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Determination of Iron(III) in N-Methyldiethanolamine Media Utilized in Sweetening Plant of Gas Treating Industry by Using Self-Assembled Monolayer on Gold Electrode

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Determination of iron(III) in N-methyldiethanolamine activated by piperazine (a-MDEA) utilized in sweetening plant of gas treating industry is described by preparation of gold 2-mercaptosuccinic acid self-assembled monolayer electrode and characterized by cyclic voltammetry and square wave voltammetry methods. The square wave voltammograms showed a sharp peak around positive potentials +0.180 V that was used for construction of the calibration curve. Factors were optimized for iron(III) stripping analysis and optimal condition was frequency of 35 Hz, step potential of 14 mV, amplitude of 40 mV, pH =3, and preconcentration time of 10 min. A calibration curve was obtained for iron(III) in a-MDEA in a linear range of 1.25×10^{-6} to 2.69×10^{-5} mol L^{-1}. The detection limit was found to be 1.94×10^{-7} mol L^{-1}. The relative standard deviation (RSD) of 3.29 % for n = 6 at 1.79×10^{-5} mol L^{-1} iron(III) in a-MDEA media was observed in the best conditions. The presented procedure was successfully applied for determination of iron(III) content in the real samples from sweetening plant of gas treating industry.

Keywords: Iron(III) determination, N-Methyldiethanolamine media, Sweetening plant of gas treating industry, 2-Mercaptosuccinic acid, Self-assembled monolayer, Osteryoung square wave voltammetry, Stripping analysis

1. INTRODUCTION

The corrosion of ferrous metals in soils is a major problem for owners and managers of water, sewerage and oil and gas distribution systems [1]. Also the problem of corrosion and the recognized expenses on standing and its control for preventing of events and unwanted stopping in production process is one of the sensations of industrial units. For this purpose the standing rate of some existing metals in fluid in industrial units is one of the identification ways of type and trend of corrosion; hence, attending to rapid and high careful analysis methods offer good help to this affair.

In refining industry, natural gas widely uses alkanolamines in sweetening process (removing acid natural gases of H_2S, CO_2) [2]. Nowadays; because of some problems in process, the use of amines mixture and also formulated amines are being increased. One of the most, popular and practical formulated amines is N-methyldiethanolamine activated by piperazine (a-MDEA) which developed based on BASF company license in the early 1970s and the first commercial unit for sweetening of natural gases installed in 1982 [3]. In this solvent by the use of piperazine, MDEA is activated and the speed and adsorption rate of CO_2 considerably increased [4]. By the use of a-MDEA we can reduce the concentration of CO_2 and H_2S in the sweetening gas to the amount 5 μg mL^{-1} and 1 μg mL^{-1}. Furthermore, the cases such as low adsorption of heavy carbohydrates, very low corrosivity (so that in the production of equipments we can use of steel carbon), very low manner of solvent in cycle, the capability of performing the operations in gas flows in high pressures (up to 120 times) and the low trend for making foam are other advantages for the use of solvent [5].

Analytical techniques for iron determination in aqueous media have been reviewed [6,7] and reported in literature including; different spectroscopic techniques such as: fluorimetry [8], atomic emission [9] and atomic absorption spectrometry, often benefit flame [10], graphite furnace [11] or inductively coupled plasma [12] for atomization; spectrophotometric methods [13]; spectrofluorescence quenching [14] and chemiluminescence's methods [15]; mass spectrometry [16]; liquid chromatography [17]; capillary electrophoresis [18]; ion chromatography with various detection systems [19]; stripping voltammetry proceeded by adsorptive collection of electroactive complexes of iron [20]. Chemical modification of the electrode surface is a major area of the current research in electrochemistry featuring a wide spectrum of promising applications such as corrosion studies [21], improvement of the electronic properties [22], or increases the electrocatalytic activity [23], analytical selectivity [24], and sensitivity [25] of the electrode. Examples of some surface modifiers include polymers [26], metal oxides [27], zeolites [28], composite materials [29], and self-assembled monolayers (SAMs) [30]. The gold thin film SAMs resulted from thiols, disulfides, and sulfides resist desorption over a wide range of pressure, temperature, pH, and electrical potential. So, recently they have been used for trace determination of different types of analyte [31]. Several voltammetric methods have also reported using different modified electrodes for the determination of iron(III) in aqueous media [32-40]. However, up to our knowledge, there is only one report about interaction of iron(III) with SAMs have quantitative calibration information in aqueous environment [41]. It seems succinic acid could selectively complex the iron(III) in the presence of some inorganic ions. Accordingly, SAMs preparation of 2-mercaptosuccinc acid (MSA) on the gold electrode (Au-MSA SAM) is convenient and its application as sensor in aqueous solutions is straightforward.

