



THE ELECTROCHEMICAL DEGRADATION OF LINEARALKYLBENZENESULFONATE (LAS) ON PLATINUM ELECTRODE

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(First received January 1997; accepted in revised form August 1997)

Abstract—The electrochemical behaviour of linearalkylbenzenesulfonate (LAS) on Pt electrode has been investigated in 0.05 M Na₂SO₄ and in 0.1 M NaCl at pH = 8 by potentiokinetic method and by electrolysis. The anodic and cathodic semilogarithmic current-potential curves were obtained between -1.6 V – + 1.6 V. The experimental discharge potentials (Eexp.) have been determined by means of current-potential curves obtained by electrolysis method between 0–3 V. The percentages of surface active material remaining in solution after specified time periods (0–6 h) were determined for different initial concentrations of LAS (30, 150, 300 ppm) by using the potentiostat (+1.6 V) and by applying constant potentials of 20 V via a direct current source. The concentrations of LAS in solution was determined by UV spectrophotometer. Results indicate that when a potential of 20 V is applied on the electrodes the concentrations of surface active materials (LAS) decreases by 40–50% at the end of 6h in 0.05 M Na₂SO₄ solution. © 1998 Elsevier Science Ltd. All rights reserved

Key words—electrochemical degradation, linearalkylbenzenesulfonate (LAS), platinum, electrode

INTRODUCTION

Synthetic detergents are a mixture of linearalkylbenzenesulfonate (LAS) and its isomers together with other additives. LAS is a surface active material and is found in relatively high amounts in domestic and industrial wastewaters, discharged mainly from the textile, tanning and cosmetic industries. Today the detergent wastes constitute a major component of organic pollutants that are carried by various means into lakes, rivers and seas and cause a great environmental problem. In this connection, the wastewater has to be treated before discharging. Biological treatment is the most economic process and is usually applied for the treatment of organic pollutants. The situation is completely different when the wastewater contains toxic or/and refractory organic pollutants. In this case, another type of treatment must be used. Recently, the electrochemical method for the treatment of wastewater containing organic pollutants has attracted a great deal of attention (Comninellis, 1994; Lin and Peng, 1994; Brillas *et al.*, 1995). The electrochemical oxidation of all organic compounds is theoretically possible before oxygen evolution (due to H₂O discharge) but in practice, the oxidation reaction is very slow as a consequence of kinetic rather than

thermodynamic limitations. To increase the electrochemical rate of oxidation, electrocatalytic anodes have been proposed.

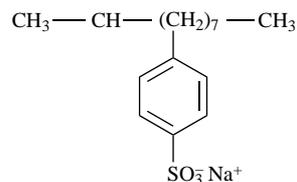
The most widely used electrode in studying electrochemical oxidation of such organic materials are Pt, Ti/IrO₂, Ti/SnO₂, PbO₂, Ti/SnO₂/PbO₂ (Breiter, 1963; Lamy, 1984; Santos and Giordano, 1984; Huser *et al.*, 1988; Kötzt *et al.*, 1991; Stucki *et al.*, 1991; Comninellis, 1994; Hwang and Lee, 1996; Olivi *et al.*, 1996). In such electrodes, the part which brings about the anodic oxidation is the oxidized surface of platinum. The kinetics of the adsorption of an organic substance on a surface is closely related to the chemical nature and properties of the surface on which it is adsorbed (Breiter, 1963).

In electrooxidation processes, the nature of the electrolyte is also an important factor as is the structure of the electrode. In chloride solutions, because the chloride ion is selectively adsorbed on platinum surfaces the organic molecules cannot be appreciably adsorbed (Snell and Keenan, 1981). On the other hand, in sulphate solutions, because the sulphate ions are only weakly adsorbed, the electrooxidation process takes place easily (Snell and Keenan, 1982).

The aim of this work is to investigate the electrochemical behaviour of LAS on a Pt electrode in solutions of 0.05 M Na₂SO₄ and 0.1 M NaCl containing varying amounts of LAS (0, 30, 150 and 300 ppm) at pH = 8.0 by potentiokinetic methods

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and by electrolysis and to examine the process of electrochemical degradation instead of a ready oxide layer such as SnO_2 during the Pt-oxide formation, i.e. the effect of the Pt-oxide formation stage on electrochemical degradation.



Scheme 1.

EXPERIMENTAL STUDIES

The electrolytes used were 0.05 M Na_2SO_4 and 0.1 M NaCl solutions containing 0, 30, 150 and 300 ppm LAS. The surface active material (LAS) used was 96.5% pure and had a molar mass of 320 g mol^{-1} as shown in Scheme 1.

The pH of the Na_2SO_4 solution was adjusted by additions of H_2SO_4 and NaOH, while that of NaCl solution was adjusted by HCl and NaOH.

