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The anodic behaviour of Ebonex® in oxalic acid solutions

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Abstract

A study of the oxidation of aqueous solutions of oxalic acid in various supporting electrolytes, at an Ebonex® anode is reported. From data obtained by cyclic voltammetry, linear sweep voltammetry, chronopotentiometry, controlled potential coulometry and gas chramatography it has been shown that oxalic acid oxidation at an Ebonex® anode, does not proceed, to any significant extent. Data collected using different electrolytes showed that sulphuric acid supported higher current densities at similar electrode potentials. Ebonex® is essentially a selective oxygen evolver and is thus a suitable anode for the electroreduction of oxalic acid to glyoxylic acid in an undivided cell. The behaviour of a platinized titanium anode is also reported and this material has similar electrochemical characteristics to Ebonex® in aqueous oxalic acid solutions.

1. Introduction

The electrosynthesis of glyoxylic acid from the electroreduction of oxalic acid has received considerable attention as a commercial process [1, 2]. As indicated in the literature [1, 2], a major cause of low performance efficiency, usually met in the practical operation, is attributed to cathode fouling, which is mainly associated with the oxalic acid feedstock and anode material. A major factor that has limited the adoption of the electrosynthesis of glyoxylic acid from oxalic acid has been the cost of the cell. In most applications a membrane electrolyser using a cation exchange membrane is used to prevent oxidation of reactant and product. The adoption of an undivided reactor would significantly reduce the cost provided that the oxidation of product and reactant can be suppressed. The selection of anode material, as an oxygen evolver, is a key factor to the possible success of the process.

Lead, graphite, platinum, dimensionally stable anodes, and lead dioxide on titanium have all been used as anode materials in divided cells during oxalic acid electroreduction. It was found that, using platinized titanium anode in an undivided cell, glyoxylic acid current efficiency was affected significantly by the anode current density, for example, 46% after operating 5 h at an anodic current density of 6250 A m^{-2} compared with only 20% at 333 A m^{-2} [2]. Thus, it is important to find a suitable anode material which should, on the one hand, provides a high current density for high productivity and, on the other hand, should be only an oxygen evolver, that is, it does not oxidize oxalic acid or glyoxylic acid to a significant extent, to ensure high yield and selectivity.

Studies of the evolution of CO$_2$ during the anodic oxidation of oxalic acid (or corresponding derivatives of) have concerned with platinum. Using platinized platinum anodes in HClO$_4$ electrolyte it has been shown that [3] glyoxylic acid can be oxidized to oxalic acid and carbon dioxide in a region of potential between 0.6 and 1.0 V vs NHE and that oxalic acid can also be oxidized to carbon dioxide at similar potentials. The oxalic acid oxidation reaction was considered to proceed according to the following mechanism [4]:

$$(\mathrm{HOOC})_2 = \mathrm{HC_2O_4^-} + \mathrm{H^+} \tag{1}$$

$$\mathrm{HC_2O_4^-} = (\mathrm{HC_2O_4})^-_{\mathrm{ads}} \tag{2}$$

$$(\mathrm{HC_2O_4})^-_{\mathrm{ads}} - \mathrm{e^-} \rightarrow (\mathrm{HC_2O_4^\bullet})_{\mathrm{ads}} \tag{3}$$

$$(\mathrm{HC_2O_4^\bullet})_{\mathrm{ads}} \rightarrow (\mathrm{HCO_2^\bullet})_{\mathrm{ads}} + \mathrm{CO_2} \tag{4}$$

$$(\mathrm{HCO_2^\bullet})_{\mathrm{ads}} - \mathrm{e^-} \rightarrow \mathrm{CO_2} + \mathrm{H^+} \tag{5}$$

It is generally accepted that adsorption of oxalic acid, or derivatives of, at the anode surface is important for the oxidation of oxalic acid [4–10]. Adsorption of (HOOC)$_2$ or oxalate was considered as a necessary condition for the oxidation at Pt anode [4]. The rate-determining step of the electrooxidation of oxalic acid at a Pt electrode is step (3), that is, the discharge of the anion chemisorbed on the surface of the Pt electrode, with the transfer of one electron [5]. It was also reported that [6] oxalic acid adsorption took place on a smooth Pt electrode surface, in 0.5 M H_2SO_4 solution, in the entire potential range of 0 to 3 V vs NHE, which determined the extent of the oxalic acid oxidation.