In this work, preparation and application of Au-MSA SAM electrode for determination of iron(III) in a-MDEA utilized in sweetening plant of gas refinery industry is described using cyclic voltammetry (CV) and square wave voltammetry (SWV) methods. The experimental data are presented and discussed from which the sensor is characterized in non-aqueous media. The sensor benefit from immobilized succinc acid functional groups for selective accumulation, and then, quantitative voltammetric determination of iron(III). Applicability of the sensor was successfully tested by determining of iron(III) in real samples including purified a-MDEA and a-MDEA utilized in sweetening plant of gas treating industry samples.

2. EXPERIMENTAL

2.1. Chemicals and solutions

2-Mercaptosuccinic acid (MSA), ferroammonium sulphate, and nitrate salts of cations such as potassium, sodium, cadmium, magnesium, manganese, nickel, cobalt, copper, zinc, and lead were supplied from Merck®. All chemicals were of analytical reagent grade and used as received. Dilute solutions of iron(III) were prepared immediately before use from a stock solution. The iron stock solutions (1 mg mL^{-1}) was obtained by dissolution of 1.0000 g of extra pure iron in 100 mL of nitric acid (sp g 1.42) with the aid of heat, and then dilution to 1 L with purified a-MDEA. The glassware were soaked in 6 mol L^{-1} HNO_3 and carefully cleaned before use to avoid contamination. The repetitive voltammetric scans started immediately after the electrode preconcentration in stirred solution containing iron(III) analyte. The real samples were used as follows: (i) purified a-MDEA sample and (ii) a-MDEA utilized in sweetening plant of gas treating industry sample. The standard addition solutions were prepared by adding an appropriate amount of standard iron(III) solution to a known volume of a-MDEA utilized in sweetening plant of gas treating industry sample.

2.2. Electrode preparation

A polycrystalline gold disk electrode (0.0314 cm^2, Metrohm) was polished using aqueous slurries of alumina (0.3 down to 0.05 μm), sonicated in water/ chloroform /water for 5min, and then cleaned electrochemically by cycling the electrode potential between 0.000 and +1.500 V versus Ag/AgCl in 0.5 mol L^{-1} sulfuric acid until reproducible voltammograms were observed [42]. A roughness factor of 1.68±0.11 was obtained from ratio of the real to geometric surface area of the electrode [43] and attempted to maintain it constant in all experiments [44]. The cyclic voltammogram obtained on cleaned bare Au electrode for $Fe(CN)_6^{3-}$ as a reversible redox probe showed a peak separation ($\Delta Ep \sim 60$ mV) that confirmed the safety of the system. Immediately before modification, the cleaned electrode was thoroughly rinsed with distilled water. Then, the electrode was modified by placing into a 40 mmol L^{-1} MSA aqueous solution for 12 h to form Au-MSA SAM electrode. The modified electrode was washed with distilled water to remove physically adsorbed MSA and used as working electrode for electrochemical measurements.

