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FABRICATION OF CARBON PASTA ELEKTRODA COMPOSITION MODIFIED WITH NANOBENTONITE AND NANO TiO_2 FOR NIACINAMIDE DETECTION SENSOR

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Abstract: Niacinamide is the amide form of vitamin B3, which is usually analyzed for concentration using UV-Vis Spectrophotometry, HPLC, and TLC. The development of niacinamide analysis methods using cyclic voltammetry has not been widely studied. The use of working electrodes in cyclic voltammetry is usually carbon paste electrodes. However, these electrodes have disadvantages, relatively low sensitivity and slower electron transfer kinetics, so it is necessary to research working electrode modification to increase electrode sensitivity. This study aims to determine the effect of the addition of nanobentonite and nano TiO_2 on carbon paste electrodes in improving electron transfer and electrode sensitivity in the analysis of niacinamide by cyclic voltammetry. Variations in electrode composition were conducted to determine the optimum composition in measuring niacinamide solution and the optimum pH in measuring niacinamide. Nanobentonite obtained from the synthesis using the sonochemical method produces an average particle size of 46.9 nm, and the composition of carbon, paraffin, nanobentonite, and nano TiO_2 electrodes with a variation of 3:2:3:2 b/v has the highest current peak. The better the conductivity of the working electrode, the greater the maximum current peak produced due to the easy transfer of electrons for the reduction and oxidation reaction process. Using a buffer solution to adjust the pH of the niacinamide solution affects the analysis process, as evidenced by the IpA value at pH 7. The effect of pH variation also affects the stability of existing ions. So that the resulting current is higher, the higher the peak current value produced indicates, the more sensitive the electrode is due to high electron transfer. It maximizes the analysis of the concentration of the test solution because the measured concentration is linear with the measured current.

Keywords: *Niacinamide, Electrode, Voltametry Cyclic, Nanobentonit, Nano* TiO_2

INTRODUCTION

Niacinamide is an amide form of vitamin B3, which is a by-product of tryptophan metabolism and also a vital component of the metabolic pathway involved in the production of nicotinamide adenine dinucleotide (NAD^+) [1]. Niacinamide has anti-aging, anti-acne, antipruritic, antimicrobial [2], and anti-inflammatory functions [3]. In addition, it can also brighten the skin [4], moisturize dry skin, and reduce sebum excretion [5]. Topical use of niacinamide at a dose of 2% can reduce sebum and brighten the skin, while 5% can reduce acne [6].

Niacinamide concentrations are general in skincare and are quantitatively measured using Spectrophotometry UV-Vis, High-Performance Liquid Chromatography (HPLC), and Thin-layer Chromatography (TLC) methods. However, the disadvantages of this method are that its use requires a reasonably high cost, sample preparation is quite complicated, and the analysis result time is not fast enough [7]. Therefore, it is necessary to develop methods that are more effective, efficient, low cost, easy sample preparation, and have high sensitivity in detecting compounds.

Electrochemical methods such as voltammetry are practical and efficient methods that can analyze niacinamide concentrations. The principle of this method is based on measuring the current intensity during electrolysis triggered by potential variables within a particular time [8]. One of the widely used voltammetry methods is cyclic voltammetry. The advantages of cyclic voltammetry are that it can analyze analytes with high concentrations, has stability in oxidizing and reducing in a high potential range, and does not cause damage to analytes or supporting electrolytes [9].