As a result of the presence of other competing adsorption reactions, in contrast to many other organic compounds, oxalic acid oxidation is completely suppressed at sufficiently high potential, that is, oxidation of oxalic acid begins at potentials more negative than those at which oxygen evolution begins. There is a reported marked inhibition of the oxidation of oxalic acid at a Pt electrode by adsorbed oxygen, particularly in the range of potentials >1.1 V vs NHE [4, 7, 8]. Even at low potentials, oxalic acid oxidation was appreciably inhibited in concentrated H_2SO_4 solution ($\geqslant 1$ M) due to the effect of sulfate-ion adsorption [4]. The decreased oxidation rate of oxalic acid in the presence of adsorbed additives, such as benzene, 1,3-butadiene, and 3-cyclohexene, was also observed [9].

Another important factor affecting oxalic acid oxidation is pH of the electrolyte which also relates to the competing adsorption processes. Oxalic acid oxidation is significantly suppressed in alkaline media [8] or even not observed [4], which was explained by the fact that with increasing pH, the potential range of oxygen evolution is displaced towards more negative values [4]. It was shown that the oxalic acid oxidation rate was a maximum at values of pH when oxalic acid was in the form of the mono-anion. The systematic analysis of the effect of pH revealed that [5], with a rise in the pH, strong chemisorption of oxygen, displaced the chemisorbed oxalate anions from the anode surface, leading to an inhibition of oxalic acid oxidation, in the potential region of the oxidation of oxalic acid, greatly lowering the rate of the oxidation, and, in the extreme, suppressing it completely. Even in acidic solutions of oxalic acid no appreciable oxidation was observed on a highly oxidized platinum electrode.

Based on the above research results, evidently, CO_2 evolution from oxalic acid oxidation depends on numerous factors such as the electrode characteristics, for example, the type and the surface state; the media (pH) etc. To prevent oxalic acid oxidation to CO_2, a suitable procedure is to select an anode material which is an highly efficient oxygen evolver. In this context Ebonex® was proposed as a possible candidate anode material with suitable oxygen evolution characteristics and material stability during the anodic oxidation of oxalic acid. Ebonex® is a magnelia phase sub oxide of titanium (typically Ti_4O_7).

2. Experimental details

The electrochemical experiments were carried out in a three-compartment H-cell divided by a glass frit which contained an Ebonex® plate anode or a platinized titanium anode (Pt/Ti) with geometric surface area of 1 cm^2 for cyclic voltammetry (CV), linear sweep voltammetry (LSV) and chronopotentiometry (CP) or 10.8 cm^2 for controlled potential coulometry (CPC). The cathode material was a lead plate with geometric area of 15 cm^2.

In CPC experiments, the anolyte chamber of the cell was connected to a gas collector. Both the anolyte chamber and the gas collector were isolated from the atmosphere. The reference electrode was a saturated calomel electrode (SCE), connected to the cell via a capillary salt bridge. All potentials reported are referred to this reference electrode. The electrolytes employed were either 0.1 M H_2SO_4 solution, 1 M H_2SO_4 solution, 1 M Na_2SO_4 solution, or 1 M NaH_2PO_4 solution with or without 0.1 M oxalic acid and 0.1 M oxalic acid alone.

All solutions were prepared with Analar-grade chemicals (BDH) and deionized (Millipore) water obtained with Milli-U10 water purification system. Before CV, LSV, and CP measurements, the anolyte was deaerated with pure nitrogen gas for 20 min. To avoid interference in electrolysis product analysis, no nitrogen was passed during the CPC experiments. The electrolyte was stirred using magnetic stirrers during the CPC experiments.

The electrochemical experiments were performed with a model 273 EG&G Princeton potentiostat/galvanostat controlled with model 270/250 Research Electrochemistry Software (4.11). Cyclic voltammograms and linear sweep voltammograms were obtained at various scan rates. Chronopotentiometric measurements were performed at different current densities: 5, 10, 20, 30 and 40 mA cm^{-2} and used to obtain the steady-state polarization. Controlled potential coulometric experiments were performed at different anode potentials and the charge passed was recorded. All measurements were carried out at ambient temperature (23 ± 0.5 °C).

