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Fabrication of ZnO Nanoparticles-Modified Carbon Paste Electrode for the Analysis of Nicotine Content in E-Cigarette Liquids by Cyclic Voltammetry

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Abstract

ZnO nanoparticles were used as composites on carbon paste working electrodes to enhance electrode performance in the analysis of nicotine content in e-cigarette liquids by voltammetry cyclic. The optimum composition and condition (pH and scan rate) were determined to identify the conditions that gave the best response. ZnO nanoparticles were synthesized using the sol-gel method and characterized by FTIR, XRD, and SEM. The determination of optimum composition and conditions was studied using cyclic voltammetry. The determination of nicotine content in e-cigarette liquids was analyzed by cyclic voltammetry. The electrode composition that gave the best response was 3:5:2 (carbon: nanoparticles ZnO: paraffin). The optimum conditions for nicotine determination by cyclic voltammetry were at pH 8 and a scan rate of 90 mVs^{-1}. The cyclic voltammetry's limit detection (LoD) using a ZnO nanoparticles-modified carbon paste electrode is 0.00678 mg/mL, and the percent recovery is 100.35%.

Keywords: Electrode, cyclic voltammetry, nicotine, ZnO nanoparticles.

INTRODUCTION

Electrodes are an important component in voltammetry. Voltammetry consists of working, reference, and auxiliary electrodes. The working electrode is where the analyte's reduction and oxidation reactions occur on the electrode surface. Carbon electrodes have a specific surface area, high chemical resistance, high electrical conductivity, more economical, non-poisonous, and high electrochemical stability (Esati & Cahyadi, 2021; Tumimomor, et al., 2017). Modification or composites to the working electrode can enhance electrode efficiency. Electrode modifications will enhance selectivity, sensitivity, and electrode stability (Apath et al., 2020). Nanoparticles provide a large surface area that can enhance electron transfer in electrochemical analysis. Nanoparticles enhance reduction-oxidation reversibility on the electrode surface (Falola, 2022) (Julita et al., 2022) (Istatik Badi'ah, 2021). Chikere et al. (2017) analyzed Gallic acid in red wine using ZnO nanoparticles-modified carbon paste electrode, resulting in limit detection of $1.86x10^{-7}$ $molL^{-1}$. It indicates that the modified electrode using ZnO nanoparticles produces high detection. ZnO is an inorganic compound that has high chemical stability, high electrochemical coefficient, wide radiation absorption range, and high photostability (Radzimska & Jesionowski, 2014).

Nicotine is an organic alkaloid compound produced from several plants, such as tobacco (Aji et al., 2015). Nicotine compounds are colourless to light yellow or brown, flammable, poisonous by inhalation and skin absorption, and produce toxic oxides when burned. Nicotine is an addictive compound that may cause dependence (Jahyadi, 2023). Nicotine induces stimulation and pleasure and relieves stress and anxiety. Smokers use nicotine to regulate arousal, control mood, increase concentration, reaction time, and performance (Benowitz, 2022) (Pagano et al., 2015). Nicotine is contained in the tobacco of conventional cigarettes and e-cigarettes in the form of extract. Nicotine content in commercial e-cigarette liquids is 0-40 mg/mL. 20 mg/mL nicotine is equivalent to one pack of conventional cigarettes containing 20 rods. Based on UK and Canadian government regulations, nicotine content in e-cigarette liquids is a maximum of 20 mg/mL (Action on Smoking and Health, 2023). Smokers assume that e-cigarettes are safer than conventional cigarettes. However, research by Bennani *et al.* (2020) indicates that e-cigarette liquids have differences in actual nicotine content with content written on the packaging of more than 20%. The difference may cause toxicity

and exceed the permitted nicotine content. Nicotine that exceeds the permitted content may cause cancer and bad conditions in the heart, reproductive system, lungs, kidneys, etc. (Mishra et al., 2015).

Determination of nicotine content in e-cigarette liquids has been conducted using High-Performance Liquid Chromatography (HPLC) (Palazzolo et al., 2019), Gas Chromatography-Mass Spectrometry (GC-MS), and Spectrophotometry UV-Vis (Elmanfe et al., 2022; Wei et al., 2018). Some of these methods require more expensive chemicals and cost. Voltammetry methods have been used to analyze nicotine in conventional cigarettes (Khoiriyah et al., 2016) and e-cigarettes that use boron diamond as working electrodes (Kowalcze & Jakubowska, 2020). A boron diamond electrode is sufficient to analyze nicotine with a low detection limit of 0.01 mg/L. However, these electrodes are quite expensive, so a voltammetry method using carbon paste electrodes modified with ZnO nanoparticles was developed. The development is based on the advantages of the carbon electrode and ZnO nanoparticles to obtain inexpensive, fast, selective, sensitive, and accurate electrodes for the analysis of nicotine content in e-cigarette liquids by cyclic voltammetry.