The anodic and cathodic current-potential curves were obtained by using a potentiostat employing the 3-electrode technique. The working and counter electrodes were made from bright platinum sheets ($1 \times 1 \text{ cm}$) and the electrical conductivity was provided by copper-wire embedded glass. The potentials were measured against a saturated calomel electrode (SCE). All experiments were carried out at room temperature and under atmospheric pressure.

The electrolyte solution was stirred continuously by a magnetic stirrer at a constant rate. After the equilibrium potential was reached, the potential was varied at a rate of 6 mV/min starting from the cathodic region at -1.6 V , moving to 1.6 V in the anodic direction and then back to -1.6 V , thus generating hysteresis current-potential loops.

Current-potential values were obtained during electrolysis in a cell in which platinum was used both as anode and cathode (Pt-Pt). From the current-potential values obtained the experimental discharge potentials (E_{exp}) were determined.

Solutions of LAS were prepared at different concentrations and a standard curve was drawn by the UV spectrophotometer (222 nm). This standard curve was then used in determining the concentration (C) of LAS at the

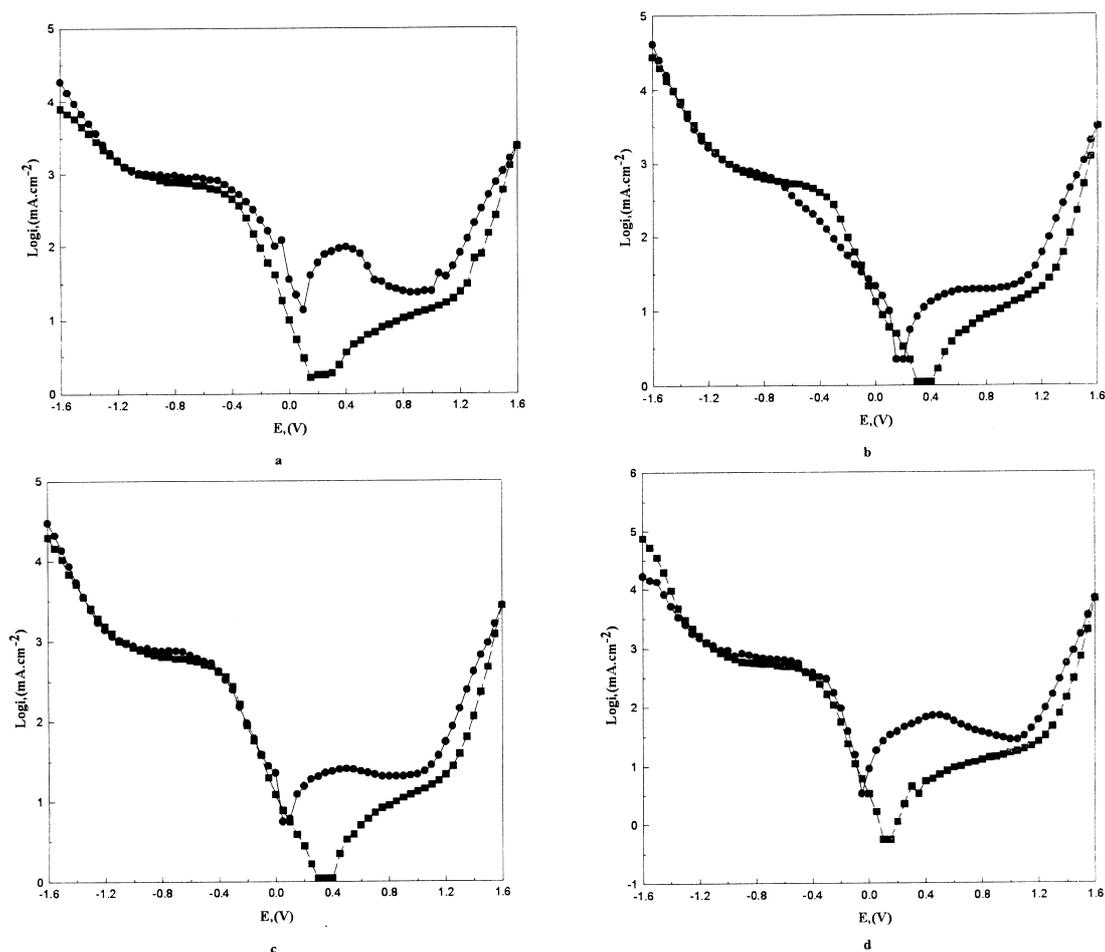


Fig. 1. The current density potential curves obtained in 0.05 M $\text{Na}_2\text{SO}_4 + x \text{ ppm LAS}$ solution at $\text{pH} = 8$ (a, $x = 0$; b, $x = 30$; c, $x = 150$; d, $x = 300$).

end of each hour for a period of 6 h in Na_2SO_4 and NaCl solutions for the following cases:

- plain solution with no metal electrode in it,
- by using the potentiostat in the solution and applying +1.6 V,
- by applying a voltage of 20 V between the electrodes from a direct current source.