Gas samples produced by the electrolysis were analysed using 8A Gas Chromatography controlled by C-R6A Chromatopac computer apparatus (Shimadzu Corporation, Kyoto, Japan). Two chromatography columns, a molecular sieve 5A (column 1, mesh size 40–60) and a Chromosorb 101 (column 2, mesh size 80–100), were used for analyses of oxygen and carbon dioxide, respectively. Helium (99.9990% purity, BOC Gases) was used as the carrier gas, which was split into two before entering the two columns. The gas flow rate were set at 30 and 35 cm^3 min^{-1} for the column 1 and 2, respectively. The inject port temperature and the column oven temperature were preset at 80 °C and 40 °C, respectively. A 1 ml sample was injected into each column in turn for full resolution of its composition. Standard samples of oxygen (99.99% purity, BOC Gases) and carbon dioxide (99.98% purity, BOC Gases) were run through the columns and the peak area for a given amount was recorded and used to construct standard curves. The retention times under the experimental conditions were 1.655 (±0.002) and 1.214 (±0.002) min for oxygen and carbon dioxide, respectively. The peak areas of the various components from the gas samples (1 ml each) collected during the electrolysis using a syringe, if any, were identified and compared to the standard curves to determine their absolute quantity.

3. Results and discussion

3.1. *CV, LSV and CP in 0.1 M (HCOO)$_2$ + 1 M H_2SO_4 solution*

The typical cyclic voltammograms obtained on Ebonex® plate anode for electrolyte compositions of 0.1 M $(HOOC)_2$ in 1 M H_2SO_4 solution or 1 M H_2SO_4 solution alone at a scan rate of 1 mV s^{-1} are illustrated in Figure 1. The cyclic voltammetric scan started at 0.6 V vs SCE and was swept first in the anodic direction. Below 1.0 V there was little anodic response in both media. The current increased sharply after 1.1 V, and then again, after 1.65 V. The scan was reversed at 1.8 V which resulted in a steep fall in current, virtually along the same path as the anodic scan, especially in 1 M H_2SO_4 solution. Below approximately 1.65 V vs SCE, Ebonex® exhibited similar anodic behaviour in both media. The rapid rise in the anodic current above 1.1 V, is predominately due to the O_2 evolution reaction, probably associated with the oxidation of the oxygen-containing species adsorbed on the electrode surface at less positive potentials. This was in contrast to the direct oxidation of the oxygen-containing species from bulk solution, at more positive potentials (above 1.65 V vs SCE) via a direct electron transfer mechanism leading to a rapidly rising current density:

$$H_2O - 2\,e^- \rightarrow \tfrac{1}{2}O_2 + 2\,H^+ \tag{6}$$

It was observed that the current density was higher in 1 M H_2SO_4 than that in 0.1 M $(HOOC)_2$ + 1 M H_2SO_4, which may have been due to the effect of oxalic acid adsorption on the anode surface. The same behaviour occurs when the H_2SO_4 concentration was 0.1 M instead of 1 M, as shown later.

Generally speaking, there was no large difference in the CVs in both electrolyte media which, in combination with other experimental evidence discussed later, suggests that the major reaction at the Ebonex® plate anode is O_2 evolution. CVs performed at higher scan rates exhibited similar anodic behaviour to that shown in the Figure 1, with only moderate increases in current density especially at low potentials. This behaviour also indirectly suggests that the principal process taking place on the Ebonex® anode surface is O_2 evolution associated with adsorption of oxygen-containing species.

Figure 2 presents typical chronopotentiograms obtained at an Ebonex® plate anode in 0.1 M $(HOOC)_2$ + 1 M H_2SO_4 solution at varying current densities. The potentials changed only slightly with current density, at low current densities, but increased significantly at the higher current density (40 mA cm^{-2}). This demonstrates that to achieve high current densities for O_2 evolution requires relatively high anode potentials.

3.2. *Effect of background electrolytes*

The choice of the background electrolyte had a significant effect on O_2 evolution at Ebonex® as can be seen in Figure 3. The anodic current densities, obtained by chronopotentiometry, at an Ebonex® plate anode in

