stability can be seen from the capsaicin oxidation peak at 0.2597 V. The capsaicin oxidation peak did not decrease without the electrode being used 10 times on different days. This excellent stability is due to two reasons: platinum is a stable metal and platinum is strongly bound to PVC. Electrode stability is needed in sensor development so that it has a long lifetime.

Based on the cyclic voltammogram in the NaOH electrolyte, the mechanism of the oxidation and reduction reactions on the surface of the platinum powder composite electrode produces one oxidation peak and one reduction peak. Capsaicin undergoes an oxidation reaction by releasing H^+ ions and two electrons, then undergoes a reduction reaction by capturing H^+ ions and two electrons. Capsaicin can occur oxidation and reduction reactions on the surface of the electrode. The oxidation reaction of capsaicin is according to the reaction mechanism in **Figure 9**. The oxidation reaction of capsaicin is almost the same as that of catechol compounds. Capsaicin in the first stage undergoes hydrolysis to o-benzoquinone. The next stage of capsaicin will undergo a reversible redox reaction (Kachoosangi et al., 2008).

Table 1. Data recovery determination test method

Added concentration of capsaicin (mM)	Concentration of capsaicin found (mM)	Recovery (%)
0.001	0.001259	125.9
0.002	0.002226	111.3
0.003	0.003234	107.8
0.004	0.004185	104.6
0.005	0.004692	93.8
	Average	108.7



(a) (b)

Figure 8. CV of the platinum powder composite in 5 mL NaOH 0.1 M + 3 mL standard capsaicin 0.005M scan rate 0.2 V/s used for 10 days (**a**) and peak oxidation current at potential 0.2597 V (**b**)

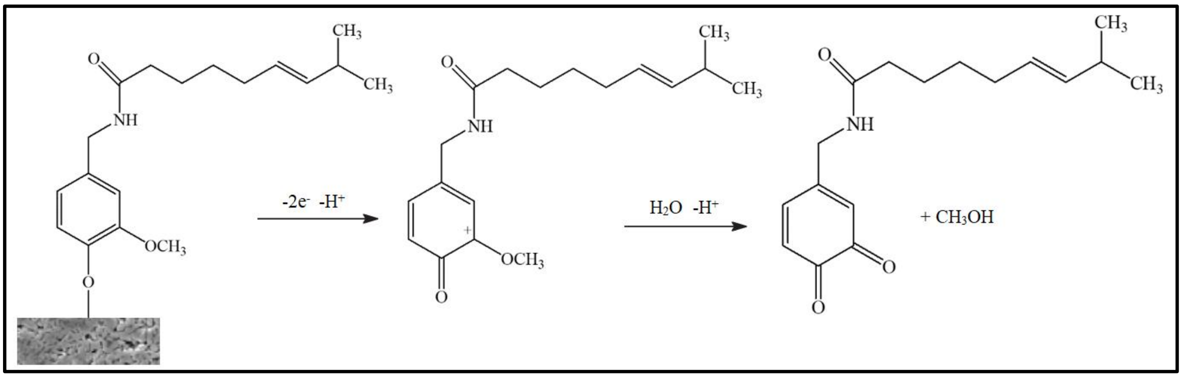


Figure 9. Mechanism for the electrochemical oxidation of capsaicin on platinum powder composite electrode

Table 2. Comparison of different types of electrodes for detecting capsaicin using cyclic voltammetry (CV)

Electrode	LOD (mM)	References
Glassy Carbon Electrodes (GCE)	6.4×10^{-3}	(Ziyatdinova & Budnikov 2014)
Multiwalled Carbon Nanotubes (MWNT)	1.9×10^{-3}	(Randviir et al., 2013)
Single Walled Carbon Nanotube (SWNT)	9.0×10^{-4}	(Randviir et al., 2013)
Carbon-Reduced Graphene Oxide (rGO-SPCE)	3.0×10^{-4}	(Jimenez et al., 2023)
Glassy Carbon Electrodes (GCEs) with Ruthenium Carbon Nanotubes (CNTs)	2.5×10^{-6}	(Baytak et al., 2017)
Platinum Powder Composite (PPC)	5.9×10^{-1}	This work

Electrode modifications for capsaicin detection have been conducted by many researchers. The purpose of the modification is to reduce the limit of detection (LOD). **Table 2** shows various types of electrode modifications using carbon. Research on electrode modifications using platinum metal is rarely carried out because of the high price. The weakness of the development of carbon-based electrodes is that the electrodes are unstable, and the advantage is that the LOD is very small. Electrode modifications using platinum such as PPC have a large LOD **(Table 2)** but the electrode is very stable. Based on the COD value, the PPC electrode is very suitable for determining capsaicin with high concentrations (≥ 5.9×10^{-1} mM) such as in samples of sauces, chili sauce, spicy food, and drink products.

CONCLUSIONS

Platinum powder composite (PPC) electrodes have been successfully prepared using a mixture of platinum powder with PVC and THF solvent. The prepared PPC has the characteristic of a perforated surface that is firmly bonded by PVC. PPC also showed a very good electrochemical response to detect capsaicin. The most suitable electrolyte for detecting capsaicin with PPC electrodes is in NaOH solution. The results of determining the respective recovery of 108.7% showed a good recovery. The test method has a LOD and LOQ of 5.9 x 10^{-1} and 19.9 x 10^{-1} mM. Further research on the manufacture of PPC material-based selective ion electrodes is urgently needed so that detecting capsaicin is easier and faster.

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