Calling the initial concentrations of LAS in the electrolyte C_0 and the concentrations at the end of each period C , the percentage of surface active material concentration (PSM) was calculated from the equation: $\text{PSM} = (C/C_0) \times 100$.

RESULTS

The semilogarithmic current-potential curves obtained in 0.05 M Na_2SO_4 and 0.1 M NaCl sol-

utions at $\text{pH} = 8$ containing various concentrations of LAS are shown in Fig. 1 and 2, respectively. The concentrations of LAS in the solutions were 0, 30, 150 and 300 ppm in (a), (b), (c) and (d), respectively, in both figures. The equilibrium potentials of the Pt electrode measured against the SCE under the experimental working conditions are collected in Table 1.

In Na_2SO_4 solutions

As seen in Fig. 1, in 0.05 M Na_2SO_4 solutions containing various concentrations of LAS, while moving from the equilibrium potential toward negative potentials, the current-density increases linearly up to about -0.5 V. In all solutions the current-density between the potentials of ~ -0.5 V and ~ -1.1 V reaches a limiting value with a magnitude of $\sim 0.95 \text{ mAcm}^{-2}$. The linear increase in current-density starts again from ~ -1.1 V.

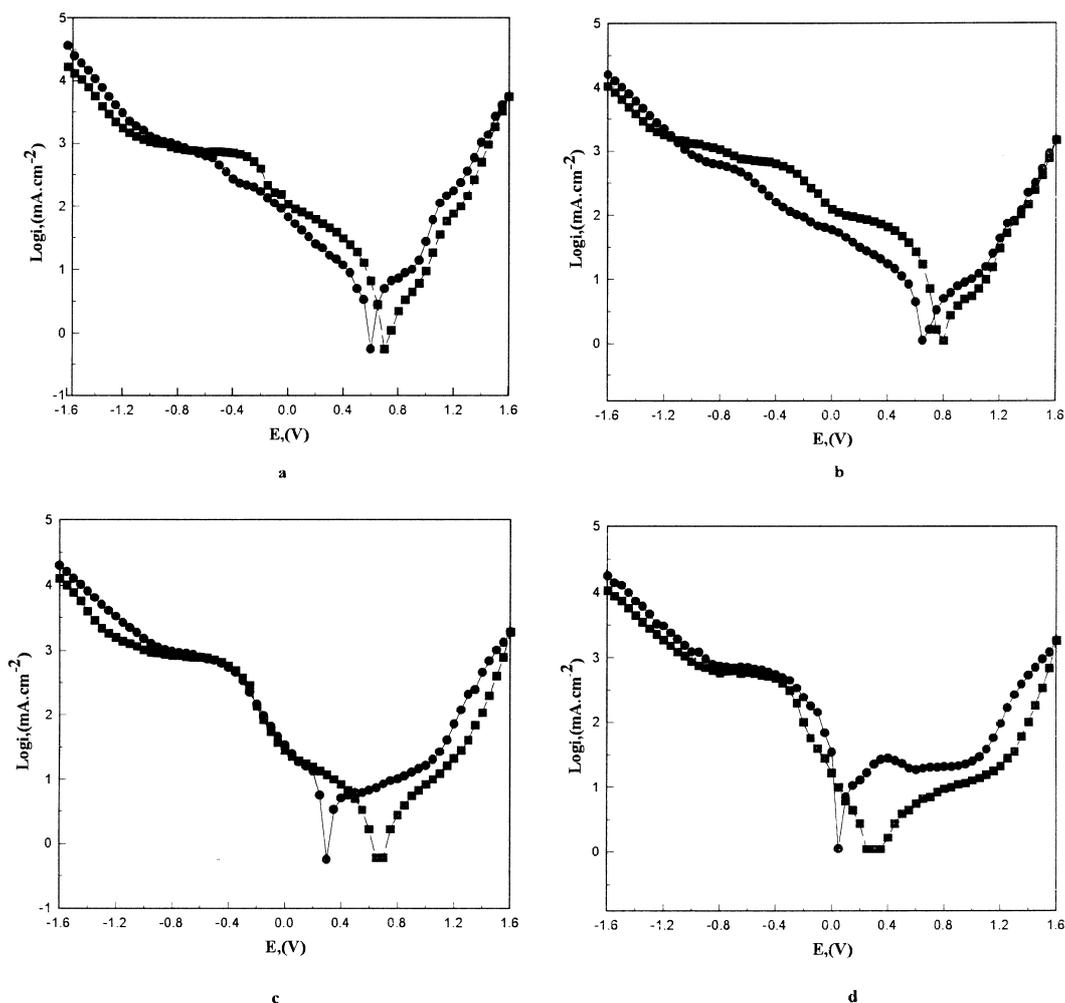


Fig. 2. The change of the surface active material concentrations (PSM) with time in 0.05 M Na_2SO_4 solution at $\text{pH} = 8$ containing (0, solution in a container with no metal in it; \blacksquare —, using the potentiostat at +1.6 V; \blacksquare , applying a voltage of 20 V from a direct current source). a, 30 ppm LAS; b, 150 ppm LAS; c, 300 ppm LAS.

