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Study of mercury deposition from chloride solutions as test reaction for mass-transfer measurements

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This paper deals with a systematic study of mercury deposition from sodium chloride solutions to determine the feasibility of using this reaction for mass-transfer measurements. The study was performed with a glassy carbon rotating disc electrode and reproducible results were obtained only when the electrode was catodically pretreated. Polarization curves at different concentrations of sodium chloride and mercury (II) are reported. Hydrogen evolution and oxygen reduction has also been considered as side reactions and the conditions where their influence may be neglected are discussed. Experimental conditions where mercury deposition can be used as test reaction are given.

Keywords: *mass transfer*, *mercury deposition*, *sodium chloride*

List of symbols

C	total mercury concentration ($\mathrm{mol\,dm^{-3}}$)
D	diffusion coefficient ($\mathrm{m^2\,s^{-1}}$)
E	electrode potential (V)
i	current density ($\mathrm{A\,m^{-2}}$)
i_L	Limiting current density ($\mathrm{A\,m^{-2}}$)
T	temperature (K)

Greek characters

μ	viscosity ($\mathrm{kg\,m^{-1}\,s^{-1}}$)
ν	kinematic viscosity ($\mathrm{m^2\,s^{-1}}$)
ρ	density ($\mathrm{kg\,m^{-3}}$)
ω	angular velocity ($\mathrm{rad\,s^{-1}}$)

1. Introduction

In mass transfer studies it is necessary to adopt a test reaction. Several reactions have been proposed [1–4]. Some systems have found general acceptance: copper deposition from acidified copper sulphate solutions and reduction of ferricyanide from alkaline solutions. Copper deposition is preferred for studies of free-convection mass-transfer or combinations of forced and free convection due to its high densification coefficient. This shows high current densities because of the high solubility of copper salts in water although precautions are necessary to prevent an excessive surface roughening. Ibl and Schadegg [5] have shown that the onset of roughness occurs only after some time, which is higher than the time necessary to achieve zero concentration at the cathode surface. Thus, measurements can be performed, but in a restricted time range. The reduction of ferricyanide is adopted for forced convection studies using nickel or platinum electrodes. This reaction does not change the electrode surface area and the concentration solution remains constant when the reactor is used without a separator between the electrodes. However, the ferricyanide solutions present some disadvantages such as decomposition by light, electrode poisoning by cyanide and possible reaction of ferricyanide with the polymeric materials of the equipment components. Thus, the use of ferricyanide reduction requires control of concentration, protection of the electrolyte against light and frequent replacement of the solution.

Likewise, mercury deposition has been pointed out as a promising reaction for mass transfer studies [6]. Furthermore, mercury deposition from chloride solutions has industrial importance related to the removal of mercury from contaminated brine streams in chlor–alkali electrolysis plants. Van der Heiden et al. [7] reported the reduction of mercury concentration from 5 ppm to 0.05 ppm. by means of a cylindrical cell with a fluidized bed electrode of copper particles. The use of a graphite felt as an efficient porous electrode for the removal of traces of mercuric ions was investigated by Oren and Sofer [8]. Matlosz and Newman [6] analysed mercury deposition both theoretically and experimentally using a flow through porous carbon electrode.

Additional to the above work carbon is recognized as an appropriate material for electrochemical ap-

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plications. Electrodes based on various forms of carbon have received growing attention, particularly for electroanalytical purposes [9], electrorganic synthesis [10], removal and/or recovery of metal contaminants [11] and for various anodic processes [12]. For many of these important applications the use of metallic electrodes is complicated by the adsorption of organic materials by the formation of various oxides, or amalgamation in the particular case of mercury deposition, and by the limited anodic potential range. Glassy carbon is an attractive electrode material because of its greater chemical inertness, compared with other types of carbon, and its very small pore size [13].

As a convective electrode system the rotating disc electrode was chosen, for which the hydrodynamic equations and the convective diffusion equation have been solved rigorously for the steady state. Therefore, the experimental results are obtained in a system with well-defined hydrodynamics, and the kinetic parameters of the test reaction can be accurately determined.