2.3. Electrochemical measurements

A conventional three-electrode cell consisting of Au-MSA modified electrode as working, a Ag/AgCl electrode as reference, and a platinum bar with large surface area as auxiliary electrode, was used for electrochemical measurements. All reported potentials are referenced to the Ag/AgCl electrode. The measurements were carried out using Potentiostat/ Galvanostat Autolab 12 interfaced with a personal computer, and controlled by GPES 4.9 software (Eco Chemie B.V. Utrecht, The Netherlands). The electrochemical characterization of Au-MSA SAM electrode was performed in the presence of 0.5 mmol L^{-1} $Fe(CN)_6^{3-}$ redox probe using CV. For quantitative determination of iron(III), the stripping measurements were performed under optimized conditions. Electrochemical blank tests were always carried out by immersing the modified electrode in the preconcentration solution before adding iron(III) ions. The regeneration procedure involving iron(III) elimination from the electrode surface was performed by holding the electrode potential in a stirred 0.1 mol L^{-1} EDTA solution (pH 3.0) for ∼100 s.

2.4. Optimization of the conditions

The solution matrix can influence on the efficiency of preconcentration and stripping processes. The 0.1 M solution of the following electrolytes; KCl, CH_3COONa, KNO_3, $NaNO_3$, NaF and phosphate buffer saline (PBS) solutions were studied and finally a maximum response was observed when 0.1 mol L^{-1} KNO_3 was used as electrolyte in preconcentration as well as stripping steps. Dependence of the square wave voltammogram (SWV) cathodic peak current on the preconcentration time of Au-MSA SAM electrode was studied. Thus, the modified electrode was immersed in a 3.58×10^{-6} mol L^{-1} iron(III) solution containing 0.1 mol L^{-1} KNO_3 at pH 3. Then, the solution was stirred for different periods of time. The results showed that the peak current was increased as a function of preconcentration time. Asymptotic value was reached within 10 min.

The SWV peak current is dependent on various instrumental parameters, which are of course interrelated. After careful examination, we found that the optimized instrument setting of amplitude and step potential, and frequency as 40 mV, 14 mV and 35 Hz, respectively. Under optimum condition, SWV showed a cathodic wave around +0.180 V for preadsorbed iron(III) on Au-MSA SAM electrode.

3. RESULTS AND DISCUSSION

3.1. Analytical characteristics

The cyclic voltammograms obtained for bare Au (a); and Au-MSA SAM electrodes before (b) and after (c) preconcentration in 20% a-MEDA solution containing 3.0×10^{-5} M iron(III), 0.1 mol L^{-1} PBS, pH 3.0 are shown in Fig. 1.

Starting potential and scan direction are important issues for iron ions determination by adsorptive stripping voltammetry. Therefore, potential scans were started from positive region, where iron(III) was stable, and continued to negative direction. Curve (a) shows that iron(III) ions are not adsorbed onto the Au in the absence of a monolayer.

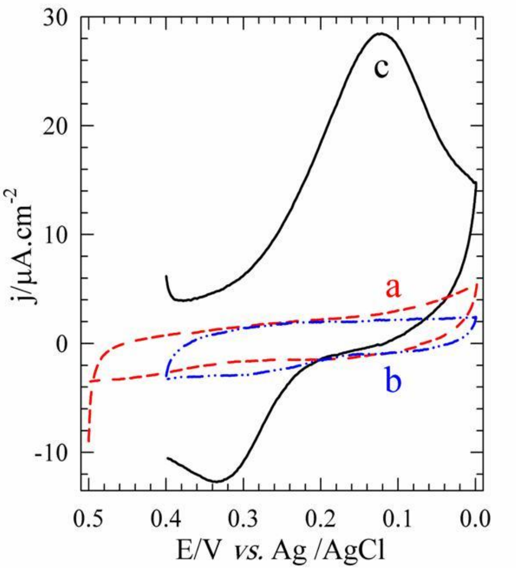

Figure 1. Cyclic voltammograms obtained on the bare Au (a) and Au-MSA SAM electrode before (b) and after (c) immersion in solution containing 20% a-MEDA and 3.0×10^{-5} mol L^{-1} iron(III) for 10 min. Preconcentration and measurement solutions conditions: ionic strength (μ= 0.1 mol L^{-1}), pH 3.0, scan rate (0.100 V s^{-1}).