Cyclic voltammetry has 3 electrodes in the analysis process, including the auxiliary, comparison, and working electrodes. The working electrode is an essential part of analyzing the test solution because it is where the reduction and oxidation reactions occur. So that whether or not the constituent material or maker of the working electrode determines the analysis process. In general, working electrodes are made using carbon because the properties of carbon are inert, have high conductivity, and have easy application on the surface. However, behind these advantages, carbon's weaknesses include relatively low sensitivity, slower electron transfer kinetics, and lower stability in various solutions [10]. These problems with carbon can be overcome by modifying the addition of suitable materials to improve the performance of the working electrode. One innovation that is quite widely developed is the addition of nanoparticles to the carbon paste mixture for working electrodes. Nanoparticles have a larger surface area compared to micro-sized powder particles so that they can increase the absorption function on the electrode surface and produce a larger potential current. Many nanoparticles are used as modifications to carbon paste electrodes, including nickel dioxide (NiO) nanoparticles, which are good at oxidizing transition metals [11], and ZnO and SiO_2 nanoparticles, which produce good analytical results in the gallic acid test, in addition, there are also CuO, MnO_2, and Fe_2O_3 nanoparticles which can improve the ability of oxidation-reduction reactions that occur during analysis [12]. Among the many nanoparticles that can be used, this study will use carbon paste electrode modification with nanobentonite, which can increase the adsorption ability of the adsorbent [13] and nano TiO_2 with good biocompatibility, large surface area, good dispersion, and fast electron transfer capability [14]. The addition of 2 different nanoparticles is expected to produce new properties that can improve electron transfer. This study obtained nanobentonite by synthesizing bentonite bulk using the sonochemical method, while nano TiO_2 was purchased directly.

This study aims to determine the effect of adding nanobentonite and nano TiO_2 to carbon paste electrodes to detect niacinamide by cyclic voltammetry as an alternative method of measuring niacinamide concentrations. In the form of increasing the electrode's sensitivity to higher niacinamide, variations in electrode composition were carried out to determine the optimum composition in measuring niacinamide solution and the optimum pH in measuring niacinamide solution.

RESEARCH METHODS

Materials and Tools

The materials used in this study are niacinamide p.a. (merck), bentonite powder, 96% ethanol, 5M HCl, carbon powder, paraffin, Na_2HPO_4, NaH_2PO_4, KCl (merck), Nano TiO_2 anastase 99% (merck), distilled water, copper wires, ballpoint pen contents.

In comparison, the tools used are beakers, measuring cups, measuring flasks, porcelain cups, mortar pestle, measuring pipettes, 300 mesh sieve, magnetic stirrer, analytical balance, oven, pH meter, ultrasonic bath Elmasonic S 30/H, cyclic voltammeter 797 VA Computrace, UV-Vis spectrophotometer Shimadzu 1800, XRD X'Pert PRO PANalytical, FTIR Thermo Scientific Nicolet iS10.

Nanobentonite Synthesize

The nanobentonite used in this study was prepared using bulk bentonite synthesized using the sonochemical method. Twenty grams of bentonite powder was put in a 250 mL beaker and added with 100 ml of 5M HCl, then heated and stirred with a magnetic stirrer at 70°C for 4 hours at 400 rpm to activate bentonite. The solution was separated between the filtrate and residue, then washed with hot distilled water repeatedly until the pH was neutral. After pH was neutral, bentonite was oven for 2 hours at 200°C. After drying, it was crushed with a mortar and sieved with a 300 mesh sieve. Bentonite powder was added with 96% ethanol as much as 50 mL. The mixture of bentonite and ethanol was put into an ultrasonic bath to be sonicated for 4 hours. After sonicating, bentonite was baked again at 100°C for 30 minutes. The dried bentonite was crushed with a mortar and oven at 100°C for 5 hours. The finished synthesized nanobentonite was characterized by XRD and FTIR.

Preparation of Modified Carbon Paste Working Electrode

The working electrode is made of copper wires with a length of 12 cm, which at the lower end of the cable is peeled with a length of 0.5 cm while at the other end is peeled with a length of 1.5 cm. A cylindrical tube is attached at the end of the cable that is peeled with a length of 0.5 cm. The cylindrical tube is a nanobentonite and nano TiO_2 composite carbon paste container.