The aim of this work was to study mercury deposition from chloride solutions as a test reaction for mass transfer measurements. Glassy carbon was chosen as electrode material. Another objective was to perform fundamental kinetic studies for electrochemical removal of mercury from wastewater.

2. Experimental details

The mass-transfer measurements were performed with a rotating disc electrode. A glassy carbon disk (Electrosynthesis Company Inc., Lancaster, NY), 3×10^{-3} m diameter, polished with emery paper and embedded in a Teflon tube 1.6×10^{-2} m base diameter, was used as working electrode. The counter electrode was a Pt foil with 2×10^{-4} m^2 geometric area. As reference a saturated calomel electrode was used and all potentials are referred to this electrode.

The counter and reference electrodes were placed in separate compartments, out of the cell, to avoid interference between the anodic reaction products and the cathodic reaction. The supporting electrolyte was a sodium chloride (p.a.) solution of varying concentration, from 1 to 4 M, with the addition of mercuric chloride (p.a.) to achieve a mercury concentration between 50 and 500 ppm, approximately. The accurate mercury concentration was determined by inductively coupled argon plasma emission spectroscopy (ICP) [14]. Each experiment was carried out with a fresh solution and in order to minimize the variation of mercury concentration during the experiment the cell volume was 800 cm^3. The solution was slightly acid, pH 3.9 and was prepared with tri distilled water.

The main electrochemical reaction studied at the working electrode was mercury deposition and the side reactions were oxygen reduction and hydrogen evolution. In the experiments with mercury deposition as sole cathodic reaction, prior to the measurements, dissolved oxygen was removed by bubbling purified nitrogen.

The experiments were performed at two temperatures, 303 K and 318 K, under a slow potentiodynamic sweep of 1×10^{-3} V s^{-1}. A PAR model 173 potentiostat/galvanostat and a PAR model 175 linear voltage sweep generator was used. The experimental results were recorded using an X-Y-t BAA recorder.

3. Results and discussion

3.1. *Reproducibility conditions*

Figure 1 shows the results of preliminary experiments, where the electrode voltage was cycled between 0 and −1.2 V in a 100 ppm Hg(II) + 3 M NaCl solution. The forward cycle (from 0 to −1.2 V) is different in comparison with the reverse cycle (from −1.2 to 0 V) showing significant hysteresis. These differences are due to the different initial state of the electrode surface before the start of each cycle. In the first case, the mercury deposition takes place on a polished glassy carbon, while in the second case, the electrode is covered with mercury. It can be observed that the mercury deposition on mercury is more reversible than the same process on glassy carbon. The rest potential at the end of the experiment was about 4×10^{-3} V, which is close to the equilibrium potential predicted by the Nernst equation, neglecting the activity coefficients.

It is important to note that when the number of cycles increases, the hysteresis disappears. Because when the second cycle starts the electrode surface is covered with mercury droplets. The same behaviour occurs for the following cycles.

Figure 2 shows the transient behaviour of mercury deposition from a 100 ppm Hg(II) in 3 M NaCl solution at an angular velocity of 1000 rpm. Independently of the applied electrode potential, the current reaches a steady state value equal to the limiting current density obtained from Fig. 1. The limiting current is $(3.5 \pm 0.1) \times 10^{-5}$ A. The same reproducibility is achieved in the all experiments. The time

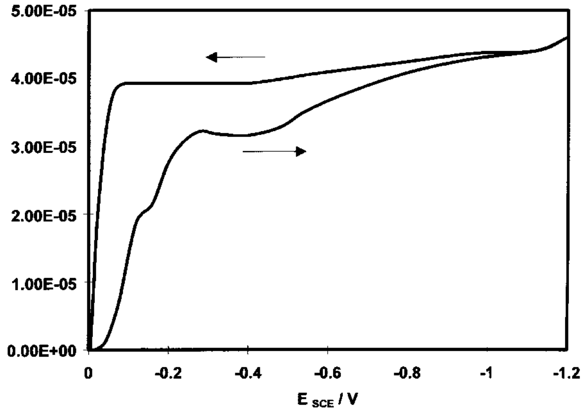

Fig. 1. Cyclic voltammogram for 3 M NaCl + 100 ppm Hg(II) solution on a polished glassy carbon electrode. Angular velocity 1000 rpm $T = 30\,°\mathrm{C}$.

