Development of Voltammetry Analysis Method of Iron Metal Ions by Solid-State Membrane with Carbon Nanotube

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Abstract: This work developed a selectively modified electrode for measuring the Fe(II) ions in continuous integration using voltammetry techniques. The study assessed various aspects, such as linearity, scan rate, repeatability, and real sample analysis. The experiment is performed using differential pulse voltammetry (DPV). The findings of the study indicated that the voltammetry method exhibited a regression line of $y = 36.507 \ln(x) + 990.73$, with a correlation value of 0.9627, with an optimum scan rate of 20 mV/s and good repeatability over five times measurement. On the other hand, when comparing the results using the UV-Vis spectrophotometric technique, the regression equation was found to be y = 0.20438x - 0.06987, with a correlation value of 0.99583. Notably, the voltammetry measurement outperformed the UV-Vis method since it allowed analysis of Fe(II) at concentrations up to 6.35×10^{-4} ppm (or 1.00×10^{-11} M), while the UV-vis measurement could only analyze up to 1.5 ppm (or 2.36×10^{-5} M). Consequently, the developed technique proves to be superior to the other methods for the analysis of Fe(II).

Keywords: Fe(II) ion; nanotube carbon; solid-state membrane; spectrophotometry; voltammetry

INTRODUCTION

Waste refers to the unnecessary disposal of substances generated during both industrial and domestic production processes, and if it is not managed appropriately, it can lead to environmental pollution. Chemical industrial wastes commonly contain substances such as silver (Ag), cadmium (Cd), cobalt (Co), iron (Fe), chromium (Cr), copper (Cu), nickel (Ni), and zinc (Zn) [1]. Among these metal ions, Fe is a robust, malleable metal frequently utilized in various applications. Fe ions are frequently present in different water sources. Elevated levels of Fe ions beyond the standard limits can result in water with an unpleasant odor, skin and eye irritation, damage to the intestinal wall, and even longterm health issues leading to fatality [2].

Numerous methods, such as UV-vis spectroscopy, atomic absorption spectroscopy (AAS), inductively coupled plasma-mass spectrometry (ICP-MS), and inductively coupled plasma-atomic emission spectroscopy (ICP-AES), have been used to measure iron metal. However, the atomic absorption spectroscopy suffered from a high detection limit. On the other hand, inductively coupled plasma mass spectrometry and inductively coupled plasma atomic emission spectroscopy are expensive instruments [3]. Given these disadvantages, there is a demand for an alternative method that offers improved iron determination. The potentiometry method, utilizing ion-selective electrodes, presents lowcost and simple analysis approach, offering good sensitivity and selectivity. Previous research demonstrated that Cu and Fe electrodes showed considerable sensitivity with a detection limit of 10^{-6} M [4]. In another study by Ganjali et al. [5], a specific compound was synthesized, composed of 4-methyl-hydrazinobenzothiazole hydrobromide and hydrazine hydrate, which is able to bind selectively with Fe ions.

Many researchers have made significant progress in the monitoring of Fe(II) ions through voltammetry, both in ionic and molecular compounds, particularly using electrometry systems [6-8]. Additionally, advancements have been made in voltammetry analysis techniques employing carbon nanotubes. Deswati et al. [9] conducted voltammetric determinations of Cd, Fe, and Pb while Thanh et al. [10] performed determinations of various heavy metal ions using a differential anodic stripping voltammetry pulse. Furthermore, Deswati et al. [11] developed a method for measuring Cd, Cu, Pb, and Zn ions in seawater. Carbon nanotubes are utilized not only as ion-selective electrodes for metal ion analysis but also for non-metal analysis, particularly organic compounds. Evtyugin and Porfir'eva [12] developed a method for the voltammetric detection of phenols and their derivatives using carbon nanotubes. Additionally, Madrakian et al. [13] successfully determined tyrosine, acetaminophen, and ascorbic acid compounds through voltammetry with carbon nanotube electrodes. The study conducted by Abd Azis et al. [14] focused on investigating the characteristics of glutamate using biosensors based on carbon nanotube electrodes.