The contents of the working electrode were made of a mixture of carbon, paraffin-synthesized nanobentonite, and nano TiO_2. These materials were weighed in various ratios of 3:3:2:2, 3:2:3:2, 3:2:2:3, 3:3:3:1, 3:3:1:3 b/v. The weighed materials were crushed with a mortar until homogeneous. The electrode content mixture was pressed into the prepared copper wires until solid. The modified electrode was allowed to stand for 24 hours at room temperature before being used in the analysis.

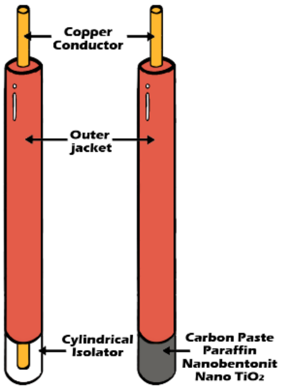

Figure 1. Nanobentonite and Nano TiO_2 Modified Carbon Paste Working Electrode

Determination of Optimum Electrode Composition

Working electrodes with various compositions were tested with a cyclic voltammeter using a 5 ppm niacinamide solution dissolved in 10 mL of 500 ppm KCl and phosphate buffer solution pH 6. Measurements were carried out at a potential of -0.8 V to 1.8 V, with a deposition time of 10 seconds and a scan rate of 0.2 V/s. The voltammograms obtained in each measurement of the modified electrode were compared to determine the highest current peak.

Determination of Optimum pH

Determination of the optimum pH is measured using electrodes with the optimum composition that has been measured previously. The test solution used a 5 ppm niacinamide solution dissolved in 10 mL of 500 ppm KCl and phosphate buffer solution with pH variations of 5, 6, 7, and 8. Measurements were made at a potential of -0.8 V to 1.8 V with a deposition time of 10 seconds and a scan rate of 0.2 V/s. The voltammogram results obtained in each pH solution variation are compared to determine the highest current peak.

RESULTS AND DISCUSSION

Nanobentonite Synthesis and Characterisation

The sonochemical synthesizes nanoparticles using an ultrasonic bath with high-frequency ultrasonic waves [15]. Ultrasonic waves can be synthesized because they break chemical bonds without high temperatures due to cavitation processes that can produce extreme temperatures and pressures of 5300 °C and 1000 atmospheres. The resulting high pressures and temperatures induce chemical reactions that impact surface morphology composition, and produce nanostructures [16].

Bentonite is activated using HCl to increase its adsorption power because HCl will react with impurities such as Al, Fe, and Mg that cover the pores of bentonite. In addition, HCl can react with Al_2O_3 to form $AlCl_3$, increasing the ratio of the amount of SiO compared to Al_2O_3 [17].

The reaction that occurs during the bentonite activation process is as follows:

$$Al^{3+}{}_{(aq)} + 3HCl_{(aq)} \rightarrow AlCl_{3(aq)} + 3H^+$$
$$Mg^{2+}{}_{(aq)} + 2HCl_{(aq)} \rightarrow MgCl_{2(aq)} + 2H^+$$
$$Fe^{3+}{}_{(aq)} + 3HCl_{(aq)} \rightarrow FeCl_{3(aq)} + 3H^+$$
$$Al_2O_{3(aq)} + 6HCl_{(aq)} \rightarrow 2AlCl_{3(aq)} + 3H_2O$$

The synthesized bentonite has a net weight of 15.8452 grams with a %yield of 79.226%. The synthesized nanobentonite was tested for chemical characterization using FTIR and physical characterization using XRD to ensure that the bentonite particle size has reached nano size.

FTIR Test Results

Chemical characterization using FTIR was used to determine the functional groups of nanobentonite. Raw bentonite has cations with weak bonds between layers, so cations are easily exchanged and cause structure swelling during the bentonite activation process using HCl. The FTIR spectra of bentonite fractions were measured at wave numbers 500-4000 cm^{-1}. The results of FTIR data of nanobentonite can be seen in Figure 2.