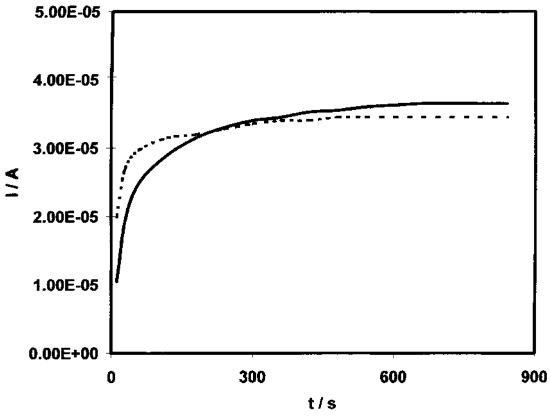

Fig. 2. Current against time curves for a 3 M NaCl + 100 ppm Hg(II) at different applied potentials. Full line: $E_{SCE} = -0.2$ V. Dashed line: $E_{SCE} = -0.6$ V. Angular velocity 1000 rpm $T = 30$ °C.

necessary to reach the limiting current is in direct relationship with the applied potential. When the applied potentials were −0.2 V and −0.6 V the times necessary to obtain limiting current were 11 min and 4 min, respectively. Likewise, the initial and final rest potential were −0.170 V and −0.004 V, respectively. These values are in agreement with the values of the rest potential observed in the experiment of Fig. 1.

Also, the limiting current value remains constant for a significant period indicating that the surface area of the electrode does not change. The surface state of the glassy carbon after the mercury deposition was examined by optical microscopy. Figure 3 shows the results. It can be observed that mercury deposits in the form of many of different sized mercury droplets. These results are in agreement with a previous study [15] of mercury deposition from noncomplexing media.

The above results were taken into account. Also, to obtain reproducible experimental data prior to the mass-transfer measurements, the electrode was potentiostated at −0.6 V at an angular velocity of 1000 rpm. The duration of the pretreatment was a function of mercury concentration. Thus, 10, 6 and 3 min were used for mercury concentration of 50, 100 and 500 ppm, respectively. This pretreatment is in accordance with common practice in ‘anodic striping voltametry’ [16].

3.2. *Mercury deposition from deaereated solutions*

Mercuric compounds are highly soluble in aqueous chloride solutions because of the complexation of mercuric ions. The following equilibrium reactions must be considered [17]:

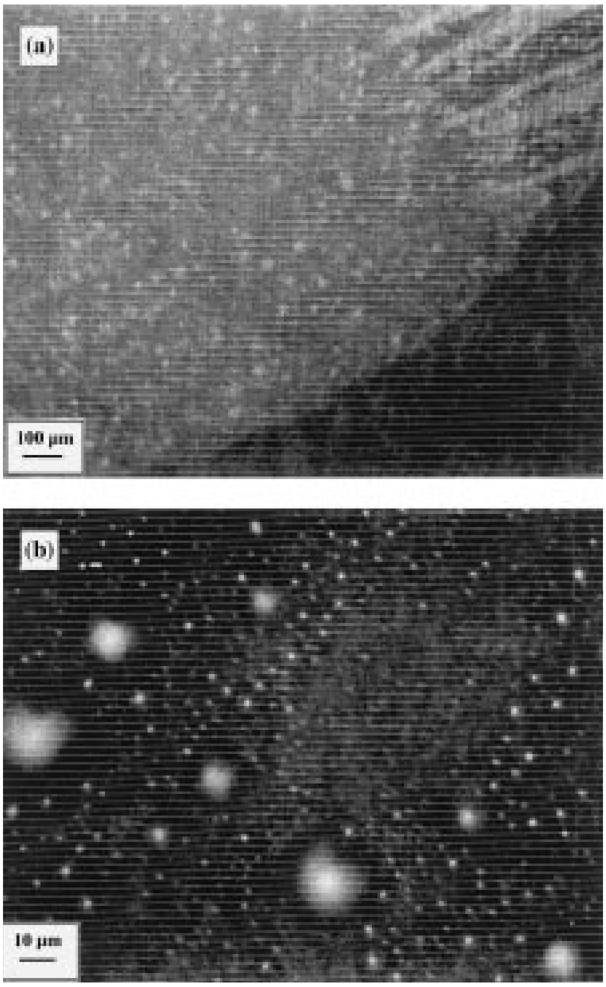

Fig. 3. Microscopic examination of the electrode surface after pretreatment at −0.6 V for 6 min (a) Magnification ×40; (b) magnification ×400.