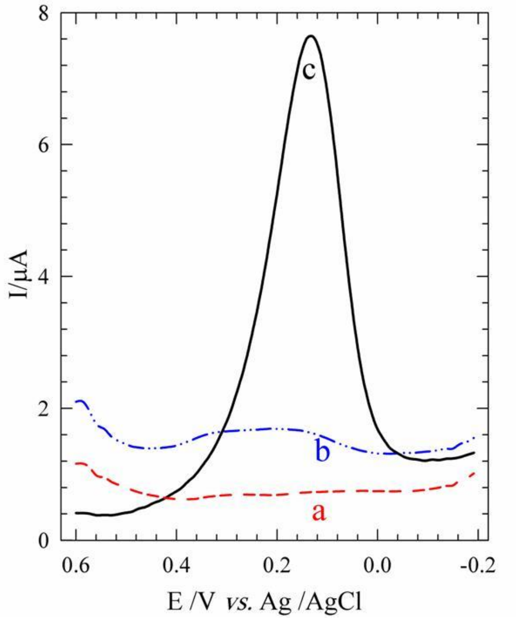

Figure 2. Square wave voltammograms obtained on bare Au (a), and Au-MSA SAM electrodes before (b) and after (c) immersion in solution pH 3.0 containing 20% a-MEDA, 0.1 M KNO_3, and 3.0×10^{-5} mol L^{-1} iron(III) for 10 min. step potential (14 mV), amplitude (40 mV), and frequency (35 Hz).

Since the electrochemical determination was carried out in an iron-free solution, curve (b) also does not show any wave for iron(III) redox reaction. Curve (c) shows a cathodic irreversible peak around +0.130 V, implying that iron(III) ions have been accumulated onto the modified surface following the electrochemical reduction to confirmed that iron(II), formed from electrochemical reduction of iron(III), has left the surface of Au-MSA SAM electrode immediately after formation, so that it has not been appropriately accessible for oxidation in reverse potential scan and shows irreversible behavior. Also, no peak was observed for the following cases: (i) the electrodes preconcentrated in iron(II) and the scans started either from positive or negative potentials, and (ii) the electrodes preconcentrated in iron(III) and the scans started from negative and continued to positive direction. The stripping square wave voltammograms obtained on bare Au (curve a) and Au-MSA SAM electrodes before (curve b) and after (curve c) preconcentration in solution pH 3.0 containing 20% a-MEDA, 0.1 mol L^{-1} KNO_3, and 3.0×10^{-5} mol L^{-1} iron(III) are shown in Fig. 2.

Potential scans were started from positive region and continued to negative direction. A clear peak was observed around +0.180 V that could be used as a base for analytical determination of iron(III). No peak was observed in the following cases: (i) the electrodes preconcentrated in iron(II) and the scans started either from positive or negative potentials, and (ii) the electrodes preconcentrated in iron(III) and the scans started from negative potential region, and then continued to positive direction. These behaviors were in good agreement with those observed by CV measurements. However, solubility product of $Fe(OH)_3$ is extremely small, which can limit determination of high concentration of iron(III) at higher pHs. Therefore, the pH for quantitative analytical method was considered and studied. According to the first round investigations, we found that both pH of the preconcentration and stripping solutions influence the response of the Au-MSA SAM toward iron(III). The pH effect of the preconcentration and stripping solutions on the current response of Au-MSA SAM was investigated in the range of 2.0–10.0. Variations of the iron(III) cathodic peak currents as a function of the preconcentration as well as stripping solution pHs, at the given conditions, are shown in Fig. 3.