Table 1. Equilibrium potentials (E_{eq}) of Pt in aqueous Na_2SO_4 and NaCl solutions containing various concentrations of LAS

LAS concentration (ppm)	E_{eq} (V) 0.05 M Na_2SO_4	E_{eq} (V) 0.1 M NaCl
0	0.100	0.600
30	0.100	0.600
150	0.050	0.300
300	-0.050	0.050

Figure 1(a) shows the anodic polarization of the Pt electrode in 0.05 M Na_2SO_4 solution containing no LAS. From the equilibrium potentials onward, as the rotatid is moved towards more positive values where polarization takes place the current-density increases until a potential of 0.45 V and current peak is formed at this potential. The current-density decreases between 0.45 V to 0.7 V, reaches a constant value of about 0.015 mA cm^{-2} in the potential range 0.7 V to 1.0 V and then starts increasing linearly again after 1.0 V [Fig. 1(a)].

Figure 1(b–d) show the current-density vs potential plots for solutions containing 30, 150 and 300 ppm LAS, respectively. These curves show that, as the concentration of LAS increases, the equilibrium potential shifts to more active (negative) values. At positive potentials, where anodic polarization takes place, in 30 and 150 ppm LAS solutions, the current-density increases from the equilibrium potential up to 0.35 V; remains around 0.013 mA cm^{-2} in the potential region between 0.35 V–1.1 V and from 1.1 V it starts increasing again linearly [Fig. 1(b,c)]. In the solution with 300 ppm LAS, in the anodic region the current-density increases starting with the equilibrium potential up to 0.45 V; it decreases between 0.45 V–1.1 V and as from 1.1 V starts increasing linearly [Fig. 1(d)].

In all test media, when the potential is reversed back from +1.6 V, the current-densities decrease in the anodic regions (\square), while the forward and reverse curves are almost the same in cathodic range (Fig. 1).

In NaCl solutions

In 0.1 M NaCl solutions containing various concentrations of LAS (0, 30, 150, 300 ppm), as one moves from the equilibrium potential towards more negative potentials, the current-density increases linearly in all solutions up to about -0.6 V (Fig. 2). In all solutions, in the potential range $\sim -0.6 \text{ V}$ to $\sim -0.95 \text{ V}$ the current-density reaches a limiting value of about 0.95 mA cm^{-2} and after about -0.95 V it starts increasing linearly again.

Figure 2(a) shows the anodic polarization of Pt in 0.1 M NaCl solution without any LAS. In the anodic region, as from the equilibrium potentials, the current-density increases almost linearly until +1.6 V.

The current-density vs potential curves in solutions containing 30, 150 and 300 ppm LAS are shown in Fig. 2(b–d), respectively. As the concen-

tration of LAS increases, the equilibrium potentials become shifted toward more active potentials. At positive potentials where anodic polarization takes place in the solution containing 30 ppm LAS, the current-density increases up to a potential of +1.6 V as in the NaCl solution without any LAS [Fig. 2(b)]. In a solution with 150 ppm LAS, the current-density increases in the anodic region until 0.45 V, remains constant at about 0.012 mA cm^{-2} in the region between 0.45 V and 1.0 V, and then increases linearly as from 1.0 V [Fig. 2(c)]. Finally, in 300 ppm LAS solution, the current-density increases until 0.45 V at which a current peak arises and decreases until 0.6 V [Fig. 2(d)]. In the potential range 0.6 V to 1.0 V the current-density reaches a value of 0.013 mA cm^{-2} and then increases linearly from 1.0 V to +1.6 V [Fig. 2(d)].

In all solutions, while the current-density decreases in the anodic region from +1.6 V when the system is reversed, the change in the cathodic region is almost the same in the forward direction. However, the equilibrium potentials of the system in the forward direction, shift to more positive values in the reverse directions for all solutions [Figs 1 and 2 marked (\square)].

Discharge potentials

Current-potential curves were drawn from data obtained using Pt for both the anode and cathode in 0.05 M Na_2SO_4 and 0.1 M NaCl solutions at pH = 8 containing 30, 150 and 300 ppm LAS. By making use of these current-potential curves, the experimental discharge potentials were determined. In 0.05 M Na_2SO_4 solution containing no LAS the discharge potential was determined as 2.2 V while in solutions containing 30, 150 and 300 ppm LAS this potential was formed to be 1.4 V and 2.4 V.

In chloride solutions two different discharge potentials were determined. In the 0.1 M NaCl solution containing no LAS, the discharge potentials were 1.4 V and 2.4 V. In solutions containing various concentrations of LAS, on the other hand, these two values have been predicted as 1.2 V and 2.2 V, respectively.

