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Cyclic voltammetry study of the electrocatalytic reduction of sevoflurane by a cobalt(III) Schiff base complex in the presence of oxygen

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ABSTRACT

The electrocatalytic reduction of sevoflurane has been carried out at a platinum electrode using cyclic voltammetry. It was found that the sevoflurane molecule is non-electroactive in the potential region between 0.5 and −2.3 V vs. Ag/AgCl (satd.) electrode in dimethyl sulphoxide (DMSO) solution. However, the results show that, in a mixture with oxygen, cobalt(III) Schiff base complex (CoLOAC), and sevoflurane, an irreversible peak appears at about −1.77 V vs. Ag/AgCl (satd) electrode. It was observed that the peak height increases linearly with sevoflurane concentration up to 7.5 mM. The detection limit is estimated to be 0.5 mM.

1. Introduction

Fluorinated ethers (enflurane, isoflurane, sevoflurane and desflurane) are replacing older anesthetic agents, such as ether, chloroform and more recently halothane in modern anesthesiology because the fluorinated ethers have desirable physical properties. They have lower blood-gas solubility and lower metabolism [1]. Sevoflurane (fluoromethyl 2,2,2-trifluoro-1-[trifluoromethyl] ethyl ether) belongs to the group of inhalation anesthetic agents commonly used in clinical practice during the last few years. It is a sweet smelling, colorless, volatile, non-flammable, highly fluorinated methyl isopropyl ether used for inhalation anesthesia [2]. Sevoflurane was first synthesized in 1968 at Baxter Laboratories [3]. However, it has only been clinically employed on a large scale since 1990. Due to its quick inhalation induction, rapid recovery, nonpungent odor, and nonirritation of the respiratory system, sevoflurane is a suitable anesthetic agent for mask induction in children and adults [4,5]. Monitoring the concentration of exhaled inhalation anesthetic agents, in the operating room's environment, where operating room personnel may be exposed, is essential. In addition, the concentration of these agents in a patient's blood must be monitored to ensure the patient's safety. The monitoring has been undertaken by measuring the concentration of these agents by different methods in the last two decades [6–17]. However, these methods require complicated and expensive instrumentation, professional operators, a time-consuming detection process, and complex pre-treatment steps. Thus, a simple and inexpensive electrochemical detection of these agents in blood and inhaled air is very important.

Reviewing the literature revealed that there are only a few studies concerning the electrochemical reduction of fluorinated ethers. Most attempts on electrochemical studies of fluorinated ethers have been performed by Compton et al. and by Hahn et al. [18–25]. Illustrative examples of these works focus on the development of amperometric sensors for estimation of anesthetic agents during surgery. Recently, the electro-reduction of the stereoisomers enflurane [21,22], isoflurane [23], and sevoflurane [24] were reported at Au, Ag, and Cu microelectrodes in DMSO. Hahn and co-workers at a variety of microelectrode substrates (Au, Ag and Cu) in DMSO investigated the electrochemical reduction of sevoflurane individually and as a component of a simple gas mixture with oxygen. Their results showed that direct electro-reductions of sevoflurane require potentials more negative than −2.5 V vs. Ag.

The large overpotential associated with the sevoflurane reduction is the major challenge, which calls for the development of a high performance catalyst. Great efforts to design and develop molecular catalysts have been made. Cobalt complexes are used effectively in many electrochemical studies owing to their good catalytic properties. For instance, cobalt Schiff base complexes and cobalt porphyrins have been used for the electrocatalytic reduction of dioxygen [26–28], sulfide [29], carbon dioxide [30], nitric oxide [31], chlorobenzenes [32], alkyl halide [33], ethyl chloroacetate [34], penicillamine [35] and so on.

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In the present study, we present the first evidence and an evaluation of homogeneous electrocatalytic reduction of sevoflurane by a novel cobalt(III) Schiff base complex (CoLOAc) in the presence of molecular oxygen. The structures of sevoflurane and CoLOAc are shown in Fig. 1. The electrocatalytic activity of CoLOAc toward the reduction of sevoflurane was studied using the cyclic voltammetric technique.