METHODOLOGY

Materials and Instrumentals

Zinc acetate dihydrate $Zn(CH_3COO)_2.2H_2O$ (Merck 108802, pure analysis, 95%), Sodium oxide NaOH (Merck 106498, pure analysis, ≥ 99%), and ethanol (Merck 100983, pure analysis, ≥ 99.9%) were used for synthesis ZnO nanoparticles. $Na_2HPO_4.2H_2O$ and $NaH_2PO_4.H_2O$ were used to make a phosphate buffer solution. The electrolytes solution used KCl with 100 times the standard concentration. Standard nicotine was used from Sigma Aldrich 36733-250 mg, pure analysis, and 98.9%. The solvent that was used for all the solution was distilled water.

ZnO nanoparticles were synthesized using the sol-gel method and characterized by FTIR, XRD, and SEM. The determination of optimum composition and condition was analyzed using cyclic voltammetry (797 VA computrace voltammeter, platinum as auxiliary electrode, Ag/AgCl as reference electrode). Nicotine content in e-cigarette liquids was determined by cyclic voltammetry with optimum composition and condition electrode. The result of nicotine content was compared with the result from the spectrophotometry UV-Vis (Shimadzu 1800). A comparison of both instruments was tested using an independent sample t-test to indicate that cyclic voltammetry using ZnO nanoparticles-modified carbon paste electrodes has no significant difference from that another instrument.

Synthesis of ZnO Nanoparticles

ZnO nanoparticles were synthesized using the sol-gel method. The first step of the sol-gel synthesis method is is dissolve 2 gr zinc acetate dihydrate in 15 mL of distilled water. Then, 4 gr sodium oxide was dissolved in 50 mL of distilled water. Both solutions were stirred using a magnetic stirrer at 70 °C temperature. The sodium oxide solution was added to the zinc acetate solution drop by drop to form a colloid. The mixed solution was titrated using 100 mL of ethanol. A colloid residue of ZnO nanoparticles was obtained. Then, the colloid residue was washed with distilled water and ethanol in turns with a centrifuge of 5000 rpm for 15 minutes. The precipitate was dried in an oven at 80 °C temperature for 15 minutes and calcined at 400 °C for 30 minutes.

Fabrication ZnO Nanoparticles-Modified Carbon Paste Electrode

The electrode is made of copper wires and is given a 1 cm long insulator pipe at the bottom edge. Copper wires with a length of 12 cm are cleaned 1.5 cm at the upper edge and 0.5 cm at the lower edge and sanded. Composite materials are weighed in the ratio of carbon: ZnO nanoparticles: paraffin, which is 3:2:5, 3:3:4, 3:4:3, and 3:5:2. The material is stirred until homogeneous and inserted into the insulator pipe until it is dense.

Determination Optimum Composition and Condition

The optimum ZnO nanoparticles-modified carbon paste electrode was determined using 4 electrodes that have been made before with the lowest variable measurement conditions. The determination of the optimum pH was conducted by the optimum electrode with phosphate buffer variables of pH 7, 8, 9, 10, and 11. The determination of the optimum scan rate was conducted by the optimum electrode with optimum pH and a variable scan rate of 50, 60, 70, 80, and 90 mVs^{-1}. All measurements were conducted over a potential range of -1.5 V to +1.5 V. The measured solution is a mixture of the highest concentration nicotine standard solution, buffer solution, and KCl electrolyte solution. The result was analyzed using the software Origin 2024.

Determination of Nicotine Content in e-cigarette Liquids

Nicotine 98.9%s dissolved in distilled water to produce a nicotine standard solution with a concentration of 0.01, 0.02, 0.04, 0.08, 0.16, 0.32, and

0.5 mg/mL. Sample solution is produced from 3 different brands of e-cigarette liquids that were dissolved in distilled water in a ratio of 1:20 (v/v). Standard and sample solutions were analyzed using cyclic voltammetry with optimum electrode composition and condition.