Iron measurement using the UV-vis method commonly uses complexing agents such as ferrocene [15]. In this study, 1,10-orthophenantroline, which has a similar structure to ferrocene, was used as an electrode modifier of carbon nanotubes and performed using differential pulse voltammetry.

EXPERIMENTAL SECTION

Materials

All materials used in this work were analytical grade. Carbon nanotube (CNTs, purchased from Timesnano) and paraffin (purchased from Uvasol, Merck) were used as received. Fe(II) sulfate salt (FeSO₄, purchased from Sigma-Aldrich), 1,10-orthophenantroline ($C_{12}H_8N_{2}$, purchased from Merck), potassium chloride (KCl, purchased from Merck), and potassium nitrate (KNO₃, purchased from Merck) were also utilized in the study. Standard Fe ion solutions were freshly prepared by dissolving Fe(II) sulfate salt in distilled deionized water. The well water samples from specific locations in Yogyakarta State University, Indonesia.

Instrumentation

Differential pulse voltammetry was conducted using the EDAQ Potentiostat (Australia). The experimental setup consisted of a one-compartment electrochemical cell with a carbon-paste working electrode, a saturated Ag/AgCl reference electrode, and a Pt wire counter electrode, following the report by Farahi et al. [16]. The electrodes were immersed in a 40 mL buffer solution containing Fe(II) ions in different concentrations and pHs. The pH values were obtained using a glass-pH electrode from Orion 915600 (USA). The spectroscopy measurements were performed using a UV-vIS spectrophotometer (Shimadzu, 2450 Series).

Procedure

This work focused on the analysis of Fe(II) ions in well water, and the parameters of interest included linearity, scan rate, repeatability, and the Fe(II) detection in the well water samples.

Electrode modification

Modification of the electrodes was conducted based on Suyanta et al. [17]. The mixture of 4 g of 1,10orthophenantroline, 3 g of CNTs, and 3 g of solid paraffin with composition 4:3:3 (w/w) was combined into a homogenized paste. These modified CNTs paste electrodes were then firmly packed into Teflon tubing with an inner diameter of 4 mm. The copper wire was connected to the tubing to obtain the electrical contact, and the opposite end served as a disc electrode. To ensure a smooth surface, the disc electrode was carefully flattened on a piece of weighing paper before conducting measurements.

Voltammetry measurement

Fe(II) ion detection was conducted using differential pulse voltammetry, which has proven as be effective in metal ion detection. This technique has been developed and utilized by various researchers [18-19]. In the differential pulse voltammetry approach, the current is measured at two specific points for each pulse. They were just before the application of the pulse and at the end of the pulse. The difference between the two measured currents for each pulse is then plotted against the base potential [20]. However, in this present research, the oxidation currents were adjusted due to variations in scan rate, repeatability, and different concentrations studied. During the accumulation of the analyte, certain conditions were applied, including the accumulation potential (initial: -1000 mV, final: 0 mV), accumulation time, and the solution's pH (7.32), which were based on previous studies by Suyanta et al. [17]. Additionally, for the determination of Fe(II) ions using spectrophotometry, the specific conditions included a maximum wavelength range of 400-600 nm and a pH of 3.5, referring to the thesis research conducted by Khasanah and Sunarto [21].

Data analysis

The study's results were obtained by examining solutions of different concentrations of Fe(II) ions, focusing on the linear concentration region and establishing a calibration plot correlating the oxidation current with the concentration. This work aimed to assess the linearity and the correlation of calibration plots based on standard solutions and the samples under study. Both the voltammetry and spectroscopy methods were employed in this analytical technique.

RESULTS AND DISCUSSION

The Electrochemical Response of Fe(II) lons on the 1,10-Orthophenantroline Modified CNTs Paste Electrodes

The presence of the active ingredient, 1,10orthophenantroline, as a modifier in the CNTs paste electrode, had a notable impact on the voltammogram of Fe(II) ions. The voltammogram results for the measurements of iron ions are depicted in Fig. 1. As depicted in Fig. 1, the carbon paste electrode modified with 1,10-orthophenantroline exhibited a distinct oxidation peak for Fe(II) ions at 0.4550 V vs Ag/AgCl, registering a current of 94.839 nA. In contrast, the unmodified electrode did not exhibit any peak response. This observation indicates that the modified electrode effectively detected the presence of Fe(II) ions, demonstrating its suitable sensing capability.