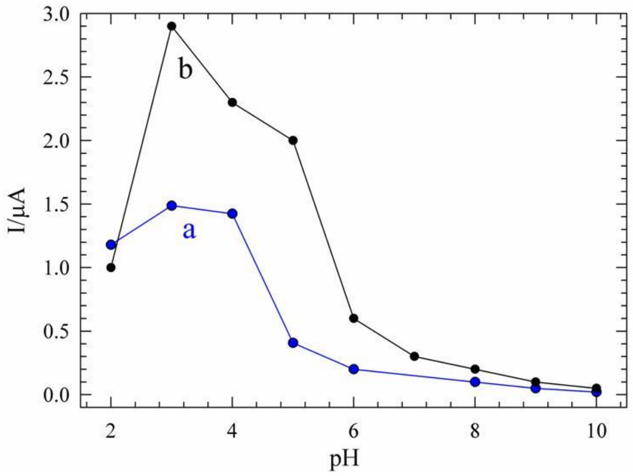

Figure 3. Variation of the stripping peak current as a pH function of the (a) preconcentration and (b) stripping solutions obtained on Au-MSA SAM preconcentrated solution, containing 20% a-MEDA, 0.1 mol L^{-1} KNO_3, and 8.90×10^{-6} mol L^{-1} iron(III), preconcentration time 10 min.

The stripping peak current increased as the pH of the preconcentration solution was increased and a maximum was appeared around pH 3.0 (Fig. 3, curve a). At upper and lower pH values the response was decreased. This behavior may be attributed to the competition of (i) the hydroxide ion for Fe^{3+} ions at higher pH and (ii) hydronium ion with Fe^{3+} for MSA SAM groups at lower pH. In the stripping step, for pH values higher than 4, the interaction between Fe^{3+} and the functionalized monolayer becomes stronger, hindering the charge-transfer kinetics between Fe^{3+} and the metallic electrode base, and at lower pH, some part of the Fe^{3+} ions leave the surface before starting the stripping step (Fig. 3, curve b). Thus, pH 3.0 was chosen for both preconcentration and stripping solutions.

3.2. Calibration and analytical application

3.2.1. Calibration and detection limit

Under the optimum conditions, the calibration curve was obtained by systematically increasing the concentration from 1.25×10^{-6} to 2.68×10^{-5} mol L^{-1} iron(III) in preconcentration solution and monitoring the response of Au-MSA-Fe^{3+} electrode in stripping solution by SWV (Fig. 4).

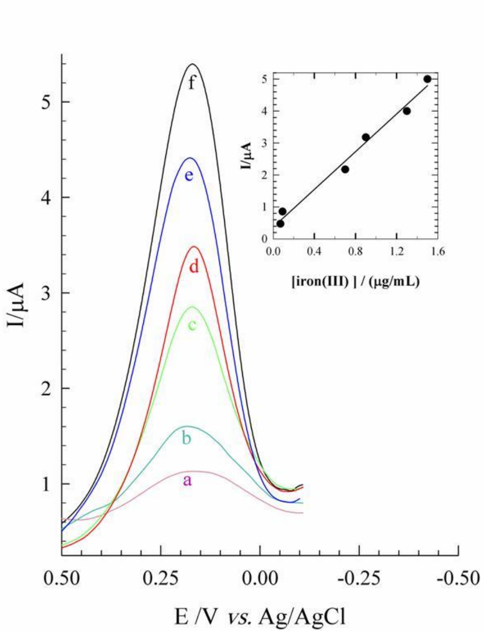

Figure 4. Square wave voltammograms, obtained on Au-MSA SAM electrode in (a) 0.07 μg mL^{-1}, (b) 0.09 μg mL^{-1}, (c) 0.7 μg mL^{-1}, (d) 0.9 μg ml^{-1}, (e) 1.3 μg mL^{-1} and (f) 1.5 μg mL^{-1} iron(III) solutions, pH 3.0 containing 20% a-MEDA, and 0.1 mol L^{-1} KNO_3, with 10 min preconcentration times. Inset shows calibration curve obtained from variation of the stripping peak current as a function of iron(III) concentrations. Stripping conditions: 0.1 mol L^{-1} KNO_3 solution (pH 3.0), potential step (14 mV), amplitude (40 mV), and frequency (35 Hz).