The sample solution for Spectrophotometry UV-Vis was made by dissolving e-cigarette liquids in distilled water in a ratio of 1:50 (v/v). The standard and sample solutions were analyzed using spectrophotometry UV-Vis at 267 nm wavelength. The result of standard solution measurements is plotted into a standard linear curve to determine nicotine concentration in the sample.

RESULTS AND DISCUSSION

ZnO Nanoparticles Characterization

The ZnO nanoparticles were characterized using FTIR, XRD, and SEM (Figure 1). FTIR is used to determine the functional groups contained in compounds. ZnO Nanoparticles were analyzed at wavenumber 400-4000 cm^{-1}. The FTIR spectrum of ZnO nanoparticles (Figure 1a) shows several functional groups (Table 1). The peak at wavenumber 554.62 cm^{-1} is identical to the Zn-O bond area (Chikere et al., 2019; Patel et al., 2022; Srujana & Bhagat, 2022).

Table 1. Functional groups of vibrations at wavenumber FTIR spectrum

Wavenumber (cm^{-1})	Functional Groups
554.62	Zn-O
880.44	C-H
910.56	C-O
1370.53	C-O
1504.69	C=C
2347.97	O=C=O
3501.91	O-H

(Pavia et al., 2001; Srujana & Bhagat, 2022)

XRD is a physical characterization instrument used to determine the distance between layers or rows of atoms, the orientation of a single crystallite, the crystallite structure of unknown compounds, crystallite size, shape, and internal pressure in small crystallite areas (Bunaciu, Udriştioiu, & Aboul-Enein, 2015). ZnO nanoparticles were analyzed at a 2θ angle range of 10° to 90°. The diffraction pattern of the ZnO nanoparticles (Figure 1b) shows the presence of a sharp diffraction peak at the position in Table 2, which is by previous research (Hasnidawani et al., 2016; Musdalifa & Purnama Muh, 2019; Srujana & Bhagat, 2022; Vishwakarma, 2020). The diffraction pattern is in accordance with International Center of Diffraction Data (ICDD) code 00-036-1451, which confirms that the compound analyzed is a ZnO nanoparticles compound in the form of hexagonal wurzite. ZnO crystallite sizes are generated by the Debye-Scherrer equation. The resulting average crystallite size is 32.96 nm. Debye-Scherrer equation:

$$D = \frac{0{,}89\lambda}{\beta \cos \theta} \quad (1)$$

(Mulyati & Panjaitan, 2021)

Where D = diameter, λ = X-ray beam's wavelength (1.54 Å), β = FWHM Left in radians and θ = Bragg's diffraction angle in radians.

Table 2. Peak position and size of ZnO nanoparticles crystallite based on the Debye-Scherrer equation

Position [°2Th]	FWHM Left [°2Th]	[h, k, l]	Crystallite size (nm)
31.77	0.168	1 0 0	48.61
34.44	0.216	0 0 2	38.07
36.23	0.144	1 0 1	57.40
47.52	0.240	1 0 2	35.76
56.58	0.432	1 1 0	20.65
62.91	0.432	1 0 3	21.31
66.45	0.432	2 0 0	21.73
67.93	0.240	1 1 2	39.46
69.16	0.240	2 0 1	39.75
72.52	0.288	0 0 4	33.84
76.87	0.480	2 0 2	30.89
81.32	0.576	1 0 4	17.97
Average			32.96

SEM is used to determine the morphological structure of the synthesized compound. The morphological structure of ZnO nanoparticles is shown in Figures 1c and 1d. The morphological structure of ZnO nanoparticle is spherical with heterogeneous size distribution. The resulting particle size is 74.26 nm. This indicates that the synthesized ZnO compound is nanoparticle-size because it is less than 100 nm. The sol-gel method is one of the bottom-up methods (synthesis by changing small particles into larger particles). Ion Zn^{2+} from precursors will change into ZnO nanoparticle size.