The relatively weak IR absorption band around wavenumbers 3387.62 and 3627.35 cm^{-1} comes from the O-H stretching vibration of Si-OH. Water molecules contained in nanobentonite can be known by the O-H bending at wavenumbers 1633.42 cm^{-1}. While at wavenumbers 1007.75 cm^{-1} is associated with Si-O-Si asymmetric stretching mode. Al-O-Si stretching vibrations can be seen to occur at 791.65 cm^{-1} and wavenumbers 471 and 526 cm^{-1} caused by Si-O bending vibrations.

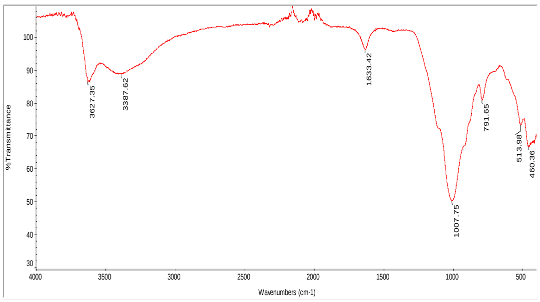

Figure 2. FTIR Spectra of Nanobentonite

Nanobentonite generally has montmorillonite and quartz as its main constituents. It can be seen in Figure 2, the presence of Si-O and Al-O-Si group bonds which are the main structures of quartz and montmorillonite constituents of nanobentonite [18].

XRD Test Results

Nanobentonite was characterized using XRD to determine the crystal form. Nanobentonite was tested using XRD at an angular scanrange of 5°-90° with a wavelength of 1.54060 Å. The XRD data analysis process was based on the International Centre for Diffraction Data (ICDD).

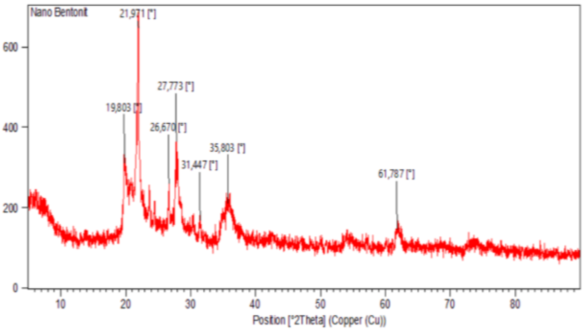

Figure 3. XRD graph of nanobentonite

Bentonite generally has the main constituents of montmorillonite and quartz, as seen in Figure 3. The diffraction peaks of 19.803° and 27.773° with d-spacings of 4.48Å and 3.21Å indicate the presence of montmorillonite and mark the presence of plagioclase which is a group of minerals containing Ca in the form of anorthic crystals and Na with reference code 00-010-0360. The 21.971Å diffraction peak is a silica mineral (SiO_2) tridymite with reference code 01-083-1339. The 26.670° peak marks quartz (SiO_2) with a hexagonal crystal form with reference code 01-085-0335 [19]. While at the peak of 31.447° marks crystobalite SiO_2 with code reference 00-001-0438. At the peak 61.787°, there is anorthic crystal-shaped aluminium silica with reference code 00-025-002.

XRD analysis can determine the size of crystals with a particular phase. The relationship between crystal size and the width of the X-ray diffraction peak through the Debye Scherrer equation approach is formulated as follows [20]:

$$D = \frac{K\lambda}{\beta\cos\theta}$$

D = crystal size
K = shape factor of the crystal (0.89)
λ = wavelength of X-ray (1.5406Å)
β = value of Full Width at Half Maximum (FWHM) (rad)
θ = angle of diffraction (degree)

Table 1. XRD diffraction peaks list of nanobentonite with particle size

Pos, [°2Th,]	FWHM Left [°2Th,]	d-spacing [Å]	Particle Size (nm)
19.7401	0.1338	4.49751	60.26636
21.9711	0.1338	4.04562	60.48269
26.6866	0.1004	3.34050	81.32168
27.7723	0.1338	3.21233	61.16184
31.4457	0.2007	2.84495	41.12152
35.8079	0.6691	2.50775	12.47733
61.7883	0.8029	1.50147	11.53047

Before being synthesized to nanoparticle size, natural bentonite typically has a particle size of 0.8 µm to 2000 µm [21]. The sonochemical synthesis method makes nanobentonite at an average particle size of 46.9 nm.