Change in surface active material

In Fig. 3 and 4 the changes in the surface active material concentration (PSM) with time are given for 0.05 M Na_2SO_4 and 0.1 M NaCl solutions, respectively. Electrodes from a direct current source (\square) was applied to the solution in the container with no metal in it, using the potentiostat at anodic +1.6 V (SCE) (Δ), and a voltage of 20 V. Solutions contained different concentrations of LAS but all experiments were carried out under the same conditions of temperature, dissolved oxygen and light intensity.

In Na_2SO_4 solutions

Figure 3 shows that the decomposition of LAS in Na_2SO_4 solution with no metal electrode in it,

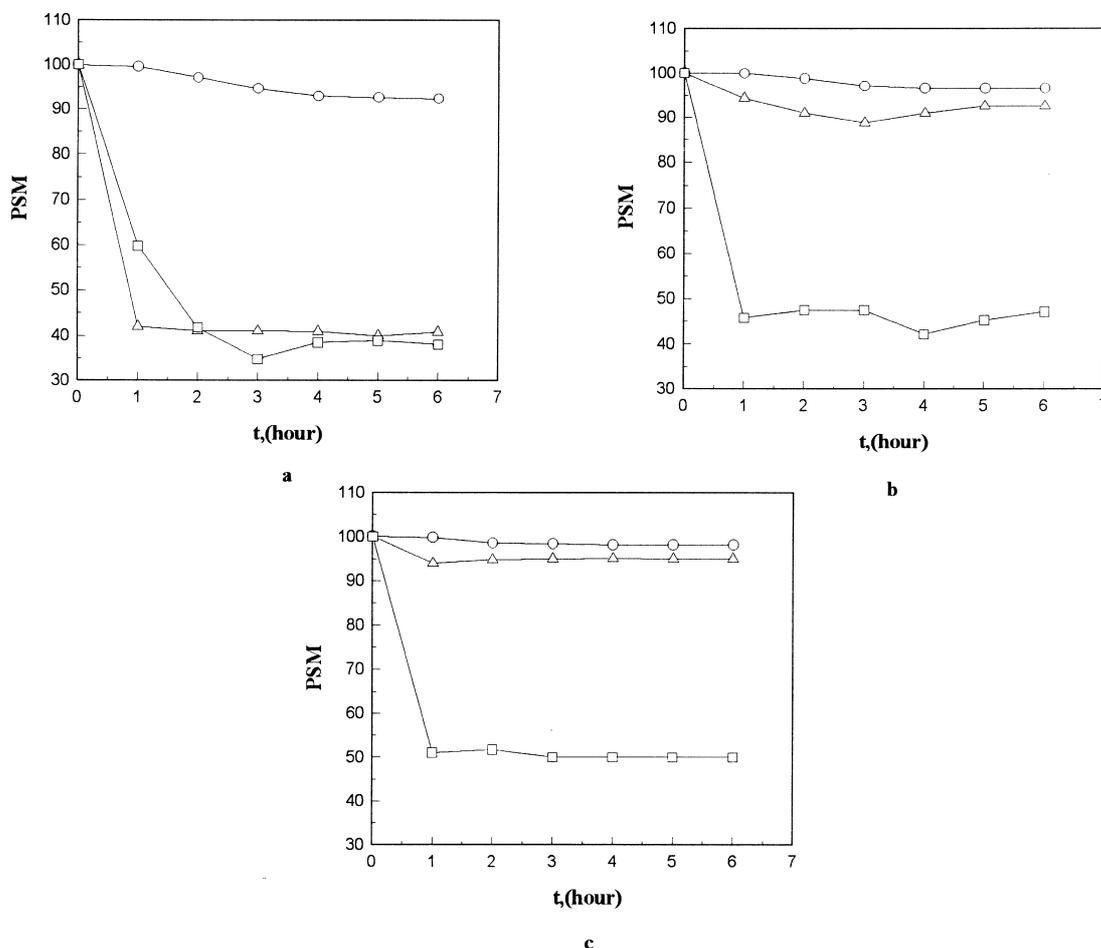


Fig. 3. The current density potential curves obtained in 0.1 M NaCl + xppm LAS solution at pH = 8 (a, x = 0; b, x = 30; c, x = 150; d, x = 300).

is very small (marked 0). The percentages of surface active material (PSM) remaining in solution at the end of 4 h with no metal in it are ~92, ~96 and ~98 for 30, 150 and 300 ppm LAS, respectively.

As seen from Fig. 3(a), when a potential of +1.6 V is applied (marked Δ), the PSM in 30 ppm solution is ~40 at the end of the first hour and remains constant at this value until the end of the experiment. In solutions with 150 and 300 ppm LAS, the PSM is ~90 after 2 h [Fig. 3(b)] and ~94 after 1 h [Fig. 3(c)] respectively, and these values do not change further with time.

When 20 V is applied between the Pt–Pt couple from a direct current source (marked \square) the PSM of LAS drops rapidly at all concentrations. For 30 ppm LAS the PSM is determined as ~38 after 3 h [Fig. 3(a)]. For 150 ppm LAS the PSM is ~45 [Fig. 3(b)] and for 300 ppm, LAS PSM is ~50 after 1 h [Fig. 3(c)]. At these concentrations the PSM does not change any more after 1 h.

