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Electrochemical Detection of Nitrite Using a Fe_3O_4/Nafion-Modified Gold Wire Electrode

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ABSTRACT

This study presents the development and evaluation of an electrochemical sensor for nitrite detection based on magnetite nanoparticles (Fe_3O_4 NPs) and a Nafion-modified gold wire electrode. Fe_3O_4 NPs were synthesized through an electrolysis route and subsequently characterized using X-ray diffraction (XRD) and field emission scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (FE-SEM/EDX). The XRD diffraction pattern is similar to the standard JCPDS card number 19-0629, confirming the structure of Fe_3O_4 NPs. Morphology analysis using FE-SEM revealed Fe_3O_4 NPs with an average size measured to be 123.4 nm. Cyclic voltammetry (CV) measurements demonstrated a high oxidation peak for nitrite detection using the proposed electrode at neutral pH (pH 7). CV analysis towards $K_3[Fe(CN)_6]$ shows that the proposed electrode gave the highest current peak. The specific surface area of the proposed electrode was found to be 0.149 cm^2. Quantitative analysis using CV showed a limit of detection (LOD) of 4.4 ppm. Using the amperometry techniques, the LOD produced was as low as 0.00105 ppm. These findings highlight the potential of the Fe_3O_4 and Nafion-modified gold electrode for sensitive nitrite analysis.

INTRODUCTION

Nitrite (NO_2^-) is one of the ions that are a result of the chemical conversion of nitrogen-containing compounds (Hasan et al., 2022). Nitrite ion usually exist as a preservative in processed food and as a contaminant in water (W. Wang et al., 2025). Excessive levels of nitrite intake through food or drinking water can pose a significant health risk. For instance, it can lead to methemoglobinemia in humans. Therefore, the European Union Scientific Committee on Food has set the maximum intake of nitrite to 0.06 mg/kg body weight, while the WHO states that the maximum allowable concentration of nitrite in drinking water should not exceed 3 mg/L (Du et al., 2023; W. Wang et al., 2025). Therefore, it is crucial to develop a simple, accurate, and sensitive analytical method to control food safety.

The advancement of nitrite analysis has been developed using many methods such as spectroscopy and chromatography. The methods mentioned before had advantages such as simple and wide application, but they also had disadvantages of time-intensive pre-treatment, low accuracy, and low selectivity (Sari et al., 2024; Y. Wang et al., 2025). One of the alternative methods for nitrite detection is the electrochemical method, which has been used to detect nitrite in food and water samples. The reason electrochemical sensors were chosen was due to their advantages, which include ease of use, low cost, and high sensitivity. Electrochemical methods can also be improved by modifying the

working electrode to achieve the best results (Chen et al., 2025; Du et al., 2023; Sari et al., 2024; Y. Wang et al., 2025).

Over recent years, considerable work has been done on electrode modification to achieve lower detection limits and improved selectivity. Transition metals have been widely used in electrochemical detection due to their unique properties, such as high catalytic activity, biocompatibility, and conductivity. Gold (Au) wire, as the main working electrode, has been used to detect many compounds and ions (Chu et al., 2023; Hutapea et al., 2025; Sari et al., 2024). The use of a bare gold electrode is shown to have low sensitivity and selectivity towards electrochemical detection. Therefore, it is crucial to modify the surface of the electrode to increase the signal produced during the analysis. Fe_3O_4 (magnetite) nanoparticles (NPs) are a good modification of the electrode surface due to their properties. One of which is good conductivity, good catalytic activity, high surface area, and easy preparation (Bhuvaneswari et al., 2023; Riahifar et al., 2021; Shalali et al., 2022). However, some disadvantages of Fe_3O_4 are that they are prone to aggregation and oxidation (El-Desoky et al., 2022). To prevent the aggregation of Fe_3O_4 NPs during analysis, some method can be deployed, one of which is using a conductive polymer. Nafion (perfluorinated sulfonic acid polymer) is a promising candidate for surface modification of Fe_3O_4 NPs to prevent agglomeration. Nafion as a surface modification is also shown to exhibit excellent resistance to electrode fouling and to increase the voltametric signal during analysis (Alemayehu et al., 2023; Rabbani et al., 2024; Yi et al., 2024).