2. Experimental

2.1. Chemicals

The anesthetic agent, sevoflurane, was purchased from Abbott Laboratories Ltd. Reagent-grade tetra-*n*-butylammonium perchlorate (TBAP, Fluka) and HPLC grade DMSO (Merck) were used as received. All reagents and solvents were used without further purification. Sevoflurane was introduced into the electrolyte solution in liquid form by gravimetric aliquots before each voltammetric measurement. Nitrogen gas with 99.999% purity (Sabalan, Tehran, Iran) was used to deaerate the solutions.

2.1.1. Preparation of CoLOAc

The CoLOAc was synthesized and characterized by spectroscopic methods and elemental analysis, as reported elsewhere [36]. Briefly, a solution of 1,2-diaminocyclohexane (0.01 mol) in methanol (15 mL) was added to a stirring solution of salicylaldehyde (0.02 mol) in methanol (15 mL). The reaction mixture was refluxed with stirring for 0.5–3 h. The precipitate was filtered off and the filtrate washed with cold methanol. The product (Schiff base ligand (Fig. 1b)) was recrystallized from methanol for further purification.

Schiff base ligand: Yellow crystal; yield 95%, m.p. = 96 °C, ^{1}H NMR($CDCl_3$, 250 MHz) δ = 1.46–1.57(m, 2H), 1.73–1.99(m, 6H), 3.3–3.4(t, 2H), 6.8–6.93(m, 4H), 7.16–7.30(m, 4H), 8.29(s, 2H), 13.35(s, br, 2H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ = 24.5, 33.5, 73, 117, 119, 131.8, 132.1, 132.6, 161.3, 165; IR (KBr, cm^{-1}) ν 764(s), 849(s), 797(m), 1145(s), 1203(m), 1280 (vs. C—O), 1415(s), 1458(s), 1500(s), 1577(s), 1627(s, C═N), 2850(m), 2913(s), 3030(w), 3475(br, OH).

A solution of $Co(OAc_2)$. $4H_2O$ (0.212 g, 1.2 mmol) in absolute ethanol (20 mL) was added to suspension of 1 mmol Schiff base ligand in 20 mL absolute ethanol under nitrogen atmosphere. The mixture was refluxed for 3 h and allowed to cool to room temperature. The precipitates were separated from the solution by filtration washed with ethanol and dried under vacuum to give cobalt(II) Schiff base complex as dark-brown crystal. This cobalt(II) Schiff base complex was effective oxidized to cobalt(III) Schiff base complex (CoLOAc) by following procedure. The cobalt(II) Schiff base complex 1 mol and acetic acid 75 mL were stirred in toluene 10 mL under air for 1 h. The solvent were removed in vacuum and the dark-brown residue was recrystallized in dichloromethane.

CoLOAc: Deep brown solid, IR (kBr, cm^{-1}) ν 650(m), 785(m), 844(s), 1080(s), 1122(s), 1165(s), 1220(br), 1335(m), 1440(s), 1530(s), 1600(br).

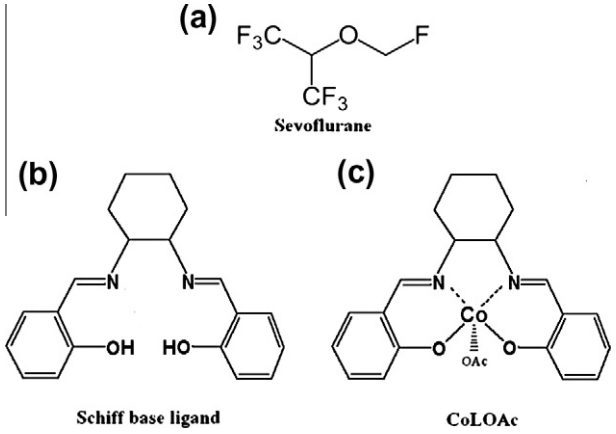

Fig. 1. Structures of (a) sevoflurane, (b) Schiff base ligand and (c) cobalt(III) Schiff base complex.