In NaCl solutions

The changes of surface active material concentration (PSM) with time in NaCl solution containing various concentrations of linearalkylbenzenesulfonate are shown in Fig. 4. With no electrode in it (marked 0) the PSM of 30 ppm LAS after 4 h is ~96, of 150 ppm LAS it is ~97 and PSM of 300 ppm LAS after 2 h is ~99. As seen from Fig. 4(a) when a voltage of +1.6 V is applied (marked Δ) the PSM remaining in solution after 4 h is ~90 from 30 ppm LAS [Fig. 4(a)]; the PSM of 150 ppm LAS is ~94 [Fig. 4(b)] and that of 300 ppm LAS after 3 h is about 99 [Fig. 3(c)].

When a voltage of 20 V is applied from the direct current source (marked \square), the concentration of LAS in solution decreases a little compared to that in a plain container with no electrode in it and when +1.6 V is applied. The PSM of 30 ppm LAS is ~88 [Fig. 4(a)] after 1 h, that of 150 ppm LAS [Fig. 4(c)] is ~96 after 3 h and these values remain constant with time after these periods.

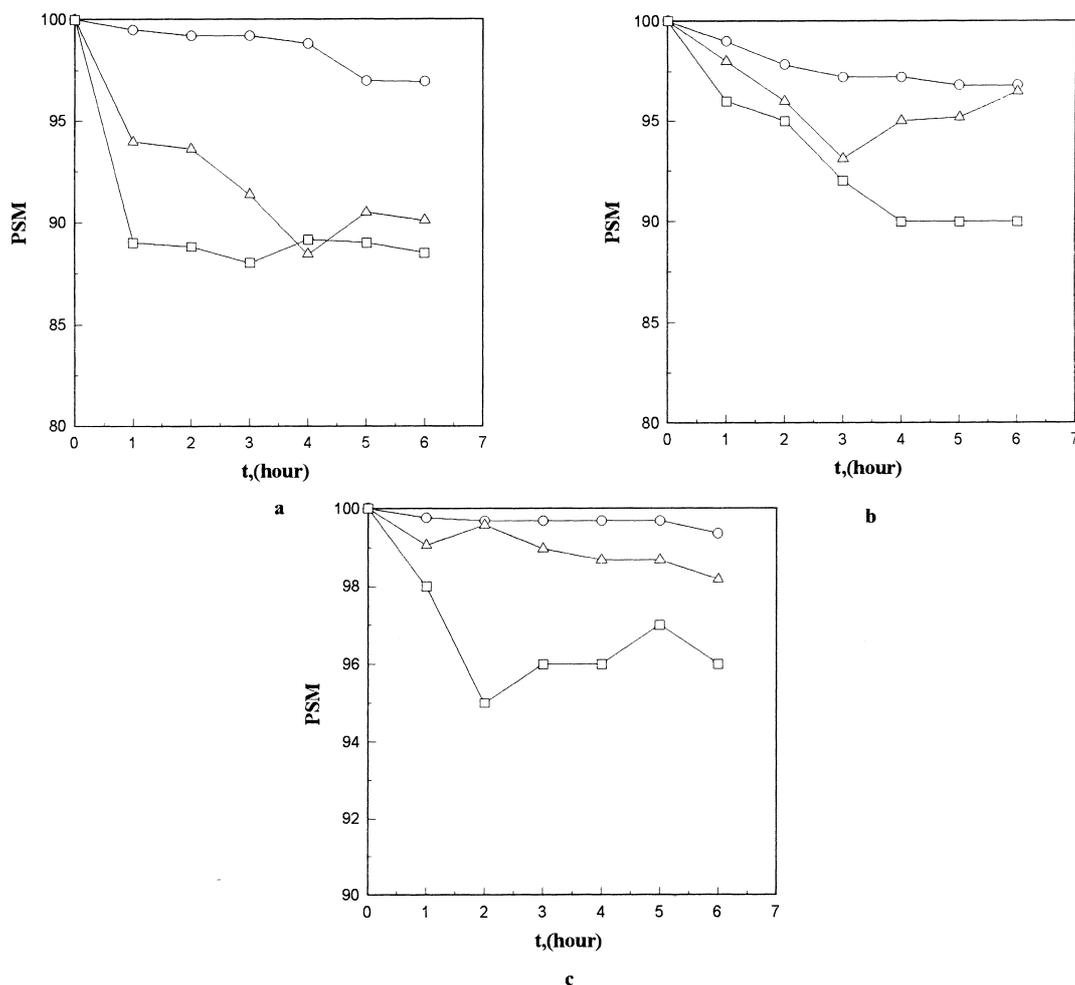


Fig. 4. The change of the surface active material concentrations (PSM) with time in 0.05 M NaCl solution at pH = 8 containing (0, solution in a container with no metal in it; △, using the potentiostat at +1.6 V; □, applying a voltage of 20 V from a direct current source). a, 30 ppm LAS; b, 150 ppm LAS; c, 300 ppm LAS.