In this work, Fe_3O_4/Nafion was modified onto the surface of a gold electrode to detect nitrite. $Fe_3O_{4_4}$ was synthesised through an electrochemical synthesis using an iron plate. The obtained Fe_3O_4 is then mixed with a Nafion solution and subjected to sonication. The analysis will be done using cyclic voltametric (CV) and amperometric methods to obtain the limit of detection (LOD) in nitrite detection.

RESEARCH METHOD

Chemicals and Reagents

The working electrode consists of 7 cm of 24-karat gold wire. The iron plate with a dimension of 10 cm x 2 cm x 0,2 cm was obtained from EELIC China. Sodium Sulfate Anhydrous (Na_2SO_4, 99%) was obtained from SAP CHEMICALS, Indonesia. Demineralized water and ethanol (CH_3OH, 98%) were purchased from PT. Brataco Chemicals, Indonesia. Sodium Nitrite analytical grade, 5% Nafion solutions, Potassium ferricyanide ($K_3[Fe(CN)_6]$), and potassium chloride (KCl) were obtained from Sigma Aldrich.

Instrumentation

The Fe_3O_4 was characterized using X-ray diffraction (XRD) (X'Pert Pro MPD), and Field emission scanning electron microscopy-dispersive X-Ray spectroscopy (FESEM/EDX) (Hitachi Regulus 8220). The electrochemical measurements were done using Autolab Methrom PGSAT 128N.

Synthesis of Magnetite (Fe_3O_4)

The synthesis of Fe_3O_4 was carried out using the electrolysis method based on previous work, with a slight adjustment (Yu et al., 2022). The iron plate is cleaned using sandpaper. The iron plate was then washed using demineralized water and ethanol. The iron plate, which acts as both the anode and cathode, is then connected to a power supply. Both electrodes were immersed in a 0.01 M Na_2SO_4 solution with a depth of 2 cm. The distance between the electrodes was kept at 2 cm. The synthesis was carried out at 50 V for 30 minutes. The Fe_3O_4 were formed as colloids dispersed in the solution. The Fe_3O_4 obtained were dried in the oven at 80 °C overnight.

Fabrication of Fe_3O_4/Nafion modified gold electrode.

The gold wire surface was polished using sandpaper and then submerged in an equal mixture of ethanol and water, and sonicated for 30 minutes to clean the electrode. After that, the electrode was left to dry. The gold wire was covered in shrinkage cable, leaving a 1 cm opening at the tip of both ends of the electrodes.

The electrode modifier was prepared by mixing 1 gram of Fe_3O_4 nanoparticles obtained, 100 µL of 5% Nafion solution was added as a binding agent, and 200 µL of demineralised water to help turn the composite into a paste. The mixture was then sonicated for 30 minutes, and the end result was a paste of Fe_3O_4/Nafion composite. The mixture was then applied to the surface of the gold electrode and left to dry at room temperature inside a desiccator for 12 hours before use.

Electrochemical Measurement

The electrochemical measurements were performed using cyclic voltammetry (CV). The three-electrode cell system consists of platinum as the counter electrode (CE), Ag/AgCl (KCl 3 M) as the reference electrode (RE), and the proposed electrode as the WE. The electrochemical performance of electrodes toward nitrite analysis was observed using CV -0.4 to 1.2 V and a scan rate of 100 mV/s. The effective surface area between the electrode was measured in 10 mM $K_3[Fe(CN)_6]$ n in 0.1 M KCl solution using CV techniques with a potential range of -0.8 to 0.8 V and a scan rate of 100 mV/s. The various concentrations of nitrite solution with CV techniques were measured in the various nitrite concentrations of 10, 25, 50, 75, 100, 125, and 150 ppm. The various concentrations of nitrite solution with amperometric techniques were measured in the various nitrite concentrations of 1-11 ppm.

RESULTS AND DISCUSSION

Fe_3O_4 Characterization

Fe-SEM analysis is shown in Figure 1. The analysis shows that the morphology of Fe_3O_4 displayed at a magnification of 110,000x shows the nanoscale of Fe_3O_4 nanoparticles with a plate-like shape. Fe_3O_4 shows that the average size of Fe_3O_4 was 123.4 nm.. The Diffractogram of the Fe_3O_4 is shown in Figure 2. The diffractogram shows five peaks that correspond to (220), (311), (400), (511), and (440), respectively. The result indicates a spinel structure of Fe_3O_4, which is similar to the standard JCPDS card no. 19-0629. The previous work done by Yu in 2022 also found the same result on the XRD analysis towards Fe_3O_4 nanoparticles using the electrolysis method (Yu et al., 2022).