Determination of Optimum Nanobentonite and Nano TiO_2 Modified Carbon Paste Electrode Composition

The optimum composition of nanobentonite and nano TiO_2-modified carbon paste electrodes was determined by measuring the peak current generated in each variation of carbon paste electrode composition using niacinamide solution with a concentration of 5 ppm at pH 6 conditions measured using cyclic voltammetry in the potential range of -0.8 V to 1.8 V.

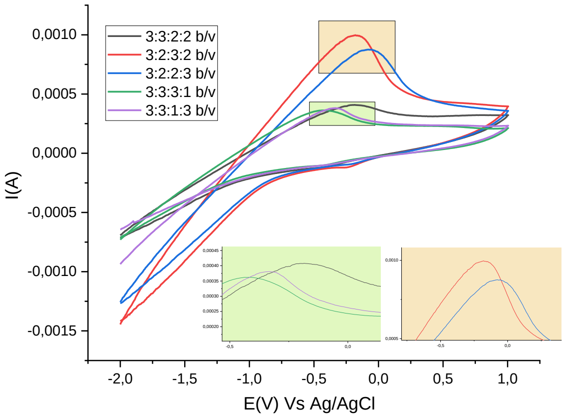

Figure 4. Voltammogram of the composition of nanobentonite and nano TiO_2 modified carbon paste electrode

Niacinamide produces a high oxidation current peak and a shallow reduction current. Even in some composition variations, there is no reduction current peak. This can be caused when the charge transfer is slow or the fast product reacts homogeneously in the oxidation process so that no reduction reaction occurs [22]. In contrast, the distance between IpA in the 3:2:3:2 and 3:2:2:3 b/v variations with the 3:3:2:2, 3:3:3:1, and 3:3:1:3 b/v variations is quite far. This could be due to paraffin, which affects the peak current formed. Paraffin, in the manufacture of electrodes, serves as an adhesive for other materials; the inert nature of paraffin and its low reactivity is very suitable. In addition, paraffin is an isolator, so when enough paraffin is used, it can affect the electron transfer process, which causes the peak current formed to be lower.

Table 2. Peak current of nanobentonite and nano TiO_2 modified carbon paste electrode composition

Composition	IpA(A)	IpC(A)
3:3:2:2	0.000408744	-0.000115025
3:2:3:2	0.000992784	-0.000120119
3:2:2:3	0.000874788	-0.000109932
3:3:3:1	0.000362054	-0.000103141
3:3:1:3	0.000379881	-0.000103141

Based on Table 3, it can be seen that the highest IpA peak is in the 3:2:3:2 b/v variation with a value of 0.000992784 A. The better the conductivity of the working electrode, the greater the maximum current peak produced due to the easy transfer of electrons for the reduction and oxidation reaction process [23].

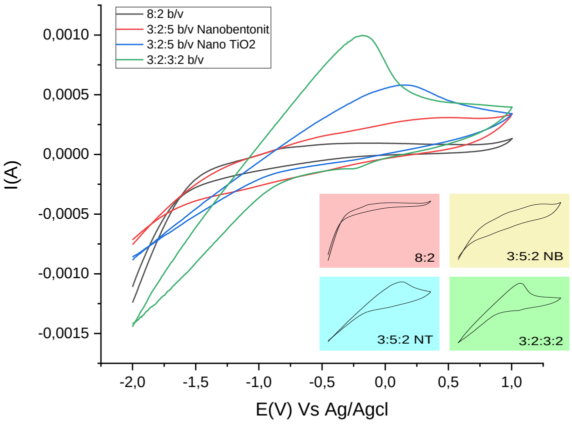

Figure 5. Voltammograms of modified and unmodified working electrodes

Modification of the working electrode significantly affects the peak current produced. Carbon paste electrodes with paraffin produce almost invisible IpA, while carbon paste electrodes modified using nanobentonite or nano TiO_2 alone show an increase in peak current due to the help of absorption of more compounds to increase the electron transfer process. However, the results of modifying carbon paste electrodes with two nanoparticle materials,

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nanobentonite and nano TiO_2, can increase current readings.