DISCUSSION AND CONCLUSION

In all test media the cathodic events taking place on a Pt electrode are the reduction of O_2 and/or H^+ . At pH = 8, the reversible electrode potentials of these reduction reactions are $E_{O_2/H_2O} = 0.758$ V and $E_{H^+/H_2} = -0.472$ V. According to these values, in all solutions studied, the oxygen reduction takes place at a certain overpotential between the equilibrium potentials and -0.5 V (Figs 1 and 2). The oxygen overpotentials determined under these working conditions are given in Table 2.

Apparently, in all solutions, as the LAS concentration increases the η_{O_2} increases but this increase in η_{O_2} is greater in sulphate than in chloride solutions. This difference may be attributed to the adsorption of LAS molecules on the platinum electrode surface. The amount of LAS adsorbed on the surface is proportional to the concentration of LAS

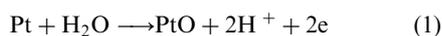
in the solution. However, the lower oxygen overpotential in chloride compared to sulphate solutions could be due to the fact that the chloride ion is adsorbed selectively. Thus in chloride solutions, while the adsorption of LAS with a micromolecular structure is diminished, the reaching of O_2 molecules with the metal surface becomes easier. The oxygen reduction in sulphate solution containing various concentrations of LAS reaches a limiting

Table 2. Oxygen overpotentials (η_{O_2} , due to LAS in Na_2SO_4 and NaCl solutions (V)

LAS Con. (ppm)	η_{O_2} (V) 0.05 M Na_2SO_4	η_{O_2} (V) 0.1 M NaCl
0	-0.658 (0.100-0.750)	-0.158 (0.600-0.758)
30	-0.658 (0.100-0.758)	-0.158 (0.600-0.758)
150	-0.708 (0.050-0.758)	-0.458 (0.300-0.758)
300	-0.808 (-0.050-0.758)	-0.708 (0.050-0.758)

current value in the potential region -0.5 V – -1.1 V and between -0.6 V – -0.95 V in chloride solution (Figs 1 and 2). The increase in current-density when the applied cathodic potential reaches -1.0 V shows that the reduction of H^+ starts in all solutions; when the reversible electrode potential of hydrogen is considered we can say that this hydrogen reduction on Pt starts after an overpotential of -0.628 V [-1.1 V – -0.472 V] in all sulphate solutions, and after -0.478 V [-0.95 V – -0.472 V] in all chloride solutions.

As seen in Fig. 1(a), in sulphate solution containing no LAS a current peak arises in the anodic region at 0.45 V/SCE (0.719 V/SHE). It is said that Pt_4OH and Pt_2OH are formed on Pt anode surfaces in this range (Santos and Giordano, 1984; Olivi *et al.*, 1996; Vasiliev and Sarghisyan, 1996). Beyond this current peak, in the passivation region where the current-density does not change, the following reactions are said to take place (Van Muylder and De Zoubov, 1958):



$$\begin{aligned} E &= 0.980 - 0.0591 \text{ pH} \\ &= 0.5072 \text{ V(SHE)} = 0.752 \text{ V(SCE)} \text{ at } \text{pH} = 8 \\ \text{PtO} + \text{H}_2\text{O} &\longrightarrow \text{PtO}_2 + 2\text{H}^+ + 2\text{e} \quad (2) \end{aligned}$$

$$\begin{aligned} E &= 1.045 - 0.0591 \text{ pH} \\ &= 0.5722 \text{ V(SHE)} = 0.822 \text{ V(SCE)} \text{ at } \text{pH} = 8 \end{aligned}$$

As can be seen in Fig. 1(a) that event takes place in the potential interval 0.7 V – 1.0 V . Accordingly, in this potential interval, the Pt surface is covered with PtO and PtO_2 . This process, which takes place in a sulphate solution, does not occur in a chloride solution [from comparison of Fig. 1(a) with Fig. 2(a)]. The oxide accumulation at the Pt surface is partly prevented by selective adsorption of the chloride ions on the surface (Snell and Keenan, 1981). When LAS is added to sulphate solutions, the reaction $\text{Pt} + \text{LAS} \rightarrow \text{Pt(LAS)} \text{ ads.}$ is added to the currently known reactions at platinum. The addition of this reaction shifts the equilibrium potential to more negative values and, due to this, the oxygen overpotentials η_{O_2} are increased. Consequently the current peak observed in sulphate solutions without LAS under anodic potentials is not observed in sulphate solutions containing LAS (Fig. 1). In LAS-containing solutions, during the potential interval of 0.3 V – 1.0 V , current-density of 0.013 mAcm^{-2} remains constant and possibly reactions 1 and 2 take place. Because of this, the platinum surface is covered with Pt(LAS) ads., PtO and PtO_2 in the LAS-containing media in this potential interval. The oxidation of water to oxygen on the other hand starts at the same potential in

all solutions with or without LAS ($\sim 1.0\text{ V}$, Fig. 1). This potential is in agreement with the values in literature (Bockris and Reddy, 1977). In chloride solution the current-density (0.013 mAcm^{-2}) remains constant in the potential interval 0.5 V – 1.0 V as the LAS concentration increases (150 ppm and 300 ppm LAS) [Fig. 2(c,d)]. This shows that LAS molecules do become adsorbed on the Pt surface despite the fact that chloride ions are selectively adsorbed.