The reaction mechanism starts with the oxidation of Fe to Fe^{2+} and Fe^{3+} in the anode. At the cathode, water is reduced, producing hydroxide ion (OH^-). The Fe^{2+} and Fe^{3+} ions will move towards the cathode and then react with the $8OH^-$. The end product of the reaction are Fe_3O_4 nanoparticles as the and water (Yu et al., 2022).

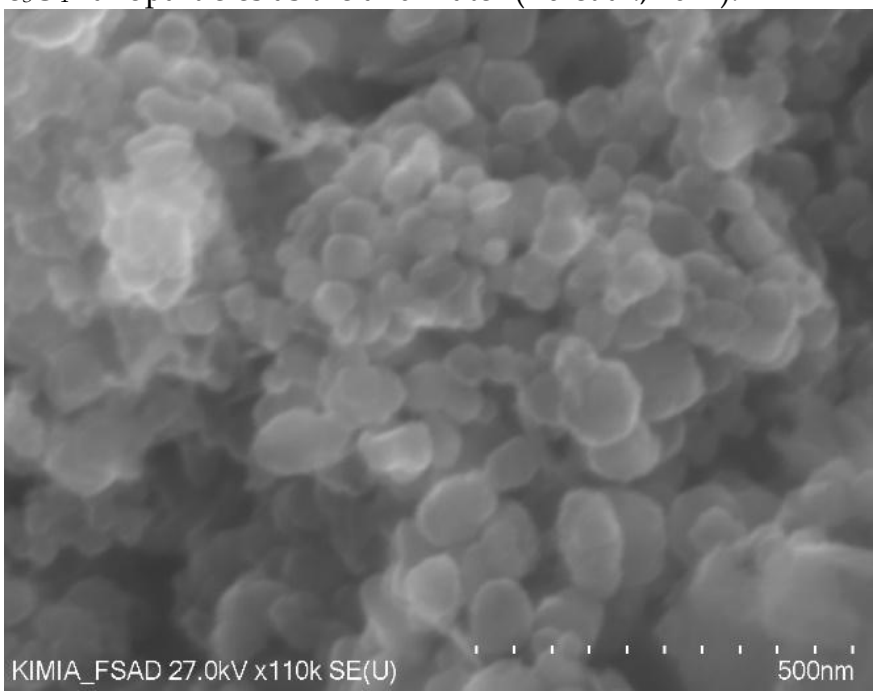

Figure 1. SEM picture Fe_3O_4 NPs and diffractogram of Fe_3O_4 NPs

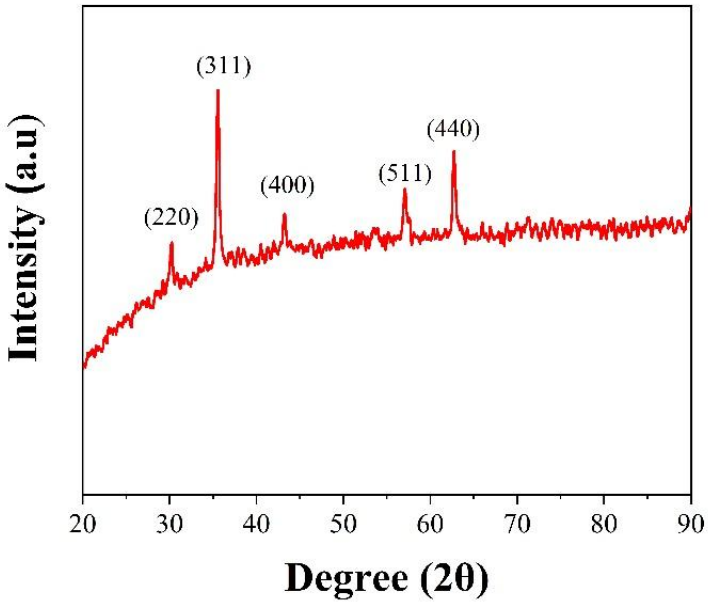

Figure 2. Diffractogram of Fe_3O_4 NPs

Electrochemical Measurement on Different Electrodes

The electrochemical measurements of nitrite using three different electrodes is shown in Figure 3. The bare gold electrode (black line) shows a really small anodic current peak (ipa) and a small cathodic current peak (ipc). The Nafion-modified gold electrode (red line) shows a higher ipa and ipc during nitrite analysis compared to the bare gold electrode, but it didn't show a distinctive oxidation peak for nitrite. The Fe_3O_4/Nafion-modified gold electrode (green line) shows a higher peak in both ipa and ipc compared to both bare gold and the Nafion-modified gold electrode. The proposed electrode also showed a faster electron transfer during the nitrite analysis. This can be caused by