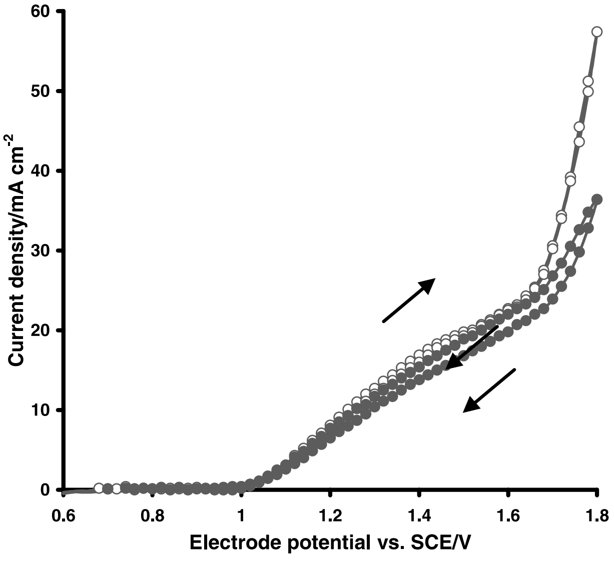

Fig. 1. Cyclic voltammograms obtained at an Ebonex® plate anode at a scan rate of 1 mV s^{-1}. Conditions: H-type glass cell divided by a glass frit. Cathode: Pt foil (12 cm^{-2}). Anolyte: 'Blank' (○) 1 M H_2SO_4 solution; 'Oxalic acid' (●) 0.1 M $(HOOC)_2$ + 1 M H_2SO_4 solution. Catholyte: 1 M H_2SO_4 solution. Temperature: 23 ± 0.5 °C. The arrows in the Figure indicate the scan directions.

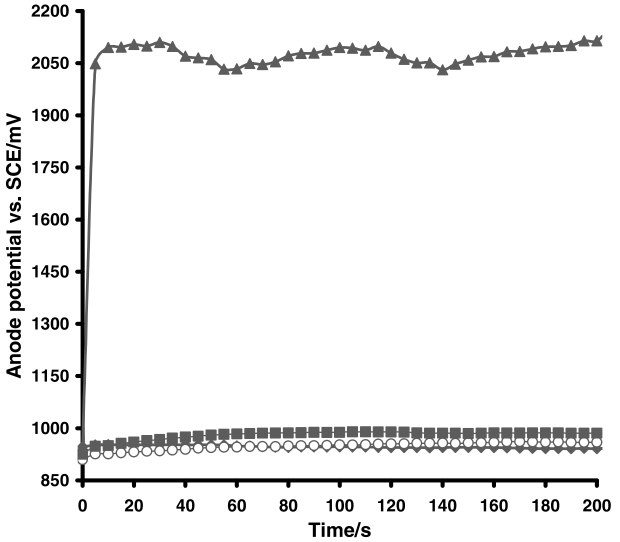

Fig. 2. Chronopotentiograms obtained at an Ebonex® plate anode at different current densities. Anolyte: 0.1 M $(HOOC)_2$ + 1 M H_2SO_4 solution. Other conditions as in Figure 1. Current density: (◆) 5, (○) 10, (■) 20 and (▲) 40 mA cm^{-2}.

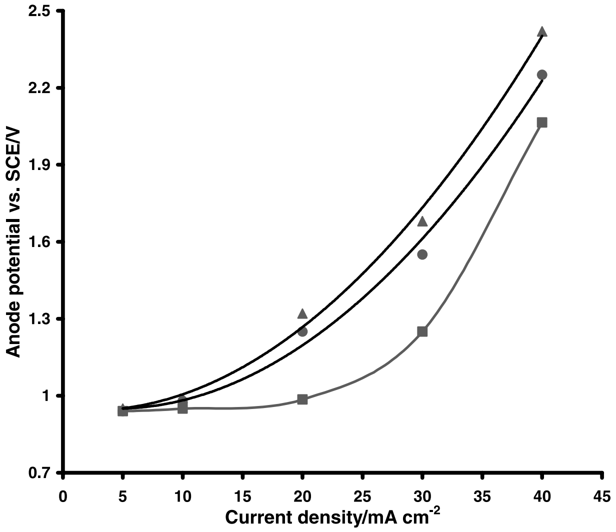

Fig. 3. Steady-state polarization curves obtained at an Ebonex® plate anode. Data collection by chronopotentiometry. Anolyte: 0.1 M $(HOOC)_2$ in 1 M H_2SO_4 solution ('Sulfuric acid', ■), 1 M Na_2SO_4 solution ('Sulfate', ●) or 1 M NaH_2PO_4 solution ('Phosphate', ▲). Catholyte: 1 M H_2SO_4 solution ('Sulfuric acid'), 1 M Na_2SO_4 solution ('Sulfate') or 1 M NaH_2PO_4 solution ('phosphate'). Other conditions as in Figure 1.

0.1 M $(HOOC)_2$ + 1 M H_2SO_4 solution were higher than those obtained with other supporting electrolytes. The pH of the solutions were as follows:

$$0.1\,\mathrm{M}\ (HOOC)_2 + 1\,\mathrm{M}\ H_2SO_4\ \text{solution}\ (\mathrm{pH} \ll 1)$$

$$< 0.1\,\mathrm{M}\ (HOOC)_2 + 1\,\mathrm{M}\ Na_2SO_4\ \text{solution}\ (\mathrm{pH} \sim 2)$$

$$< 0.1\,\mathrm{M}\ (HOOC)_2 + 1\,\mathrm{M}\ NaH_2PO_4\ \text{solution}\ (\mathrm{pH} \sim 3).$$

The results indicate that electrolyte pH had a very important effect on the O_2 evolution process which was particularly favourable in 0.1 M $(HOOC)_2$ + 1 M H_2SO_4 solution at sufficiently high potentials. Sulfuric acid appears a better electrolyte for O_2 evolution at an Ebonex® anode, in terms of supporting higher current densities.