$$\mathrm{HgCl^+ \leftrightarrow Hg^{2+} + Cl^-} \quad K_1 = 1.8 \times 10^{-7} \tag{1}$$

$$\mathrm{HgCl_2 \leftrightarrow Hg^{2+} + 2Cl^-} \quad K_2 = 6.0 \times 10^{-14} \tag{2}$$

$$\mathrm{HgCl_3^- \leftrightarrow Hg^{2+} + 3Cl^-} \quad K_3 = 8.3 \times 10^{-15} \tag{3}$$

$$\mathrm{HgCl_4^{2-} \leftrightarrow Hg^{2+} + 4Cl^-} \quad K_4 = 8.3 \times 10^{-16} \tag{4}$$

Table 1 shows the equilibrium concentrations of the mercuric complexes in terms of the chloride concentration for a total mercury (II) concentration of 5×10^{-4} M and neglecting the activity coefficients. According to these results, the $\mathrm{HgCl_4^{2-}}$ is the predominant species in the solution and its stability

Table 1. Mercuric complexes equilibrium concentrations as a function of chloride concentration

Total mercury concentration: 5×10^{-4} M

$[Cl^-]$ /M	$[HgCl_4^{2-}]$ /M	$[Hg^{2+}]$ /M	$[HgCl^+]$ /M	$[HgCl_2]$ /M	$[HgCl_3^-]$ /M
1	4.47×10^{-4}	3.71×10^{-19}	2.06×10^{-12}	6.18×10^{-6}	4.47×10^{-5}
2	4.73×10^{-4}	2.45×10^{-20}	2.72×10^{-13}	1.63×10^{-6}	2.36×10^{-5}
3	4.82×10^{-4}	4.94×10^{-21}	8.22×10^{-14}	7.41×10^{-7}	1.61×10^{-5}
4	4.86×10^{-4}	1.58×10^{-21}	3.50×10^{-14}	4.21×10^{-7}	1.21×10^{-5}

increases at higher chloride concentrations. Furthermore, the $HgCl_3^-$ concentration, which follows the $HgCl_4^{2-}$ concentration in order of stability, decreases when the chloride concentration increases. Therefore,

$$HgCl_4^{2-} + 2e^- \rightarrow Hg + 4Cl^- \qquad (5)$$

is the predominant reaction during the mercury deposition from a solution containing chloride ions in high concentration. Likewise, the $HgCl_4^{2-}$ concentration coincides practically with the total Hg (II) concentration when the chloride concentration is higher than 3 M.

Figures 4 and 5 show experimental polarisation curves for pretreated electrodes, in deaereated solutions as a function of the angular velocity at two sodium chloride concentrations. They show the typical behaviour corresponding to a mixed control process with a well-defined plateau.

For the case of 2 M sodium chloride solution, the system practically achieves limiting current conditions immediately following small polarization and the current plateau is sustained to −0.45 ~ −0.5 V. By contrast, the 1 M NaCl solution shows a small cathodic peak just following polarization. This peak may be attributed to the reduction of $HgCl_3^-$ species, whose concentration is significant when the sodium chloride concentration decreases. Due to the peak, the current density plateau is not well defined . Thus, solutions with 1 M sodium chloride as supporting electrolyte are not suitable for accurate mass-transfer measurements.

Typical polarization curves at different sodium chloride concentrations are shown in Fig. 6. For 1 M and 2 M the limiting current densities are the same. This behaviour is a consequence of two opposite effects as the chloride concentration increases. An increase in chloride concentration produces an increase in $HgCl_4^{2-}$ concentration and, consequently, in the limiting current density but the increase in chloride concentration also produces an increase in viscosity, which reduces the mass-transfer rate. The same argument explains the relative position of the polarization curves for the cases of 3 and 4 M sodium chloride. Therefore, for the concentrated solution the influence of the increase in $HgCl_4^{2-}$ concentration is higher than that of increase in viscosity on the limiting current density. Thus, the limiting current density for 4 M is higher than that for 3 M.