the Fe_3O_4 properties that are conductive and show an excellent catalytic activity; these properties can enhance the electron transfer towards the analyte (Bhuvaneswari et al., 2023; Riahifar et al., 2021; Shalali et al., 2022). This means that the proposed electrode shows a higher sensitivity towards nitrite during analysis.

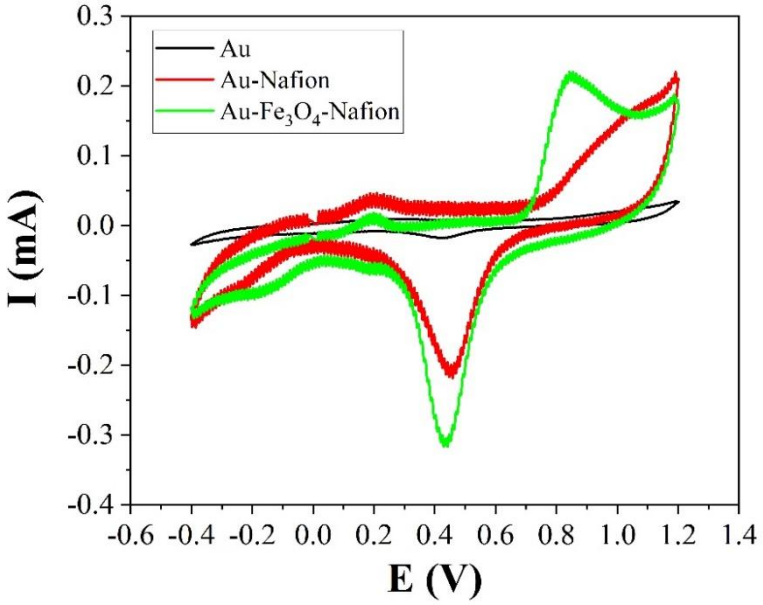

Figure 3. Electrode performance towards nitrite detection

Electrochemical measurement in a 10 M $K_3[Fe(CN)_6]$ solution is conducted to obtain a specific area of the electrode (Figure 4). The bare gold electrode shows to exhibit a small ipa and ipc compared to the gold modified with Nafion and gold modified Fe_3O_4/Nafion. The gold electrode modified with Nafion showed an increase of ipa and ipc. The proposed electrode produces a higher signal for both ipa and ipc than a bare gold electrode or a Nafion-modified gold. The proposed electrode showed the highest ipa and ipc compared to the other electrode, and it also exhibits the fastest electron transfer. Using the Randles-Sevcik equation and the diffusion constant of $K_3[Fe(CN)_6]$, the effective surface area of the bare gold electrode, the gold electrode modified with Nafion, and the proposed electrode, respectively, is 0.094, 0.129, and 0.149 cm^2.