Optimum Electrode Composition

The composition variable voltammogram (Figure 2) shows that the electrode composition 3:5:2 (carbon: ZnO nanoparticles: paraffin) is the electrode with the optimum composition, providing the sharpest and highest current peak. The peak current of

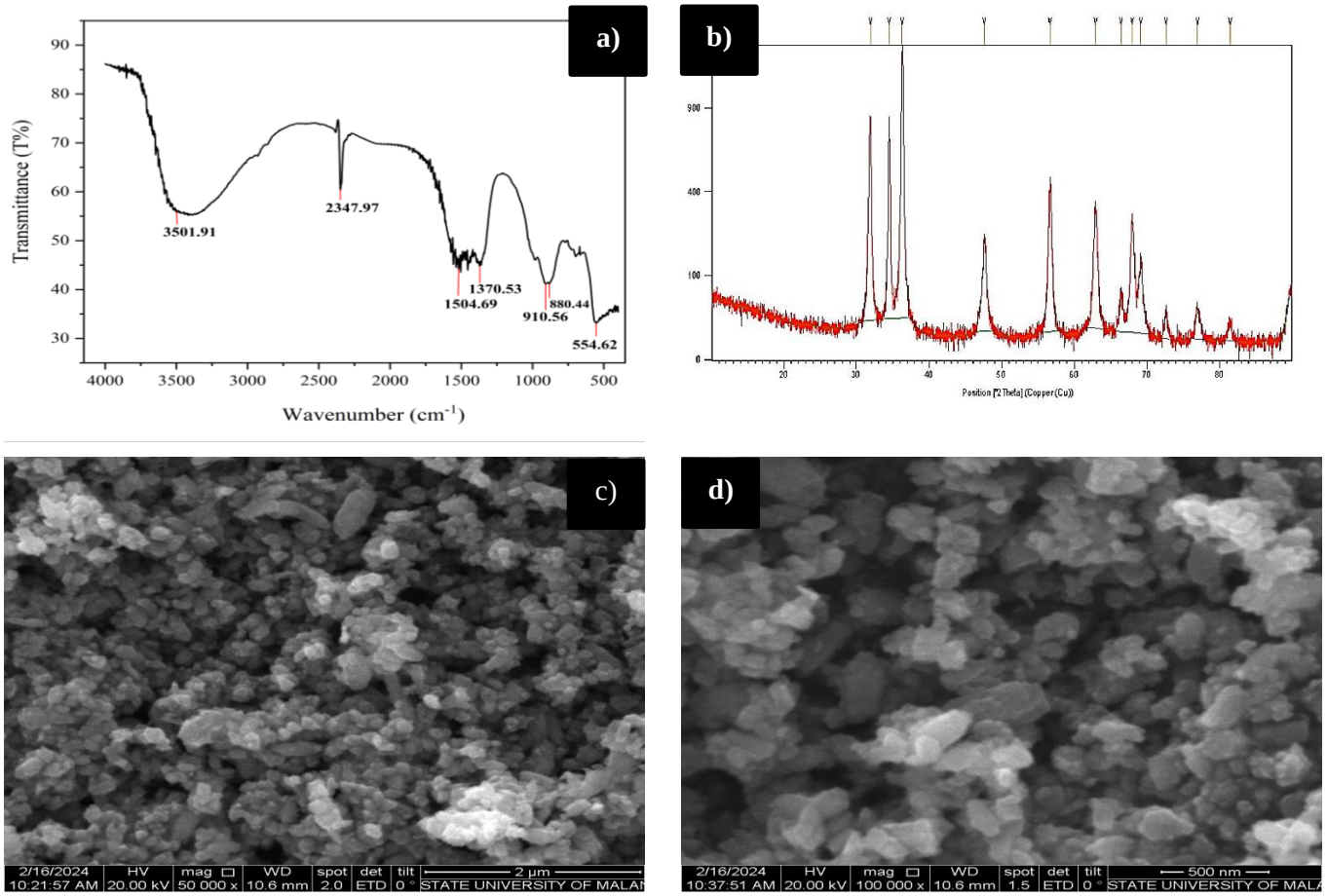

Figure 1. Characterization of ZnO nanoparticles (a) FTIR Spectrum of ZnO nanoparticles (b) Diffraction pattern of ZnO nanoparticles (c) SEM image of ZnO nanoparticles (50.000x magnifications) (d) SEM image of ZnO nanoparticles (100.000x magnification)

determining optimum electrode composition is shown in Table 3. The more nanoparticles, the greater the surface area of the electrode and the greater the adsorption carried out by the electrode on electron transfer (Shetti et al., 2019). The composition of the electrode is determined based on the phase balance of the 2 components of the electrode material.