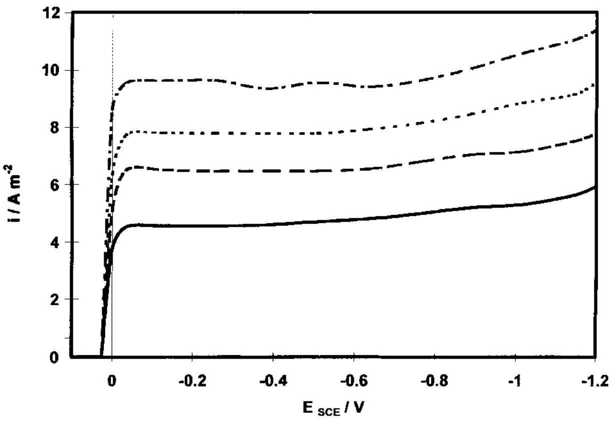

Fig. 5. Polarization curves as a function of the angular velocity. Solution: 2 M NaCl + 100 ppm Hg(II), deaerated. $T = 30$ °C. Key for ω: (——) 500, (– –) 1000, (- - - -) 1500 and (– · – ·) 2000 rpm.

Figures 4, 5 and 6 also show that the current density increases for potentials lower than −0.5 V, which indicates the onset of a side reaction such as the reduction of another mercuric (II) complex or hydrogen evolution. From the shape of the curves in Figs 4, 5 and 6 it is not possible to attribute the increasing current to the reduction of another mercuric (II) complex due to the fact that the current density increases in the same potential range in spite of the different chloride concentration. The only possibility is proton reduction. But, hydrogen evolution on mercury requires high overpotentials, approximately −0.8 V [18], to occur at an appreciable rate and taking into account that the hydrogen equilibrium potential under the present conditions is −0.471 V, proton reduction is expected at potentials lower than −1.3 V. To elucidate this behaviour additional experiments with a glassy carbon rotating disc were performed. In Fig. 7

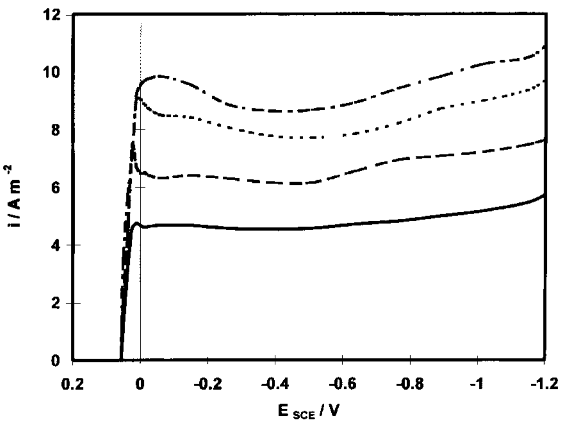

Fig. 4. Polarization curves as a function of the angular velocity. Solution: 1 M NaCl + 100 ppm Hg(II), deaereated. $T = 30$ °C. Key for ω: (——) 500, (– –) 1000, (- - - -) 1500 and (– · – ·) 2000 rpm.

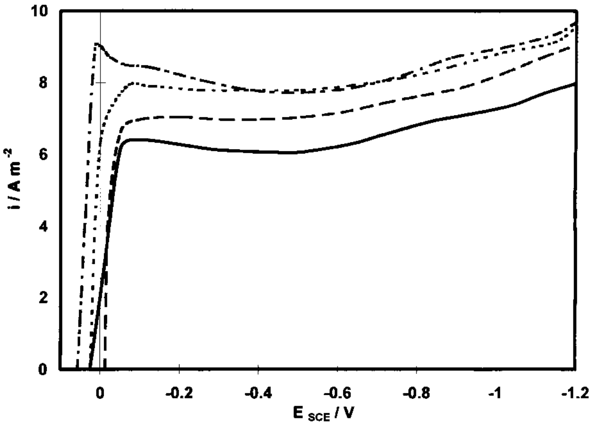

Fig. 6. Polarization curves at different sodium chloride concentrations. Total mercury (II) concentration: 100 ppm. Angular velocity 1000 rpm. $T = 30$ °C. [Cl$^-$]: (– · – ·) 1 M, (- - - -) 2 M, (– –) 4 M, (——) 3 M.