3.3. *Effect of $(HCOO)_2$ concentration*

Figure 4 gives cyclic voltammograms obtained at Ebonex® in 0.1 M H_2SO_4 solution with different oxalic acid concentrations. The cyclic voltammogram obtained in 0.02 M $(HOOC)_2$ + 1 M H_2SO_4 solution showed a similar behaviour as that obtained in 0.1 M H_2SO_4 solution. The increase of oxalic acid concentration generally promotes more O_2 evolution, as shown in Figure 4 and was probably associated with the decrease in pH. This behaviour is further seen in Figure 5 where the effect of H_2SO_4 concentration, at same concentration of oxalic acid, is shown. It can be seen that current densities, at a given potential, were higher for electrolytes with a lower pH (i.e., increase in sulfuric acid concentration). To reach the same current density, much higher potentials were needed in the case of 0.1 M $(HOOC)_2$ solution, compared with those in the other two electrolytes. Thus lower pH appears to be more favourable for oxygen evolution at Ebonex®.

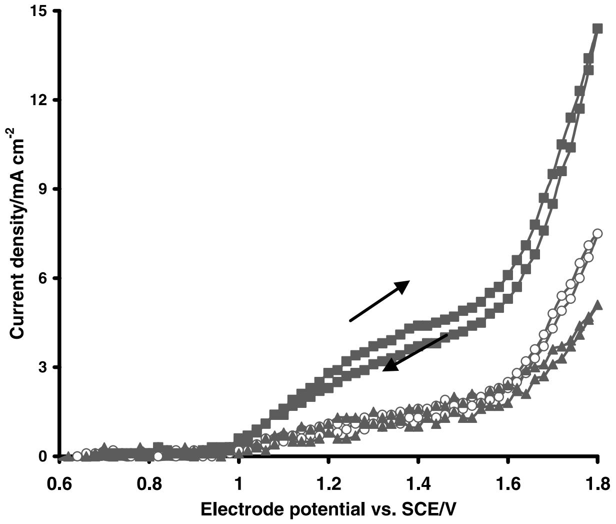

Fig. 4. Cyclic voltammograms obtained at an Ebonex® plate anode at a scan rate of 1 mV s^{-1}. Effect of oxalic acid concentration. Anolyte solvent and catholyte: 0.1 M H_2SO_4 solution. Other conditions as in Figure 1. Oxalic acid concentration: (○) blank, (▲) 0.02 and (■) 0.10 M.

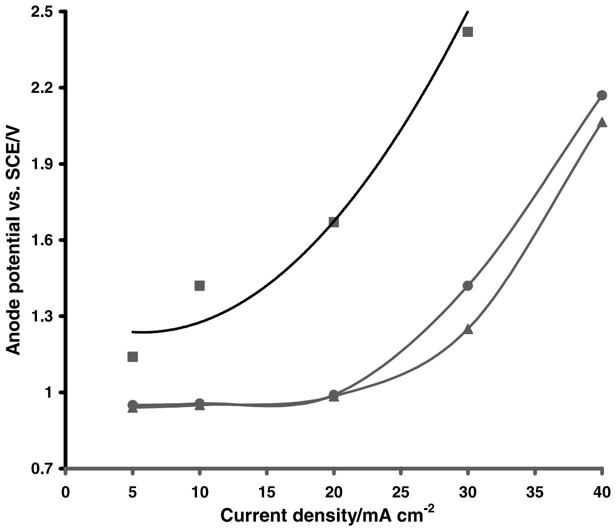

Fig. 5. Steady-state polarization curves obtained at an Ebonex® plate anode in 0.1 M oxalic acid solutions. Effect of sulfuric acid concentration. Data collection by chronopotentiometry. Other conditions as in Figure 1. Sulfuric acid concentration: (■) no acid, (●) 0.1 and (▲) 1 M.