2.2. Apparatus

Electrochemical measurements were carried out with a μAutolab(III) computer-controlled potentiostat and run with the General Purpose Electrochemical System (GPES) software. A three-electrode system, including a platinum working electrode and Ag/AgCl reference electrode and platinum counter electrode, was employed. A Metrohm platinum disk electrode (geometric area of 0.0314 cm^2) was used as working electrode. All experiments were carried out with the electrochemical cell immersed in a thermostat at 25 ± 1 °C.

3. Results and discussion

3.1. Electrochemical properties of CoLOAc complex in DMSO solution

Cyclic voltammograms (CVs) of the CoLOAc in DMSO solution at various scan rates on the surface of a Pt disk electrode are shown in Fig. 2. This complex exhibits two well defined reversible voltammetric responses corresponding to the Co(III)/Co(II) and Co(II)/Co(I) couples, as was reported earlier for some other complexes [26,37]. The formal potentials $E^{o\prime}$, taken as the mid-point of the

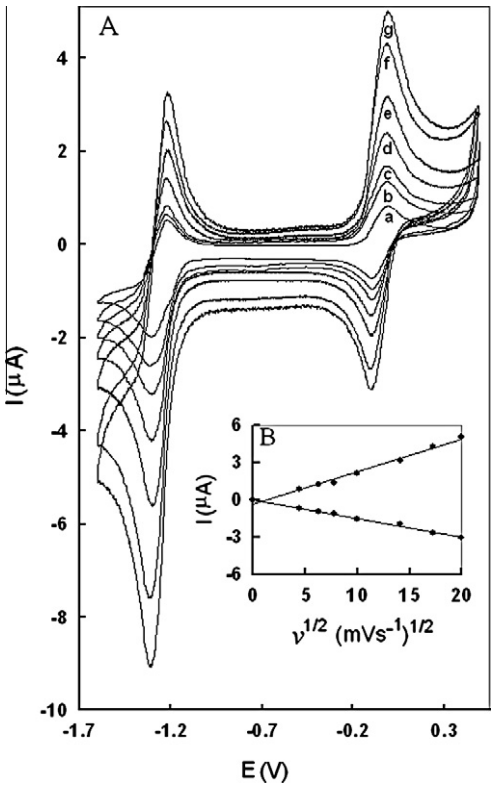

Fig. 2. (A) Cyclic voltammograms for 1.0 mM CoLOAc on a Pt disk electrode (1-mm diameter) in an DMSO solution containing 0.05 M TBAP at scan rates: (a) 20; (b) 40; (c) 60; (d) 100; (e) 200; (f) 300; (g) 400 mV s^{-1}. (B) Plots of anodic and cathodic peak currents against (sweep rate)$^{1/2}$ for both anodic and cathodic peaks for first couple at sweep rates of 20–400 mV s^{-1}.

anodic and cathodic peak potentials, are located at about −0.055 and −1.26 V vs. Ag/AgCl (satd.), 0.05 M TBAP in DMSO, as the reference electrode. Plots of peak currents vs. (scan rate)$^{1/2}$ for both anodic and cathodic peaks are linear for sweep rates of 20–400 mV s^{-1} (inset of Fig. 2 for first couple). For both couples, the ratios of anodic to cathodic peak currents are nearly one and the separation between the cathodic and anodic peak potentials is about 100 mV at low scan rate (20 mV s^{-1}). The electrode processes were quasi-reversible, with ΔEp, greater than the $(59/n)$ mV expected for a reversible system. Similar results have been reported for several cobalt Schiff base and porphyrin complexes in nonaqueous media [38–41].