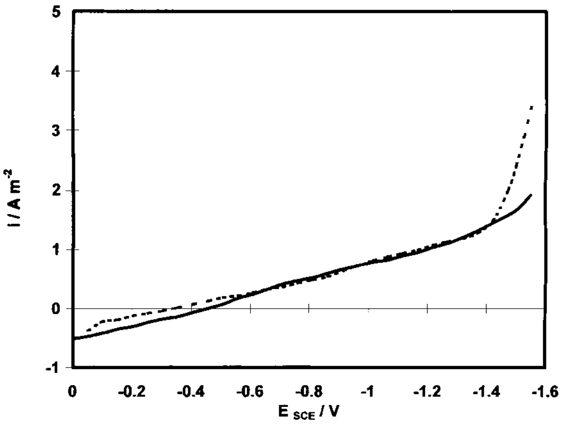

Fig. 7. Polarization curves as a function of the angular velocity. Solution: 3 M NaCl, without mercury, deaereated. $T = 30\,°C$. $\omega = 1000$ rpm (—); $\omega = 1000$ rpm (pretreated electrode) (- - - -).

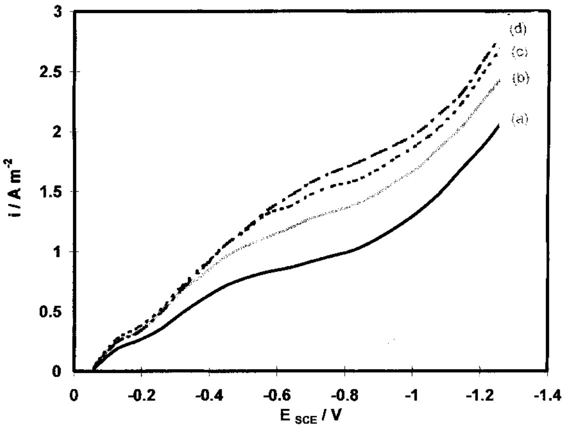

Fig. 9. Polarization curves for a 4 M NaCl aerated solution on a pretreated electrode at different angular velocities ω: (a) 500, (b) 1000, (c) 1500 and (d) 2000 rpm.

polarization curves of pretreated and nonpretreated electrodes in 3 M NaCl solution, without mercury (II) ions, are reported. Under these conditions the only cathodic reaction is hydrogen evolution and in both cases cathodic currents are not detected until a potential of approximately $-0.45 \sim -0.5$ V is achieved. Consequently, the increase in current density observed in Figs 4, 5 and 6 for electrode potentials lower than -0.5 V may be attributed to hydrogen evolution, which takes place at patches of the glassy carbon surface not in contact with mercury droplets (Fig. 3).

3.3. *Oxygen reduction as side reaction in mercury deposition*

Figure 8 shows polarization curves for pretreated electrodes from 2 M and 4 M sodium chloride solutions with 100 ppm Hg (II), with and without oxygen at an angular velocity of 1000 rpm.

Figure 9 shows polarization curves in 4 M NaCl solution, saturated with air, and without mercury (ii) ions for pretreated glassy carbon, at different angular velocities. Under these experimental conditions the possible cathodic reactions are oxygen reduction and hydrogen evolution. Taking into account Fig. 7, the increase of current density in Fig. 9 for potentials lower than -0.5 V can be attributed to hydrogen evolution. Thus, the current densities observed at potentials higher than -0.5 V correspond to oxygen reduction. Thus, oxygen reduction explains the higher currents observed in Fig. 8 for aerated solutions. It is also noted that oxygen reduction takes place at limiting current density within the same potential range as mercury deposition. However, the partial contribution of oxygen reduction to the total current decreases with increases in chloride concentration. This is mainly due to the low oxygen solubility in concentrated solutions.