3.4. *Gas chromatographic analysis of the electrolytic products*

An ideal anode for the electrosynthesis glyoxylic acid from oxalic acid should provide high anodic current and not oxidise oxalic acid at an appreciable level. Therefore, to determine the extent, if any, of oxalic acid oxidation at Ebonex®, gas chromatographic analysis of the anode gas product was undertaken. The electrolysis of oxalic acid was carried out using a controlled potential coulometric technique with H_2SO_4, Na_2SO_4 or NaH_2PO_4 solutions. During the electrolysis, the charge passed for each run was recorded and the gas products were collected for gas chromatographic analysis. In the gas chromatographic analysis, 1 ml samples were injected and, to ensure reproducibility, at least two runs for each condition were performed and blank solution electrolysis was also carried out as a reference.

The typical chromatographic analyses are presented in Table 1. The main product during the oxidation of aqueous oxalic acid solutions at an Ebonex® anode was O_2. No significant peak was observed for CO_2 in chromatograms for all samples, including those obtained at very high anode potentials, which suggests that only trace CO_2 was presented in the samples. The amount (relative concentration) of CO_2 obtained from the electrolytic products and from air are at similar trace levels, that is, 0.1157% to 0.1409%, as shown in Table 1. Obviously, O_2 evolution efficiently suppressed the possible process of oxalic acid oxidation to CO_2.

3.5. *Comparison of Ebonex® and platinized titanium*

Linear sweep voltammograms for the oxidation of sulfuric acid solutions at Ebonex® and platinized titanium (Pt/Ti) are shown in Figure 6. In the lower potential region ($< \sim 1.45$ V vs SCE), no difference was observed for Ebonex® and Pt/Ti anodes in the same medium. However, in the higher potential region the Pt/Ti anode gave higher current densities, especially in 0.1 M H_2SO_4 solution. The difference between the two anodes is less pronounced in 1 M H_2SO_4 solution even at high potential. The Pt/Ti anode generally showed better characteristics for O_2 evolution than Ebonex®.

Table 1. Summary for chromatographic analysis of the anodic electrolysis products – CO_2

System	Concentration* /%	Quantity of CO_2	*E* /V†
Air	0.13	trace	–
0.1 M $(HOOC)_2$ + 1 M H_2SO_4 solution	0.14	trace	4
1 M H_2SO_4 solution	0.14	trace	4
0.1 M $(HOOC)_2$ + 1 M Na_2SO_4 solution	0.12	trace	5
1 M Na_2SO_4 solution	0.12	trace	5
0.1 M $(HOOC)_2$ + 1 M NaH_2PO_4 solution	0.13	trace	5
1 M NaH_2PO_4 solution	0.13	trace	5
0.1 M $(HOOC)_2$ + 1 M H_2SO_4 solution	0.12	trace	3
0.1 M $(HOOC)_2$ + 1 M H_2SO_4 solution‡	0.81	small amount	3

* Relative concentration from chromatographic charts.

† Controlled potential for electrolysis referred to SCE.

‡ Platinized titanium anode. All other data obtained at Ebonex® anode.

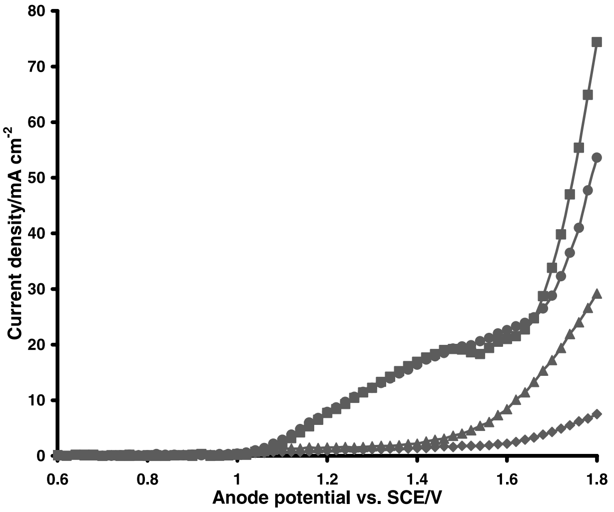

Fig. 6. Linear sweep voltammograms obtained at an Ebonex® plate anode or platinized titanium anode at a scan rate of 1 mV s^{-1}. Other conditions as in Figure 1. Anode and electrolyte: (■) Pt/Ti, 1 M sulfuric acid; (●) Ebonex®, 1 M sulfuric acid; (▲) Pt/Ti, 0.1 M sulfuric acid; (◆) Ebonex®, 0.1 M sulfuric acid.