The electrochemical behavior of CoLOAc in DMSO in the absence (scan a) and presence of molecular oxygen (scan b) is shown in Fig. 3A. As seen over a potential range of −0.3 to −1.6 V, the CV of DMSO solution containing the CoLOAc and saturated with O_2, shows a single reduction peak and a corresponding oxidation peak. The ability of the cobalt(II) complexes to bind molecular dioxygen has been known for quite a long time [41,42]. It seems that the adduct formed in the presence of O_2 is non-electroactive in the negative potential (curve b of Fig. 3A) and no peaks are appeared with the cobalt(II)–cobalt(I) process at ca. −1.3 V. To be able to characterize the observed redox couple, cyclic voltammetric measurements were carried out under identical experimental conditions in the CoLOAc-free DMSO solution (Fig. 3B). The results show that the same redox couple appears over a potential range of −0.3 to −1.6 V. On the basis of the above results, it can be concluded that the observed redox couple over a potential range of −0.3 to −1.6 V is related to the oxygen redox reaction.

3.2. Electrochemical behavior of sevoflurane in the presence of CoLOAc and oxygen

Preliminary studies to show the catalytic activity of the CoLOAc complex toward the electro-reduction of sevoflurane were made using cyclic voltammetry. Fig. 4 (scan a) shows the CV for the electrochemical behavior of molecular oxygen in the presence of sevoflurane at a Pt electrode in DMSO solvent containing 0.05 M TBAP. As shown, an irreversible reduction peak for molecular oxygen in the presence of sevoflurane is observed with a peak potential of −0.86 V vs. Ag/AgCl (satd.) electrode. It also shows that sevoflurane is non-electroactive between the potential region of 0.5 and −2.3 V vs. Ag/AgCl (satd.) electrode. This behavior is in agreement with previous results [21–24]. The CV experiments on sevoflurane in the presence of the CoLOAc and the effect of molecular oxygen

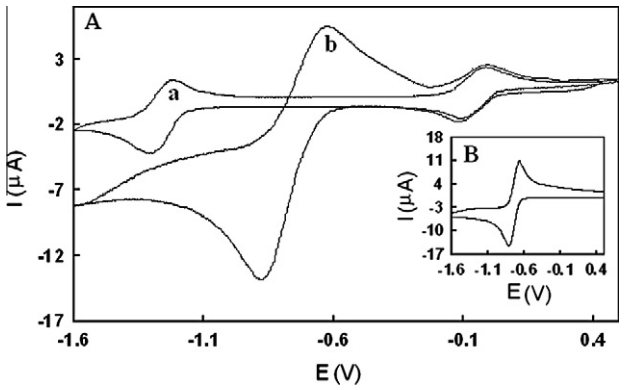

Fig. 3. (A) Representative cyclic voltammograms for 1.0 mM CoLOAc at a Pt disk electrode (1-mm diameter) in DMSO solution containing 0.05 M TBAP in the absence (a) and presence (b) of oxygen. (B) Cyclic voltammograms of the DMSO solution containing 0.05 M TBAP and saturated with oxygen. Scan rates were 50 mV s^{-1}.

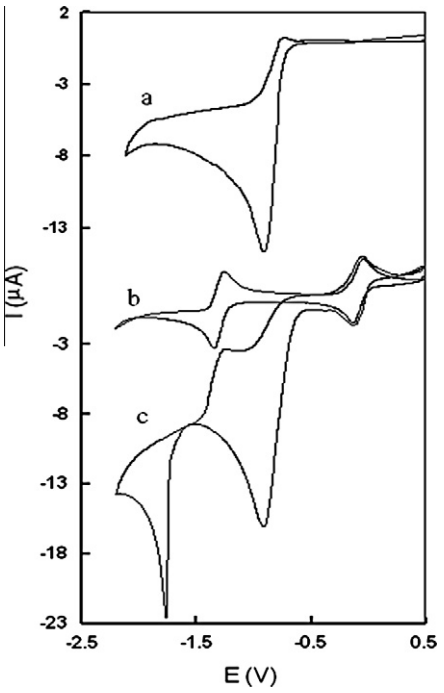

Fig. 4. Influence of oxygen on the detection of sevoflurane. Trace (a), cyclic voltammogram for DMSO solution containing 0.05 M TBAP in the presence of 7.5 mM sevoflurane and saturated with oxygen. Trace (b) cyclic voltammogram for DMSO solution containing 0.05 M TBAP in the presence of 7.5 mM sevoflurane, 1 mM CoLOAc, and in the absence of oxygen. Trace (c) as trace (b) in the solution saturated with oxygen. Scan rates were 50 mV s^{-1}.