Table 3. Peak current (IpA) of determining optimum electrode composition

Composition	IpA (A)
3:2:5	6.51×10^{-4}
3:3:4	3.30×10^{-4}
3:4:3	3.92×10^{-4}
3:5:2	8.34×10^{-4}

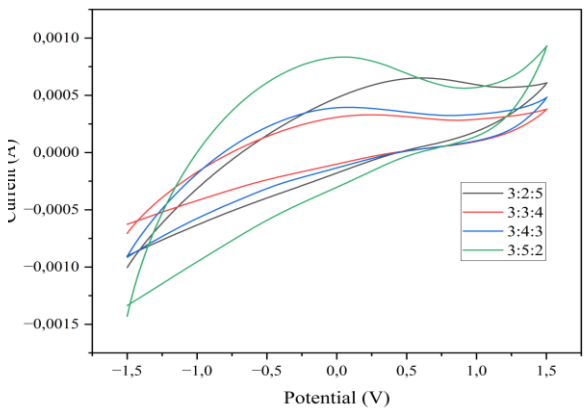

Figure 2. Voltammogram of determining optimum electrode composition

The voltammogram (Figure 2) showed that a ZnO nanoparticles-modified carbon paste electrode produced a peak current for nicotine analysis. This shows that the electrode is selective in separating or analyzing nicotine. The peak current that appears is the oxidation peak (anodic), but the reduction peak (cathodic) does not appear. It shows that the reaction is irreversible. Nicotine or 3-(1-methyl-2-pyrrolidinyl) pyridine undergoes oxidation due to the potential provided by voltammetry. Nicotine, which undergoes oxidation, will release electrons that will bind and be adsorbed by the electrode surface. The reaction will produce a current, which is then translated by

voltammetry to form a peak current for the oxidation of the nicotine compound.

Optimum pH Condition

The optimum electrode composition is then used to determine the optimum pH. This determination aims to prevent changes in stability on the electrode surface due to the ongoing reaction. The pH was 7, 8, 9, 10, and 11. Previous research (Kowalcze & Jakubowska, 2020) stated that the optimum pH for nicotine analysis was 7.5. According to Benowitz (2022), nicotine has an alkaline pH, namely 8. The voltammogram for determining the optimum pH (Figure 3) and the resulting peak current (Table 4) shows that pH 8 has the highest peak current.

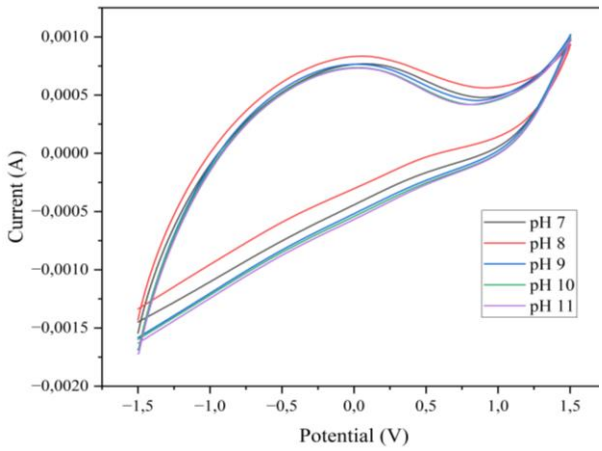

Figure 3. Voltammogram of determining optimum pH

Table 4. Peak current (IpA) of determining optimum pH

pH	IpA (A)
7	7.69 x 10^{-4}
8	8.34 x 10^{-4}
9	7.64 x 10^{-4}
10	7.35 x 10^{-4}
11	7.33 x 10^{-4}

Nicotine undergoes oxidation and causes electron transfer to the electrode surface. The reaction of nicotine oxidation and electrode reduction causes a change in pH so that electrode stability is maintained by adding a pH buffer. The presence of proton and electron from pH will affect the electron transfer on the electrode surface. Optimum pH will provide a higher peak current and maintain electron transfer stability. A higher peak current indicates increased electrode surface, electron transfer, and conductivity (Wijeratne et al., 2018).

Optimum Scan Rate Condition

The optimum electrode composition and pH are used to determine the optimum scan rate. Scan rate determination was carried out at 50, 60, 70, 80, and 90 mVs^{-1}. The Voltammogram of determining the optimum scan rate (Figure 4) and peak current (Table 5) shows that a scan rate of 90 mVs^{-1} is the optimum scan rate that produces the highest peak current. Faster scan rate, faster electron transfer that occurs, and a higher peak current.