The peak currents were extracted and plotted versus concentration (Fig. 4, inset). The related linear regression equation was i_p/μA = 2.95 $C_{iron(III)}$/μg mL^{-1} + 0.370 and correlation coefficient was

R = 0.992. The detection limit of this electrode, calculated from the standard deviation of the background (signal equals 3S where S was the standard deviation of background for 7 measurements), was 1.94×10^{-7} mol L^{-1} iron(III), and the relative standard deviation (RSD) for n = 6 at 1.79×10^{-5} mol L^{-1} iron(III) was 3.29%. The analytical features are compared with relevant literature data in Table 1. These results show that the Au-MSA SAM modified electrode is an appropriate sensor for detection of iron(III) in a-MEDA at low concentrations.

Table 1. Comparison of the analytical results with relevant literature data

Method	Electrode	Sample	[a]LOD (mol L^{-1})	Linear range (mol L^{-1})	Ref.
[b]AdSV	[c]HMDE	Water	1.0×10^{-10}	$(0.36–3.6) \times 10^{-7}$	[32]
AdSV	HMDE	Seawater	6×10^{-10}	$(0.6–5) \times 10^{-8}$	[33]
[d]IEV	[e]NCGE	Waters	1.5×10^{-6}	$(1.5\text{-}100) \times 10^{-6}$	[34]
[f]ASV	[g](Ag/Hg)RDE	Coastal Waters	0.3×10^{-9}	$(15\text{-}90) \times 10^{-9}$	[35]
[h]ACSV	HMDE	Pore Waters	1.3×10^{-9}	$(2.6\text{-}300) \times 10^{-9}$	[36]
ASV	[i]Pt-RDE	[j]Aq. of Food Chelators	1.3×10^{-5}	$(0.026–15) \times 10^{-3}$	[37]
[k]CSV	[l](Pt-Ag)TE	Suspension	1.2×10^{-3}	$(1.8–70) \times 10^{-3}$	[38]
AdCSV	CPE	Waters	1.7×10^{-9}	$(5.6\text{-}446) \times 10^{-9}$	[39]
[m]FIA	[n]MWCNTs/Pt NPs/GCE	Lixiviated aq. from Soil	3.0×10^{-9}	$(0\text{-}10) \times 10^{-3}$	[40]
AdSV	Au-MSA SAM	20% a-MEDA	1.94×10^{-7}	$(1.25\text{-}26.8) \times 10^{-6}$	This work

(a) Limit of Detection; (b) Adsorptive Stripping Voltammetry; (c) Hanging Mercury Dropping Electrode; (d) Ion-Exchange Voltammetry; (e) Nafion Coated Glassy Carbon Electrode; (f) Anodic Stripping Voltammetry; (g) Silver/Mercury Alloy Rotating Disk Electrode; (h) Adsorptive Cathodic Stripping Voltammetry; (i) Platinum-Rotating Disk Electrode; (j) Aqueous solution; (k) Cathodic Stripping Voltammetry; (l) Platinum-Silver Twin Electrode; (m) Flow Injection Amperometry; (n) Multiwalled Carbon Nanotubes /Pt nanoparticles modified glassy carbon electrode.

3.2.2. Interferences

Possible interferences in the determination of 1.80×10^{-6} mol L^{-1} iron(III) in 20% a-MEDA were examined under the optimum experimental conditions. The interfering effect is defined as the concentration of interfering species that can change the electrode response toward the analyte by more than $3S_A$ where S_A is the standard deviation of the replicate analyte measured signal. The results showed that the peak current of 1.80×10^{-6} mol L^{-1} iron(III) was not affected by 10^9-fold of Mg^{2+}, 10^6-fold of Cd^{2+}, Mn^{2+}, Cr^{6+}, 10^5-fold of Zn^{2+}, 10^3-fold of Ni^{2+}, 10^2-fold of C_O^{2+}, and 10-fold Cu^{2+} for SWV measurements. However, when 0.01 mol L^{-1} of pyrophosphate ($P_2O_7^{4-}$) was added to the preconcentration solution, the interferences of Cu^{2+} up to 10^{-5} mol L^{-1} were eliminated. Also when 0.01 mol L^{-1} $Mg(OH)_2$ was added to the preconcentration solution, the interferences of Co^{2+} up to 10^{-4} mol L^{-1} was eliminated.