In mass transfer studies it is necessary to avoid side reactions. The present results suggest that in dilute mercury (II) solutions and at chloride concentrations lower than 2 M, it is convenient to work in deaereated solutions. At higher chloride concentrations the partial current of the side reaction is negligible, as seen in Fig. 8.

3.4. *Stability of the solutions*

To determine the stability of the solutions, experiments with aged solutions were performed. Figure 10

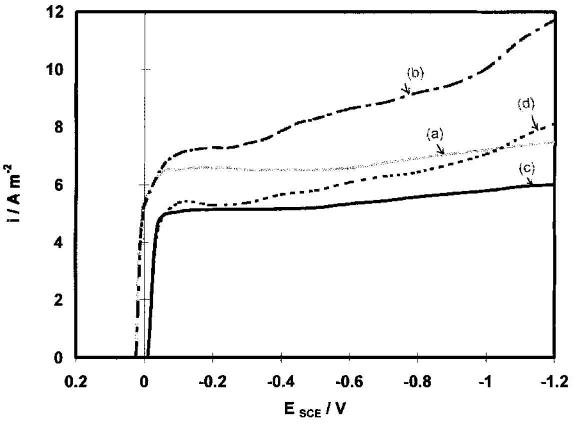

Fig. 8. Polarization curves for 2 M and 4 M NaCl + 100 ppm Hg(II) solution with and without dissolved oxygen. Angular velocity: Key: (a) 2 M NaCl; (b) 2 M NaCl (+O$_2$); (c) 4 M NaCl; (d) 4 M NaCl (+O$_2$).

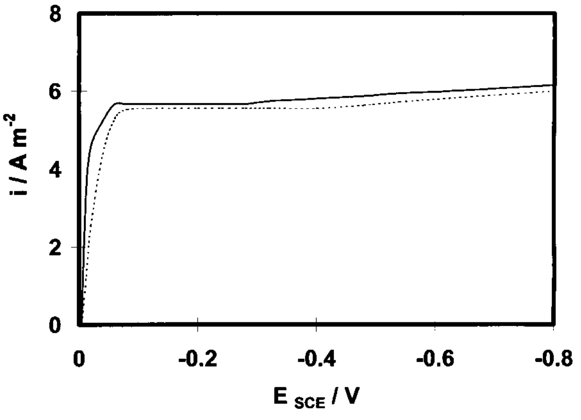

Fig. 10. Polarization curves for a deaereated fresh and aged 3 M NaCl + 100 ppm Hg(II) solutions. Angular velocity 1000 rpm. $T = 30\,°C$. Key: (- - - -) *i* (aged); (——) *i* (fresh).

shows polarization curves obtained with a fresh solution (full line) and with the same solution after approximately 300 days from the first measurement (dashed line). During this time the solution was stored in a typical glass reservoir. It can be concluded that the concentration and physicochemical properties are not changed during the storage time

3.5. *Physicochemical properties of the solutions*

Figure 11 shows the limiting current densities as a function of the square root of angular velocity for different mercury (II) and sodium chloride concentrations at two temperatures. From the slope of the lines in Fig. 10, and using the Levich equation, the diffusion coefficients of $HgCl_4^{2-}$ were determined. Table 2 summarizes the composition and physicochemical properties of the solutions.

Column 9 in Table 2 shows the Stokes–Einstein parameter. The mean value of the ratio $D\mu/T$ is $5.98 \times 10^{-15}\,\mathrm{kg\,m\,s^{-2}\,K^{-1}}$ with a standard deviation of $0.59 \times 10^{-15}\,\mathrm{kg\,m\,s^{-2}\,K^{-1}}$. The Stokes–Einstein parameter is reasonably constant in the investigated range.

4. Conclusion

It is concluded that mercury deposition from chloride solutions is an interesting test reaction for mass-transfer studies owing to the following characteristics:

(i) It shows a wide range of potential, approximately 0.5 V, where the limiting current is sustained.