Linear sweep voltammograms for oxidation of oxalic acid solution alone at Ebonex® and Pt/Ti anodes are shown in Figure 7. In 0.1 M oxalic acid solution Pt/Ti produced higher anodic current densities than Ebonex®, at similar potentials, which was related to the different surface states of the anodes in this medium. Noticeably the current densities achieved, at the same values of potential, below 1.6 V, in 0.1 M oxalic acid at Pt/Ti were greater than those achieved in 0.1 M H_2SO_4 solution. This might indicate that oxidation of oxalic acid takes place, as observed on Pt electrodes [6]. In this latter work the current density for oxalic acid oxidation

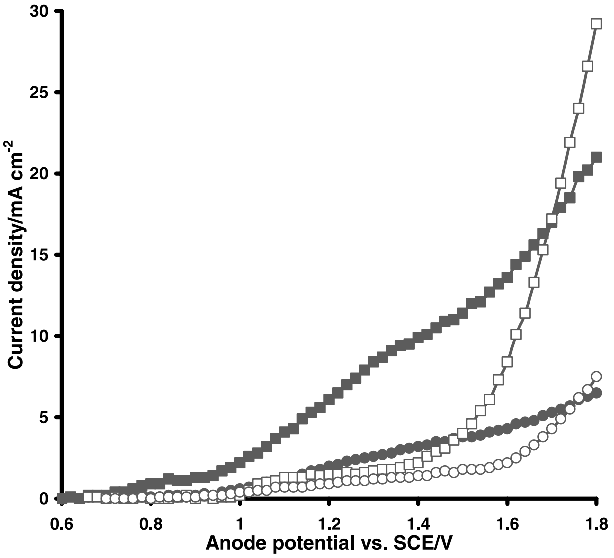

Fig. 7. Linear sweep voltammograms obtained at different electrodes and a scan rate of 1 mV s^{-1}. Catholyte: 0.1 M H_2SO_4 solution. Other conditions as in Figure 1. Anolyte and electrode: (■) 0.1 M oxalic acid, Pt/Ti; (●) 0.1 M oxalic acid, Ebonex®; (□) 0.1 M sulfuric acid, Pt/Ti; (○) 0.1 M sulfuric acid, Ebonex®.

reached a limiting value of approximately 10 mA cm^{-2}, with a 0.1 M oxalic acid solution, due to surface adsorption behaviour.

Figure 8 presents linear sweep voltammograms obtained at Pt/Ti or Ebonex® anode in 0.1 M $(HOOC)_2$ + 0.1 M or 1 M H_2SO_4 solutions. The voltammograms were similar to those obtained in 0.1 M or 1 M H_2SO_4 solutions. The Pt/Ti anode seems to be a superior anode than Ebonex® in that it supports higher current densities at the same potential. This was also supported by the steady-state polarization data, as shown in Figure 9.

The electrolysis of oxalic acid was carried out using a controlled potential coulometric technique with H_2SO_4 supporting electrolyte for Pt/Ti (Table 1). Under the conditions used, a small amount of CO_2, approximately 0.81% by volume, in the anode product gas was formed at the Pt/Ti anode.

3.6. *Stability of Ebonex®*

To assess the anodic stability of Ebonex® under conditions appropriate to the electrosynthesis of glyoxylic acid by the electroreduction of oxalic acid, extended electrolyses were undertaken. Figure 10 shows results obtained at a current density of 50 mA cm^{-2} with 0.5 M oxalic acid in sulfuric acid (1.0 M) over a period of 17 days. After four days the cell voltage rose from 2.0 V to about 5.5 V and remained almost constant for the remaining electrolysis period. After 16 days there was an indication of a further rise in potential, although at this point the electrolysis was terminated due to factors external to cell operation. Thus the data indicates that Ebonex® may have the required stability when used as an anode for oxygen evolution from mixed oxalic and sulfuric acid solutions.