Fig 1. Differential pulse voltammetry waveforms of CNTs modification with 1,10-orthophenantroline (blue line) and without modification (red line)

Linearity

The modified carbon paste electrode was analyzed to identify its linear concentration range, where a direct relationship between the peak current height and the Fe(II) ions concentration was observed. The concentration range investigated was between 1×10^{-7} and 1×10^{-11} M. The corresponding voltammograms for this analysis are presented in Fig. 2. According to Fig. 2, the modified electrode effectively detects the presence of Fe(II) ions across various concentrations. Higher Fe(II) ion concentrations yield higher voltammogram responses. It is worth noting that the standard potential value for Fe metal is -0.44 V [22]. However, based on potentiometry theory, changes in concentration do not directly impact the potential [23]. In this study, the potential value showed a shift due to the use of a modified 1,10orthophenanthroline solid membrane electrode, forming a complex that affected the potential value, unlike the pure Fe metal used in the standard potential determination.

Fig. 2 exhibits a linear relationship between the concentration and the measured current shown in Table 1, and it produced a linear graph. As shown in Table 1, plot of oxidation current (I) vs concentration of Fe(II) was obtained (Fig. 3). Based on the findings of this study, a clear association between the current and the concentration can be observed. This relationship is represented mathematically by the regression line equation: $y = 36.507 \ln(x) + 990.73$, with a correlation coefficient



(r) of 0.9627. Similarly, for the UV-vis spectroscopy

Fig 2. Differential pulse voltammetry waveforms of the 1,10-orthophenantroline modified CNTs paste electrode at 1.0×10^{-7} , 1.0×10^{-8} , 1.0×10^{-9} , 1.0×10^{-10} , and 1.0×10^{-11} M of Fe(II) ions concentrations

Table 1. The detection of various concentrations of Fe(II) ions using 1,10-orthophenantroline modified CNTs paste electrode

_	Concentration (M)	Current (nA)		
	1.0×10^{-7}	408.735		
	$1.0 imes 10^{-8}$	339.427		
	$1.0 imes 10^{-9}$	198.210		
	$1.0 imes 10^{-10}$	132.870		
	$1.0 imes 10^{-11}$	91.712		



Fig 3. The current response of 1,10-phenanthroline modified CNTs paste electrode towards 1.0×10^{-7} - 1.0×10^{-11} M concentration of Fe(II) ions



Fig 4. Iron(II)- 1,10-orthophenantroline calibration curve

method, the linearity is described by the equation y = 0.2044x - 0.0698, and it exhibits a higher correlation value (r = 0.9959), as demonstrated in Fig. 4 [21].

Both works obtained correlation value, r > 0.950 indicating they are good methods of analysis.

Scan Rate

In differential pulse voltammetry, the height and width of the peak of the current are affected by the scan rate [24]. The effect of the scan rate is shown in Fig. 5. Fig. 5 illustrates the influence of the scan rate on the peak current. Notably, peak currents differ at scan rates of 10, 20, 30, 40, and 50 mV/s. Moreover, variations in the scan rate result in slight shifts in the potential for each measurement, which may be attributed to electric current instability within the voltameter and kinetic constraints in the electrochemical reaction. A comprehensive record of the detection at different scan rates is presented in Table 2.

Fig. 5 and Table 2 demonstrate that as the scan rate increases, the obtained current value decreases. The optimum scan rate occurs when the voltammogram is

Table 2. The current response of 1,10-orthophenantroline modified CNTs electrode atdifferent scan rate

Scan rate (mV/s)	Current (nA)
10	109.411
20	111.028
30	107.554
40	105.678
50	100.185



Fig 5. Differential pulse voltammetry waveforms of 1,10-orthophenantroline modified CNTs electrodes towards 1.0×10^{-11} of Fe(II) ions at different scan rates

not broadened and the peak current reaches its highest value. In this study, the optimal scan rate for readings was determined to be 20 mV/s. Wei Wei Yao, Hong Mei Peng, and Richard D. Webster [25] have suggested that higher scan rates lead to increased peak currents because the thin diffusion layer formed allows for effective electron transfer around the surface of the modified solid membrane electrode. Conversely, lower scan rates result in smaller peak currents due to a thicker diffusion layer on the electrode surface, which can hinder the electron transfer process. The selection of the scan rate is based on previous research conducted by Suyanta et al. [26].