3.2.3. Analysis of iron(III) in a-MDEA samples

To further demonstrate the practicality of the proposed sensor, the recovery tests were studied by addition of different quantities of iron(III) into solutions containing different amounts of a-MDEA. The results were summarized in Table 2. The recoveries were from 93.1% to 104.1%.

Table 2. Recovery studies of iron(III) in solutions containing different amounts of a-MDEA

a-MEDA (%)	Added (ng mL^{-1})	Founded (nmol L^{-1})	Recovery (%)	^{a}RSD (%)
-	80.0	83.3	104.1	3.12
20	80.0	76.2	95.2	3.34
40	80.0	83.1	103.9	4.17
60	80.0	77.9	97.4	5.48
80	80.0	75.8	94.7	5.71
80	160.0	149.0	93.1	5.92

[a] Calculated for n = 5 measurements

The proposed sensor was also used for direct determination of iron(III) in real solutions containing a-MDEA utilized in sweetening plant of gas treating industry samples. Standard additions of iron(III) were made by stepwise addition of the same concentrations of iron(III) into the real sample containing 0.1 mol L^{-1} KNO_3, pH 3. The stripping square wave voltammograms obtained in these conditions are displayed in Fig. 5. The peak currents were extracted and plotted *versus* concentration (Fig. 5, inset).

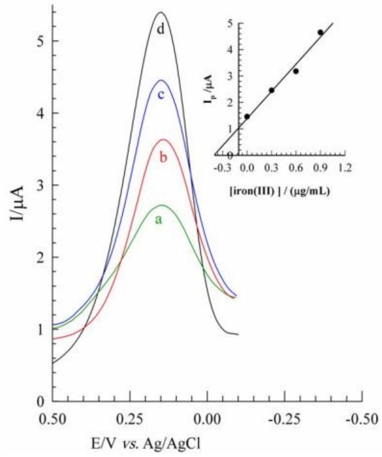

Figure 5. Square wave voltammograms obtained on Au-MSA SAM electrode preconcentrated in real sample solution containing a-MDEA utilized in sweetening plant of gas treating industry solutions prepared by standard addition of 0.00 μg mL^{-1} (a), 0.3 μg mL^{-1} (b), 0.6 μg mL^{-1} (c), and 0.9 μg mL^{-1} (d) of iron(III). Inset shows calibration curve obtained from variation of the stripping peak current as a function of iron(III) concentration in standard addition method. Stripping conditions are the same as in Fig. 4.

The related linear regression equation was $i_p/\mu A = 3.43\ C_{iron(III)}/\mu g\ mL^{-1} + 1.39$ and correlation coefficient was R = 0.990. When $i_p = 0$, the concentration of iron(III) in the real sample was obtained. The result (0.406 $\mu g\ mL^{-1}$) was in good agreement with the value of iron(III) obtained by atomic absorption spectroscopy method (0.436 $\mu g\ mL^{-1}$). The results indicated that the proposed method is highly accurate, precise and reproducible. It can be used for direct analysis of iron(III) in non-aqueous real samples. When the sensor was not in use, it was stored at water in oxygen free condition. No obvious decrease in the response of the sensor was observed in the first week storage. After a month storage period, the sensor retained 93 % of its initial current response. Anyway, the washing of the cell containing the electrode with distilled water did not produce any response dropping, because of the high stability of the sensor due to covalent immobilization of the MSA on the gold electrode.

4. CONCLUSIONS

In this work, we described the use of 2-mercaptosuccinic acid self assembled monolayers on polycrystalline gold electrode as a selective and sensitive sensor for quantitative determination of iron(III) in highly contaminated nonaqueous industrial samples by adsorptive stripping square wave voltammetry. The sensor benefits of simple fabrication, low detection limit, and does not suffer from drawbacks related to the mercury electrode. Applicability of the electrode for analysis of the real samples was successfully tested by determination of iron(III) in different quantities of a-MDEA and a-MDEA utilized in sweetening plant of gas treating industry samples with interference of the sample matrix. The results indicated that the proposed method is highly accurate, precise and reproducible. The responses also exhibited antifouling property compared to the bare gold electrode.

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