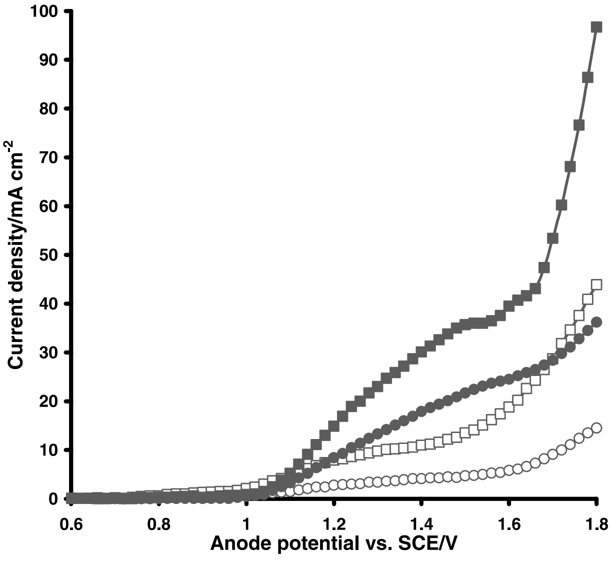

Fig. 8. Linear sweep voltammograms obtained in 0.1 M oxalic acid solution at different electrodes and a scan rate of 1 mV s^{-1}. Catholyte: 0.1 M or 1 M H_2SO_4 solution. Other conditions as in Figure 1. Anolyte solvent and electrode: (□) 0.1 M sulfuric acid, Pt/Ti; (○) 0.1 M sulfuric acid, Ebonex®; (■) 1 M sulfuric acid, Pt/Ti; (●) 1 M sulfuric acid, Ebonex®.

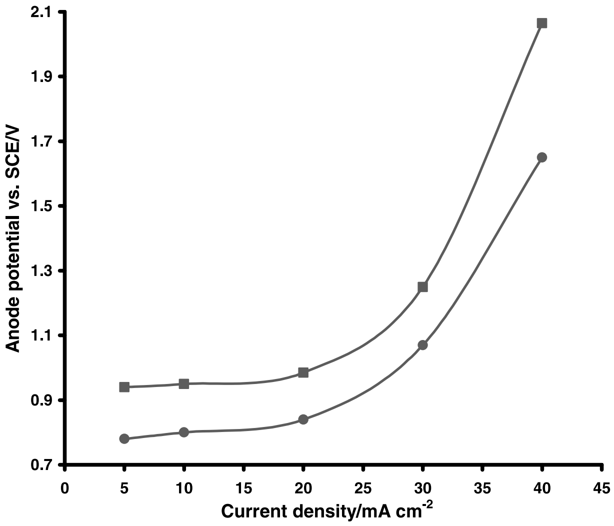

Fig. 9. Steady-state polarization curves obtained in 0.1 M oxalic acid plus 1 M H_2SO_4 solution at different electrodes. Data collection by chronopotentiometry. Other conditions as in Figure 8. Electrode: (●) Pt/Ti; (■) Ebonex®.

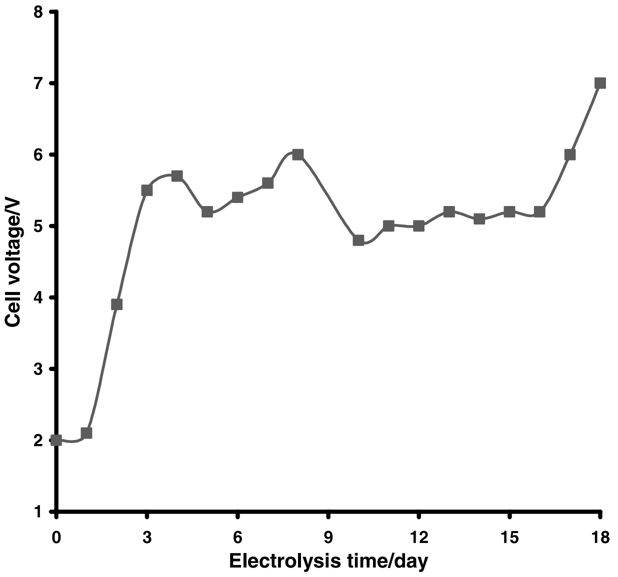

Fig. 10. Variation of cell voltage and electrolyte pH value with electrolysis time during oxalic acid oxidation at 50 mA cm^{-2}. Anode: Ebonex® (1 cm^2). Cathode: Pt foil (12 cm^2). Electrolyte: 0.5 M $(HOOC)_2$ in 1 M H_2SO_4 solution. Other conditions as in Figure 1.

4. Conclusions

Data obtained from cyclic voltammetric, linear sweep voltammetric, chronopotentiometric, controlled potential coulometric, and gas chromatographic measurements showed that oxalic acid oxidation at an Ebonex® anode, does not proceed, if at all, to any significant extent. The background electrolyte has a significant effect on O_2 evolution. H_2SO_4 produced much better characteristics, giving higher current densities at similar potentials, for O_2 evolution, than Na_2SO_4 and NaH_2PO_4. As an oxygen evolver, Ebonex® displayed the best performance in 0.1 M $(HOOC)_2$ + 0.1 M or 1 M H_2SO_4 solution. The results showed that Ebonex® had a anodic behaviour similar to that of Pt/Ti, especially in mixed $(HOOC)_2$–H_2SO_4 electrolytes. In accordance with these results, Ebonex® can be selected as an anode for synthesis of glyoxylic acid from oxalic acid in an undivided cell electrolysis.

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