Repeatability of the Modified Electrode

The repeatability of the modified electrode was evaluated by performing five measurements using a single electrode with the same conditions at the optimum conditions, as shown in Fig. 6. The repeated measurements, performed five times, demonstrated consistent and stable peak currents, indicating excellent reproducibility of the modified 1,10-orthophenantroline carbon paste electrode. As supported by Silva et al. [27], the modified electrode exhibiting good precision suggests the absence of any matrix effects that could impact the detection process. Furthermore, the measurement results revealed a slight rightward shift in the potential value, though not significantly, which implies a kinetic limitation in the electrochemical reaction of Fe(II) ions within the 1.10orthophenanthroline-modified carbon paste electrode. The complexation reaction (Eq. (1)).

 $2Fe^{2+}(aq) + 3C_{12}H_8N_2(aq) \rightarrow [Fe(C_{12}H_8N_2)_3]^{2+}(aq), \text{ or:}$ $Fe^{2+} + 3 \text{ o-Phen } \rightarrow [Fe(o-Phen)_3]^{2+} [21]$ (1)



Fig 6. Differential pulse voltammetry waveforms of 1,10-phenanthroline modified CNTs electrode for 5 times of 1×10^{-7} M of Fe(II) ions



Fig 7. Differential pulse voltammetry waveforms of detection Fe(II) ions in well water

Table 3. Comparison performance of differential pulse voltammetry (DPV) and UV-vis spectrophotometry techniques for Fe(II) ions detection

Matalian	Correlation coefficient		Limit of detection (M)	
Metal Ion	DPV	UV-vis	DPV	UV-vis
Fe(II)	0.9627	0.9959	$1.00\times10^{\scriptscriptstyle -11}$	$2.36\times10^{\scriptscriptstyle-5}$

The Measurement Result of Fe(II) lons in the Well Water

The 1.10-orthophenantroline modified CNTs electrode was applied to detect Fe(II) ions in well water. Fig. 7 shows the detection of Fe(II) ions in well water for 0, 10, and 100 times dilution.

The measurements of well water samples indicated a very low Fe(II) ions content. Based on the Fe(II) ions voltammogram, the obtained current from the analysis exceeded 350 nA. Considering this current value, the concentration of Fe(II) ions in the well water, calculated using the regression conditions mentioned earlier, was found to be 2.3×10^{-9} M.

Comparison of Validation Results

The validation results were compared to assess the superior technique for detecting Fe(II) using UV-vis spectrophotometry and voltammetry. The comparison involved assessing the correlation coefficient and analytical capability for Fe(II) in both methods, as presented in Table 3. The study findings indicate that the voltammetry method outperformed the UV-vis spectrophotometry technique in measuring Fe(II) ions content in both solutions and well water. The differential pulse voltammetry exhibited greater sensitivity with a lower detection limit, resulting in a more effective analysis process.

CONCLUSION

The 1,10-orthophenantroline-modified carbon nanotubes electrode was successfully developed, obtaining an exceptional oxidation response of Fe(II) ions. This modified electrode provided a low limit of detection of 1.0×10^{-11} M with regression equation of y = 36.507 In (x) + 990.73, r = 0.9627. The optimum scan rate of 20 mV/s and good repeatability further contributed to this work's reproducibility. Moreover, the voltammetry technique proved superior to UV-vis

spectrophotometry in the detection of Fe(II) ions in both solutions and well water.

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AUTHOR CONTRIBUTIONS

The experimental work and manuscript preparation were undertaken by Suyanta. Ilyas Md. Isa supplied the necessary experimental materials. Data analysis was performed by Regina Tutik Padmaningrum and Qonitah Fardiyah. Sunarto, Rahadian, Fredy Kurniawan, Illyas Md. Isa, and Qonitah Fardiyah contributed to the manuscript revisions. Karlinda assisted in conducting the experiments. All authors reached a consensus on the final version of the manuscript for publication in the journal.

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