When the discharge potentials are considered, in the electrolysis of $0.05\text{ M Na}_2\text{SO}_4$ and 0.1 M NaCl solution at $\text{pH} = 8$ using Pt electrodes, in both electrolytes hydrogen is evolved at the cathode. On the anode, O_2 is formed in sulphate solutions the theoretical discharge potential of oxygen is 1.23 V , but under the effect of a total of 0.961 V overpotential ($\eta_{\text{ox.}} + \eta_{\text{hyd.}} = 0.721 + 0.24$) this value becomes 2.188 V . Accordingly, in sulphate solutions with and without LAS, the experimental and theoretical discharge potentials are almost the same. In chloride solutions, in addition to oxygen, chlorine is also discharged at the anode. Because of this two different experimental discharge potentials can be detected in chloride solutions. On the Pt electrode the experimental discharge potential of chlorine is almost the same as the theoretical value determined (theoretical discharge potential of $\text{Cl}_2 = 1.42\text{ V}$). When the discharge potentials of oxygen and chlorine are considered, it will be seen that first chlorine and then oxygen is evolved. In chloride solutions the chloride ions are selectively adsorbed on the Pt surface and cause an overpotential in the oxidation of OH^- ions and correspondingly causes an increase in the experimental discharge potential of oxygen. The presence of surface active material in the media after 6 h, which decomposes as a result of the electrooxidation of LAS molecules on a Pt surface, has been detected quantitatively by UV spectrophotometry. The extent of degradation of LAS molecules at different concentrations in various solutions (sulphate, chloride) by different methods (by itself, applying $+1.6\text{ V}$, applying 20 V) are given in Figs 3 and 4. The degradation of linearalkylbenzenesulfonate (LAS) has been investigated by different test methods (Takada and Ishiwatari, 1987; Holt *et al.*, 1989). From the studies carried out under natural environment conditions, it is argued that the length of an alkyl chain and the position of the carbon atom in LAS to which the alkyl chain is attached do not have any effect on the rate of biological degradation of LAS (Ward and Larson, 1989; Larson, 1984). There is very little information related to the kinetics of biological breaking of the benzene ring in LAS (Takada and Ishiwatari, 1987; Holt *et al.*, 1989). Many researchers have claimed that the half-life of LAS is 15–33 h under natural conditions where there is no chloride and it is 15–21 days in solutions contain-

ing 3.5% NaCl salt (Hand and Williams, 1987; Quiroga and Sales, 1989; Yazıcı *et al.*, 1993). As can be seen from Figs 3 and 4, when left alone with no metal in it the rate of degradation of LAS in sulphate and chloride solution is small. At the same concentrations, the rate of degradation in chloride solution is much smaller than that in sulphate solution as pointed out elsewhere (Quiroga and Sales, 1989).

When a potential of +1.6 V is applied to sulphate solutions the degradation of the surface-active material is more observable at low concentrations (30 ppm LAS). Because the Pt electrode surface is not large enough, and its porosity is low, the concentration of LAS increases, as the amount of surface active material being degraded is decreasing (Fig. 3). In sulphate solutions an oxide layer is formed on the Pt anode (Fig. 1) and, while O₂ is being evolved, it increases the degradation rate of LAS molecules adsorbed on the Pt surface by electrooxidation. In chloride solutions the amount of surface-active material degrades when a potential of +1.6 V is applied and does not change much compared to the PSM of plain solutions with no metal in it because the chloride ions are selectively adsorbed on the Pt surface and inhibit the formation of PtO and PtO₂. While a marked decrease in the amount of degraded surface active material has been observed in sulphate solutions at all concentrations of LAS when a potential of 20 V is applied between the two electrodes, in chloride solutions not much decrease has been noticed compared to the values determined by other methods. When a 20 V potential is applied, plenty of the O₂ generation occurs at the anode in sulphate solutions and an increase in O₂ concentration of the solution brings about the simultaneous degradation by chemical and electrochemical processes. Consequently the rate of degradation in all test media increases. On the other hand in chloride solutions, when a potential of 20 V is applied, more Cl₂ is evolved at the anode and the O₂ formed is not sufficient to degrade or decompose the LAS.

In conclusion we can say that when all the conditions are the same (temperature, dissolved oxygen, pH) the important factors affecting the degradation of LAS are the type of the solution, the concentration of LAS, the test method and the potential applied. These considerations may be appropriate for other organic compounds and also different materials.

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