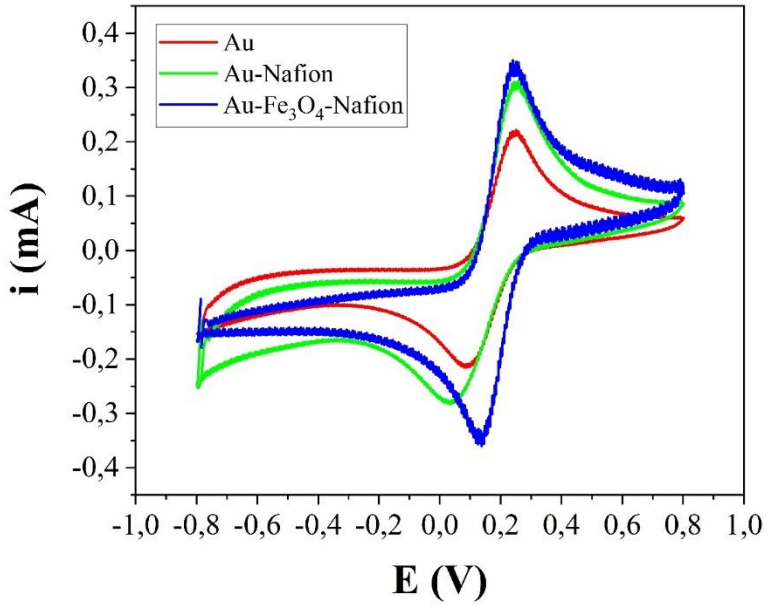

Figure 4. Electrode performance towards $K_3[Fe(CN)_6]$ detection

Efffect of pH on Nitrite Detection

The electrochemical measurement of nitrite in 0.1 M PBS with a variation of pH value is conducted using the Fe_3O_4/Nafion-modified gold electrode. Figure 5 shows the different performance of the pH condition during nitrite measurement. The analysis of nitrite in PBS pH 4 shows no anodic peak during the analysis, only showing the reduction peak (red line). Analysis of nitrite in PBS pH 10 shows a small ipa and ipc during the nitrite analysis (blue line). Analysis of nitrite in PBS pH 7 (black line) shows a good high ipa compared to other PBS variations. The pH 7 is a perfect condition for the nitrite analysis, which could be because at pH 7, the nitrite ion can keep its form without undergoing an oxidation process, turning it into the nitrate ion. The same optimum result has also been found at pH 7 for nitrite oxidation in the previous work using different electrodes (Sari et al., 2024).

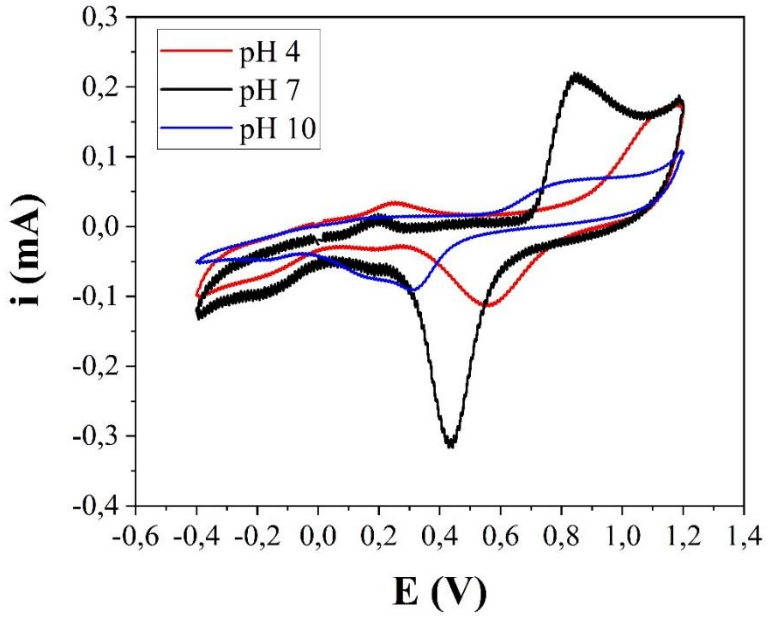

Figure 5. pH analysis of nitrite detection

Calibration curve for Nitrite determination by Fe_3O_4/Nafion modified Au electrode

The electrochemical measurement of nitrite analysis using Fe3O4/Nafion modified Au electrode is shown in Figure 6. The increase of nitrite concentration from 10 to 150 ppm shows an increase in ipa . The ipc, as shown in Figure 6, does not indicate the reduction of nitrite, since the addition of nitrite gave no increase in the reduction peak. The reduction peak could be the result of dissolved hydrogen reduction. The only possible reaction that can occur is the oxidation of nitrite. Therefore, nitrite measurement and limit of detection (LOD) analysis will be focused on Ipa rather than Ipc. The Ipa is plotted against concentrations (ppm), which gives the linear equation of ipa=3.818x10-3[NO2-]+1.142x10-1 with R^2 = 0.998 (Figure 7). The limit of detection and sensitivity values are calculated and produce values of 4.4 ppm and 0.025 mA ppm cm-2 for CV techniques. The reduction peak, as shown in Figure 6, does not indicate the reduction of nitrite, since the addition of nitrite gave no increase in the reduction peak. The reduction peak could be the result of dissolved hydrogen reduction.