on the CVs are shown in Fig. 4 (traces b and c). As can be seen in Fig. 4, scan b, the sevoflurane in the presence of CoLOAc is non-electroactive in the potential range studied (i.e., 0.5 to −2.3 V vs. Ag/AgCl). However, a sharp cathodic peak with a peak potential of −1.77 V vs. Ag/AgCl (satd.) is obtained after adding molecular oxygen to the solution (see Fig. 4, scan c). In addition, this behavior does not correspond to effects of pure sevoflurane or oxygen as have been reported previously [21–24]. Comparison of the voltammograms obtained in Fig. 4 (traces b and c) shows that the cathodic peak at −1.77 V (observed only in the presence of molecular oxygen) may be related to the reduction of sevoflurane.

Nucleophilic reactions and electrochemical studies of the reactivity of electrogenerated superoxide ion with alkyl halides have been investigated in nonaqueous media [43–45]. Based on the results obtained on the electro-reduction of sevoflurane in the presence of oxygen by Moorcroft et al. [22] and by Floate et al. [23,25], the following ECE-type mechanism and one-electron process in rate determining step is represented for reaction between sevoflurane and superoxide ion (Eqs. (1)–(3)).

$$O_2 + e^- \rightarrow O_2^{\cdot -} \quad (1)$$

$$R - F + O_2^{\cdot -} \rightarrow R - O_2^{\cdot -} + F^- \quad (2)$$

$$O_2^{\cdot -} + R - O_2^{\cdot} + e^- \rightarrow R - O_2^- \quad (3)$$

where R = $F_3C{-}C(CF_3)H{-}O{-}CF_2H$ (sevoflurane)

According to our results, the observed peak at −1.77 V does not correspond to binary mixtures including Schiff base complex/sevoflurane, oxygen/sevolfurane or schiff base complex/oxygen. Consequently, we can conclude that there is an interaction between the sevoflurane, superoxide species and the electrogenerated Co(I). Therefore, it seems that the following reaction probably occurs in the system under study here.

$$[LCo(I)] + O_2 + e^- \rightarrow [LCo(I)O_2^{\cdot}]^- \quad (4)$$

$$[LCo(I)O_2^{\cdot}]^- + R - F \rightarrow [LCo(I)RO_2^{\cdot}] + F^- \quad (5)$$

$$[LCo(I)RO_2^{\cdot} + e^- \rightarrow [LCo(I)RO_2]^- \quad (6)$$

$$[LCo(I)RO_2]^- + [LCo(I)] \rightarrow R{-}OO - R + [LCo(II)] \quad (7)$$

or

$$[LCo(I)RO_2]^- + R - F \rightarrow [LCo(I)] + R - OO - R + F^- \quad (8)$$

where LCo is schiff base complex and $R = F_3C{-}C(CF_3)H{-}O{-}CF_2H$ (sevoflurane).

3.3. Effect of sevoflurane concentration

Fig. 5A illustrates a series of cyclic voltammograms recorded with the Pt electrode in the presence of different concentrations of sevoflurane ranging from 0 to 9 mM in DMSO containing 1 mM CoLOAc. The correlation between the reduction peak intensity and sevoflurane concentration is shown in Fig. 5B. As is obvious, the plot is linear up to a concentration of 7.5 mM. The linear regression equation was expressed as: $I(\mu A) = 8.01 + 1.82C_{sevoflurane}$ (mM) ($R^2 = 0.999$), and the detection limit was 0.5 mM (three times of the ratio of signal to noise).