Table 3. Peak current of modified and unmodified working electrode

Composition	IpA(A)	IpC(A)
8:2	0,000050509	-
3:5:2 (NB)	0,000308574	-
3:5:2 (NT)	0,000580221	-
3:3:2:2	0,000992784	-0,000120119

The increase in IpA of nanobentonite and nano TiO_2 modified carbon paste electrodes can increase as much as 20 times compared to those without modification. It can be proven that modification of the electrode surface can produce new properties and new devices that can provide better properties in sample adsorption or electron transfer.

Determination of Optimum pH

Determination of the optimum pH is carried out to determine the optimum pH that can produce the highest current peak in the niacinamide measurement process. The pH setting is done with the help of a phosphate buffer solution.

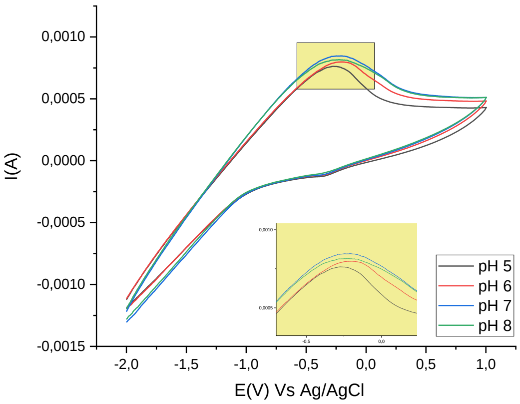

Figure 6. Voltamogram of pH variation

Based on Figure 6, there is no significant peak current comparison in each pH variation, but pH still influences the increase in anodic peak current. The use of buffer solutions can affect the test solution because it can increase the conductivity of the solution, maintain a constant ionic strength, and eliminate the transport of species through ion migration in an electric field [24].

Table 4. Peak current of pH variation

pH	IpA(A)	IpC(A)
5	0.000762875	-0.00012889
6	0.000799448	-0.00011955
7	0.000845359	-0.00011644
8	0.000815011	-0.00010555

The effect of modification of carbon paste electrodes with nanobentonite and nano TiO_2 as niacinamide analysis significantly influences ordinary carbon paste electrodes or carbon paste electrodes with nanobentonite or nano TiO_2 modification alone. The effect of pH variation also affects the stability of existing ions. So that the current produced is higher, the higher the peak current value produced indicates, the more sensitive the electrode is due to high electron transfer. It maximizes the analysis of the concentration of the test solution because the measured concentration is linear with the measured current.

CONCLUSION

Using nanocomposite materials on the working electrode in cyclic voltammetry in niacinamide assay increases sensitivity. The optimum ratio variation between carbon, paraffin, nanobentonite, and nano TiO_2 in making electrodes for niacinamide analysis is 3:2:3:2 b/v, and the optimum pH is 7. Carbon paste electrodes modified with nanobentonite and nano TiO_2 increase anodic peak current compared to electrodes without modification. This increase in peak current is due to an increase in the electrode in adsorbing niacinamide solution increases, so there is an increase in the electron transfer process. The effect of pH variation also affects the stability of existing ions. So the resulting current is higher, the higher the peak current value produced indicates, the more sensitive the electrode is due to high electron transfer. It maximizes the analysis of the concentration of the test solution because the measured concentration is linear with the measured current. For further research, conducting niacinamde analysis experiments on niacinamide samples in the product using the cyclic voltammetry method using modification of carbon paste electrodes with nanobentonite and nano TiO2 is necessary.

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