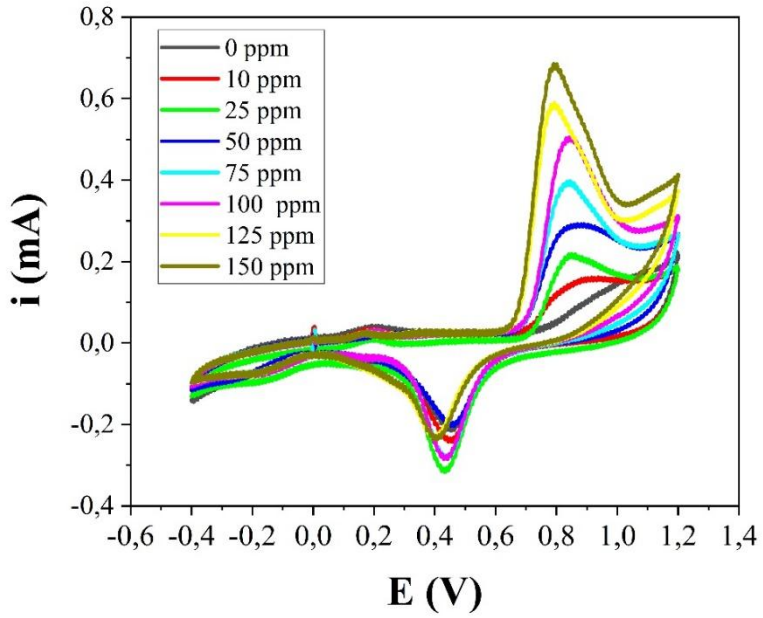

Figure 6. Cyclic Voltammogram of nitrite detection in different concentration

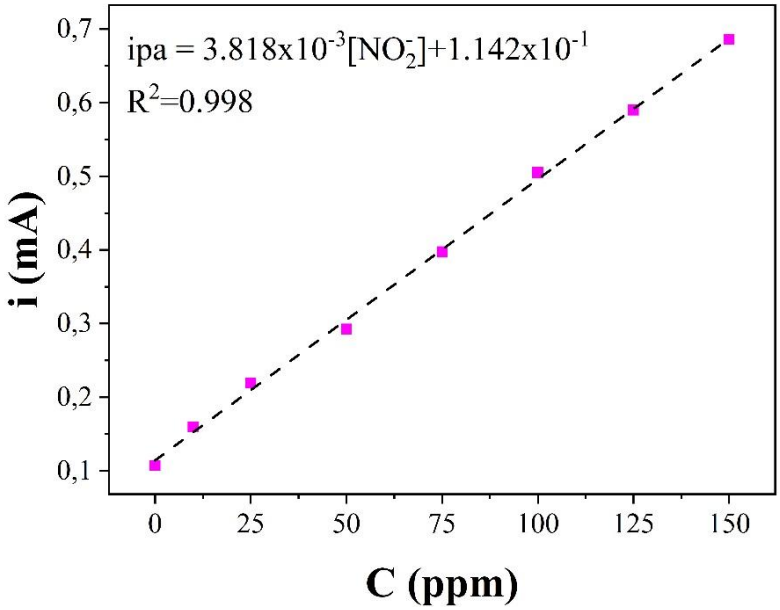

Figure 7. Linear curve of nitrite concentration vs anodic peak current

Amperometric analysis for Nitrite ion determination

The analysis using amperometry methods shows an increase in current with an increase in nitrite concentration (Figure 8). The oxidation of nitrite causes the current in amperometry. This can be observed when the addition of nitrite to the solution increases the current and remains stable for 50 50-second duration. The plot between concentration and current produces a linear curve of ipa=1.344[NO2-]+0.577 with an R^2=0.998 (Figure 9). The detection limit was calculated to be 0.00105 ppm with a sensitivity of 9.02 µA ppm

cm-2. The results of both methods are compared to the previous work on nitrite analysis using different electrodes, which can be found in Table 1. The results of the LOD produce more sensitive detection towards the nitrite analysis.