Fig. 6A shows cyclic voltammograms for the electro-reduction of 7.5 mM sevoflurane in a 0.05 M TBAP DMSO solution, containing 1 mM CoLOAc, 7.5 mM sevoflurane and saturated with O_2 at various scan rates. As is obvious, the peak potential for the reduction of sevoflurane shifts to increasingly negative potentials with increasing scan rate. This result suggests a kinetic limitation in the reduction of sevoflurane in the presence of CoLAOc and oxygen in DMSO. It is worth mentioning that, in all nonaqueous electrochemical investigations, a supporting electrolyte (TBAP) of high concentration (0.05 M) was employed; thus, the possibility of potential shift to more positive values due to higher ohmic drop is expected to be negligible. As is seen from Fig. 6B, there is a linear correlation between the cathodic current and $v^{1/2}$ up to 100 mV s^{-1}, suggesting that the kinetics of the overall process is controlled by mass transport of sevoflurane from the bulk solution to the electrode surface.

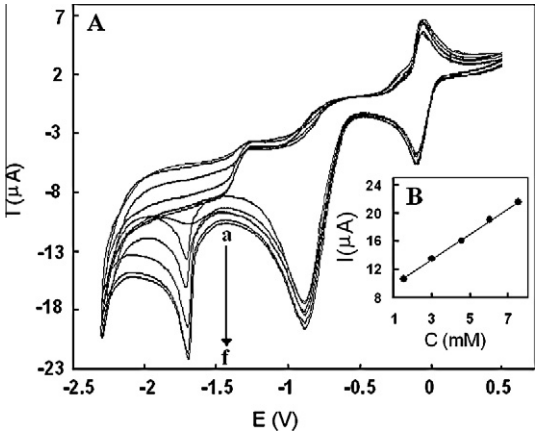

Fig. 5. (A) Cyclic voltammograms for DMSO containing 0.05 M TBAP in the presence of 1 mM CoLOAc and saturated with oxygen at different concentrations of sevoflurane: (a) 1.5, (b) 3.0 (c) 4.5, (d) 6.0, (e) 7.5 and (f) 9 mM. (B) Plot of peak currents vs. sevoflurane concentration. Scan rates were 50 mV s^{-1}.

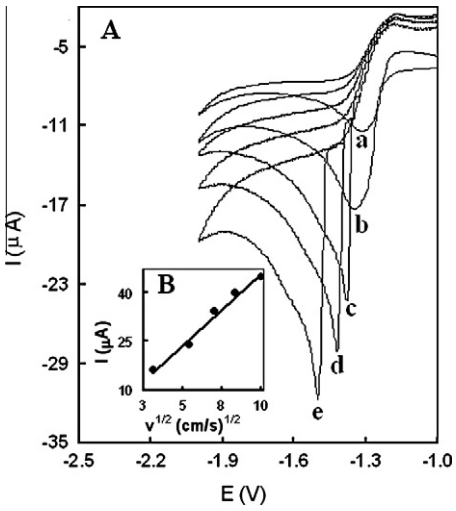

Fig. 6. (A) Cyclic voltammograms for DMSO containing 0.05 M TBAP in the presence of 7.5 mM sevoflurane, 1 mM CoLOAc, and saturated with oxygen at various scan rates: (a) 10, (b) 30, (c) 50, (d) 70, and (e) 100 mV s^{-1}. (B) Plot of I_p vs. $v^{1/2}$.

4. Conclusion

In this paper, we have illustrated the effect of a cobalt Schiff base complex toward sevoflurane in presence of molecular oxygen. Resulting voltammograms obtained with a Pt electrode in a tertiary mixture containing sevoflurane, molecular oxygen, and cobalt complex indicate a well defined peak at approximately −1.77 V vs. Ag/AgCl (sat'd KCl). It seems that the appearance of this new cathodic peak is associated with the presence sevoflurane. Sevoflurane reduction in the presence of oxygen and cobalt complex is complicated by the reaction between electrogenerated superoxide, Co(I) species and the sevoflurane. A dependence of the peak current on the concentration of sevoflurane is linear up to 7.5 mM. This study is a continuation of developing a rapid, sensitive, and inexpensive electroanalytical method for the determination of sevoflurane, which would be applicable in clinical practice.

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