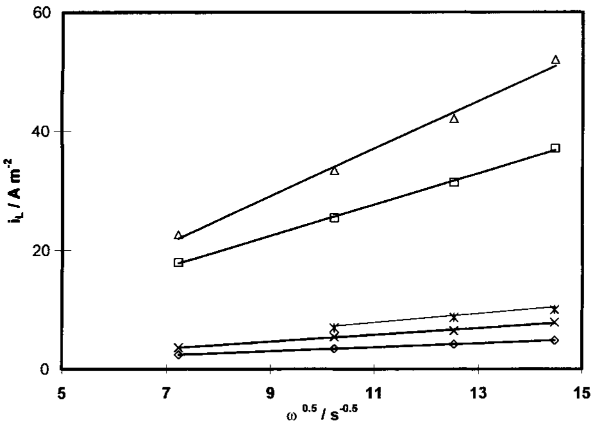

Fig. 11. Limiting current density against square root of angular velocity. (◇) 50 ppm Hg(II) + 4 M NaCl, $T = 318$ K; (□) 500 ppm Hg(II) + 4 M NaCl, $T = 303$ K; (△) 500 ppm Hg(II) + 4 M NaCl, $T = 318$ K; (×) 100 ppm Hg(II) + 3 M NaCl, $T = 303$ K; (*) 100 ppm. Hg(II) + 4 M NaCl, $T = 318$ K.

(ii) Mercury deposition does not alter the surface area. Likewise, poisoning or fouling of the substrate is not observed.

(iii) The mercury concentration can easily be determined by ICP (this work), atomic absorption spectroscopy, gold film mercury analysis [8], atomic absorption spectroscopy with cold vapour technique and complexometric analysis.

(iv) The solution does not decompose nor do properties vary with time.

(v) The chemicals used are inexpensive.

(vi) Mercuric compounds are highly soluble in aqueous chloride solutions due to of complexation so that higher limiting current densities can be reached. It is convenient to work with a

Table 2. Physicochemical properties of electrolytes

$[Cl^-]$ /M	10^4C /M	$10^4[HgCl_4^{2-}]$ /M	T /K	ρ /kg m^{-3}	$10^3\mu$ /kg m^{-1} s^{-1}	$10^6\nu$ /m^2 s^{-1}	$10^{10}D$ /m^2 s^{-1}	$10^{15}D\mu$ /kg m s^{-2} K^{-1}
1	5	4.477	303	1040	1.19	1.14	13.83	5.43
2	5	4.734	303	1080	1.46	1.35	14.37	6.92
3	5	4.819	303	1120	1.64	1.46	9.50	5.14
4	5	4.861	303	1160	1.83	1.58	10.59	6.40
4	2.5	2.431	303	1160	1.82	1.57	9.86	5.92
4	2.5	2.431	318	1160	1.45	1.25	12.91	5.89
4	25	24	303	1160	1.85	1.59	9.25	5.65
4	25	24	318	1160	1.49	1.28	13.80	6.47

Table 3. Comparison of alternative test reactions for mass transfer measurements

Property	*Cu deposition*	*Ferricyanide reduction*	*Hg deposition from chloride solutions*
Increasing of roughness	Important after an induction time	None	None
Poisoning of the electrode	None	Important	None
Stability of the solutions	High	Poor	High
Main side reaction	O_2 reduction	O_2 reduction	O_2 reduction but the effect is negligible at high chloride concentration
Definition of limiting current plateau	Good	Very good	Very good when the chloride concentration is higher than 2 M

concentrated sodium chloride solution, that is, 3 M or higher, for which the $HgCl_4^{2-}$ complex is practically the sole species, the limiting current density is higher and the influence of oxygen reduction as side reaction can be neglected.

(vii) The test solution and the electrode can be easily restored to their initial states. The deposited mercury can be chemically dissolved by sodium hypochlorite.

(viii) For the interpretation of limiting currents, physical properties of the electrolyte must be known accurately over large ranges of composition. In the present case the electrolyte is mainly brine, whose properties are widely known.

(ix) Electrode pretreatment is necessary to obtain reproducibility in the experimental measurements. Thus, prior to the experiment the electrode must be cathodically polarised for a short time, depending on the mercury concentration.

Table 3 summarizes the main characteristics of mercury deposition from chloride solutions as an alternative test reaction to two other accepted systems.

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