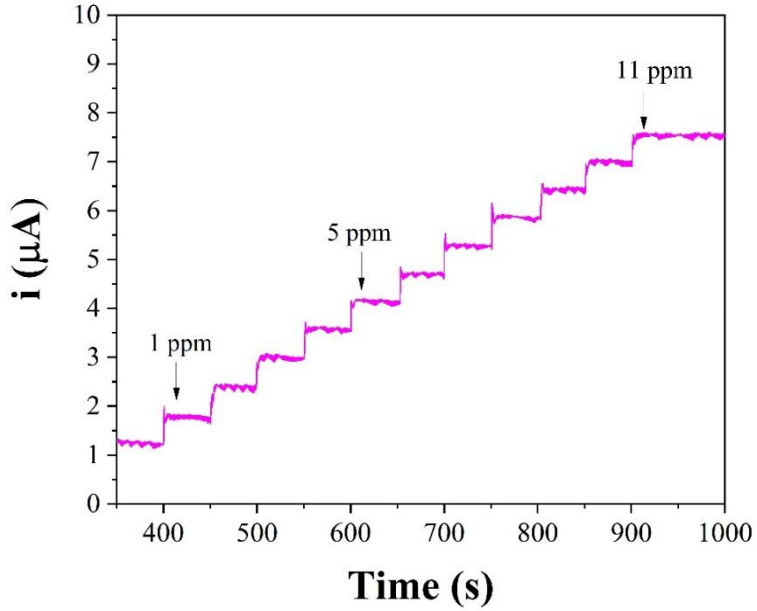

Figure 8. Amperometry measurement of nitrite in different concetration

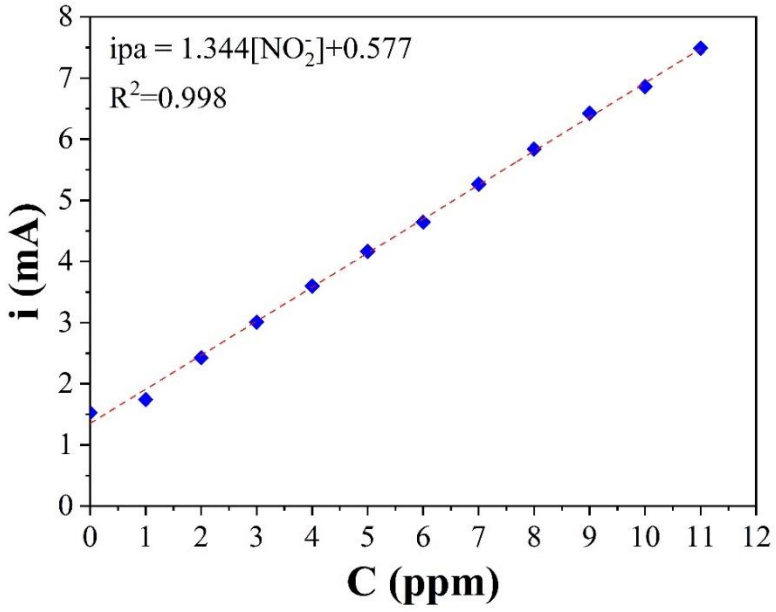

Figure 9. Linear curve of nitrite concentration vs current

Table 1. Limit Detection Result compared with other electrode

Electrodes	Methods	Range	LOD (ppm)	Source
ERGO/AuNPs/SPCE	DPV	1-6000 uM	0.00598	(Jian et al., 2018)
MWCNTs/Co-MOF/GCE	DPV	80-1100 μM	8.282	(Salagare et al., 2022)
Au-NPs/PPyC/SrTiO3 NCs/PEDOT:PSS/GCE	CV	150-1500	0.873	(Faisal et al., 2024)
Fe_3O_4/SiO_2/GCE	DPV	0.01-1 mM	0.153	(Zhang et al., 2024)
CuO/MnO_2/SPE	LSV	0.2-60 μM	0.00327	(Farina et al., 2025)
Fe_3O_4/Nafion modified Gold Wire	CV Amperometric	10-150 ppm 1-11 ppm	4.4 0.001	This Work

CONCLUSION

The Fe_3O_4/Nafion-modified gold electrode has been successfully fabricated. Fe_3O_4 is synthesized with electrolysis methods, shows a particle size averaging at 123.4 nm, and the diffractogram shows the formation of Fe_3O_4 NPs that are similar to JCPDS card no. 19-0629. CV analysis towards nitrite and $K_3[Fe(CN)_6]$ analysis shows a good result compared to the bare gold electrode and the gold electrode coated with Nafion. The pH analysis using CV methods shows the optimum pH condition during nitrite analysis at pH 7. LOD analysis of nitrite measurement reaching up to 4.4 ppm using the CV method, and 0.00105 ppm for the amperometry method.

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