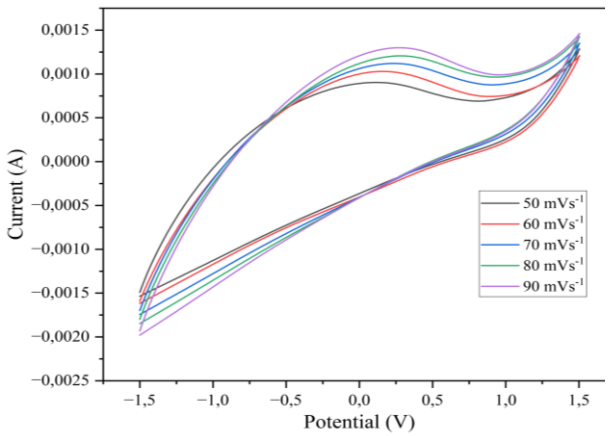

Figure 4. Voltammogram of determining optimum scan rate

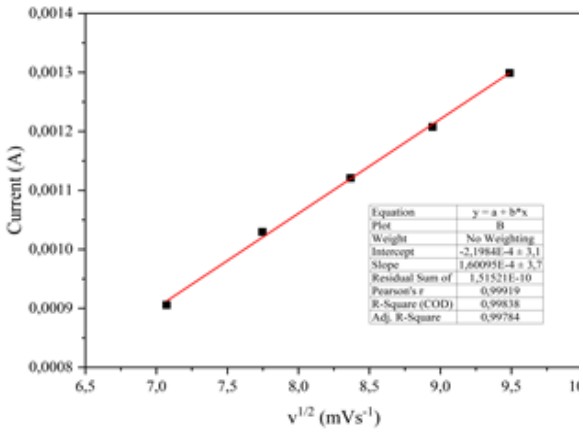

Figure 5. Linear curve between the square root of scan rate and IpA

Table 5. Peak current (IpA) of determining optimum scan rate

Scan Rate (mVs^{-1})	IpA (A)
50	9.05 x 10^{-4}
60	1.03 x 10^{-3}
70	1.12 x 10^{-3}
80	1.20 x 10^{-3}
90	1.29 x 10^{-3}

Figure 5 shows that the square root of the scan rate is directly proportional to the peak current. It indicates that the diffusion process controls the electrochemical process that occurs. Scan rates that are too fast and too slow will cause the reaction to be more irreversible and will remove the active layer on the electrode surface (Zhu et al., 2020).

Analytical Performance

The ZnO nanoparticles-modified carbon paste electrode in cyclic voltammetry is optimized based on selectivity, linearity, limit of detection (LoD), and accuracy. The electrode has been proven selective, as indicated by the appearance of an oxidation peak current. There is a potential shift at peak current. This phenomenon occurs in the determination of organic compounds (Chikere et al., 2019). It is due to electron transfer resistance between the oxidized compound and the active surface of the electrode.

The relationship between increasing concentration and IpA value shows a linear curve with R^2 = 0.99988 (Figure 6). The linear curve is a calibration curve with a linear equation of y = 0.0017x + 0.0004473. The LoD of this research was 0.00678 mg/mL, obtained from the following equation.

$$\text{LoD} = \frac{\mathbf{3x\ s\ (y/x)}}{\mathbf{slope}} \quad (2)$$

Where s(y/x) = standard deviation of the blank, slope = slope of the calibration curve

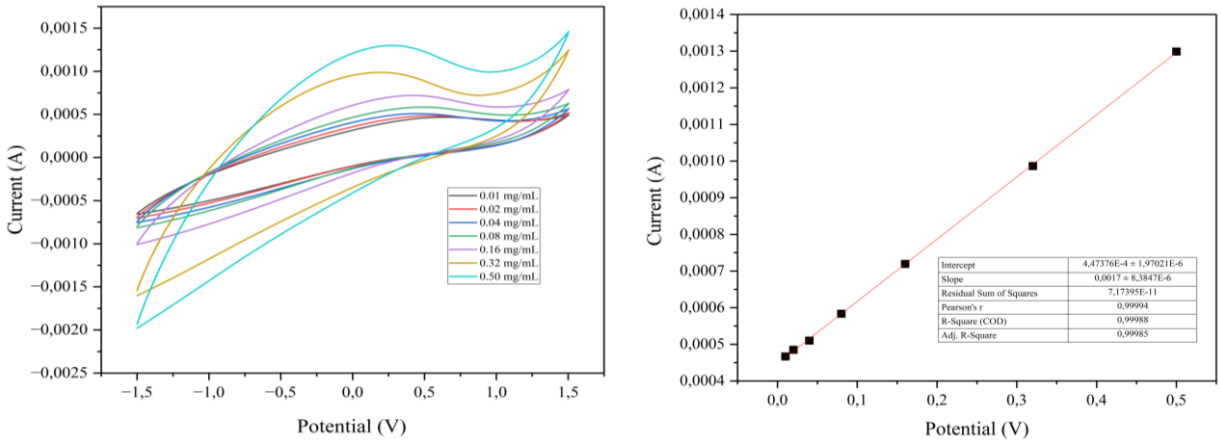

Figure 6. (a) Voltammogram of different concentrations (b) Calibration curve

Table 6. Percent recovery to determine the accuracy of the method

Actual conc. (mg/mL)	IpA (A)	Voltammetry conc. (mg/mL)	Recovery (%)
0.16	7.194 x 10^{-4}	0.160	100.00
	7.099 x 10^{-4}	0.154	96.52
	7.105 x 10^{-4}	0.155	96.74
	7.292 x 10^{-4}	0.166	103.61
	7.298 x 10^{-4}	0.166	103.84
	7.320 x 10^{-4}	0.167	104.64
	7.168 x 10^{-4}	0.158	99.03
	7.174 x 10^{-4}	0.159	99.26
	7.130 x 10^{-4}	0.156	97.66
	7.255 x 10^{-4}	0.164	102.24
		Average	100.35

A standard nicotine solution with a concentration of 0.16 mg/mL was analyzed by cyclic voltammetry 10 times to determine the method's accuracy. Table 6 shows the percent recovery value is 100.35%, which means the method can analyze 100.35% accurately from the actual value. It is in accordance with the permitted percent recovery, namely 95-105%, for a concentration of 0.1% (Hizbullah & Setiarso, 2024).

Analytical Application

Three different brands of e-cigarette liquids produced in Indonesia (Table 7) were analyzed using cyclic voltammetry with the optimum electrode composition and condition. The voltammogram (Figure 7) shows an oxidation peak current in the potential 0.05V, proving nicotine's presence. The results show that ZnO nanoparticles-modified carbon paste electrode has proven to be used for analyzing nicotine content in e-cigarette liquids using cyclic voltammetry.

Table 7. Nicotine content in e-cigarette liquids

Brand	IpA (A)	Average IpA (A)	Nicotine conc. (mg/mL)
A1	9.166×10^{-4}		
A2	9.058×10^{-4}	9.033×10^{-4}	5.36
A3	8.877×10^{-4}		
E1	8.324×10^{-4}		
E2	8.300×10^{-4}	8.288×10^{-4}	4.48
E3	8.240×10^{-4}		
M1	1.037×10^{-3}		
M2	1.037×10^{-3}	1.035×10^{-3}	6.91
M3	1.031×10^{-3}		

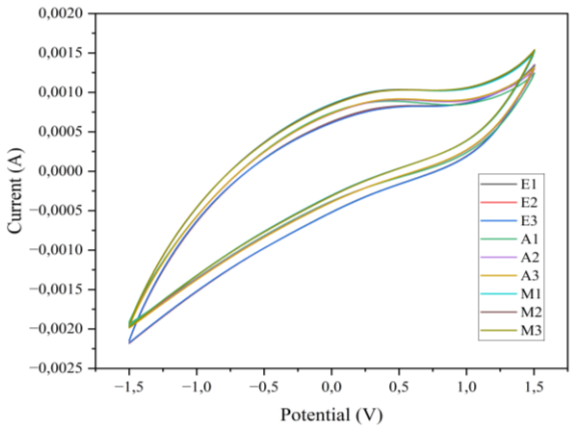

Figure 7. Voltammogram of determining nicotine content in e-cigarette liquids

CONCLUSION

ZnO nanoparticles that have been synthesized and characterized are used to make ZnO nanoparticles modified carbon paste electrodes. The optimum electrode composition is 3:5:2 (carbon: ZnO nanoparticles: paraffin) with an optimum condition of pH 8 and a scan rate of 90 mVs^{-1}. Nicotine concentration calibration curve 0.01 to 0.5 mg/mL with R^2= 0.99988 and LoD = 0.00678 mg/mL. The analytical method was proven by determining nicotine content in 3 different e-cigarette liquid brands. The electrode is selective, sensitive, and accurate for determining nicotine in e-cigarette liquids.

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