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ORIGINAL PAPER

Electrochemical behaviour of phenylethanolamine at gold in aqueous solution

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Abstract The electrooxidation of phenylethanolamine (2-amino-1-phenylethanol) at a gold electrode in alkaline electrolyte has been studied. Measurement of the differential capacitance of the electric double layer versus the electrode potential has shown that the adsorption of phenylethanolamine at the gold-solution interface plays a significant role in the oxidation mechanism. The effect of amine concentration, electrolyte pH and potential scan rate on the electrooxidation is analysed.

Keywords Gold electrode · Aliphatic amines · Alcoholoamines · Adsorption · Electrochemical oxidation · Electrocatalysis

1 Introduction

Of the noble metals, gold and platinum are the most active and most often used electrocatalysts for the electrooxidation of organic substances. Gold stands out because of its good electrocatalytic activity in alkaline environments [1–17], whereas platinum is the most active in acid media [1, 18, 19]. Oxidation of organic substances at platinum is accompanied by a characteristic, though highly undesirable, effect of surface blocking by strongly adsorbed intermediate species [19]. This effect does not apply to gold which has no vacancy in its d-bands and thus is characterized by relatively poor adsorption properties [2, 20, 21] as compared with platinum. Though these adsorption properties of gold account for its weak catalytic activity in acid solution, in alkaline solutions, when hydroxide ions are adsorbed on the gold surface [1–17], gold proves to be a more effective catalyst for dehydrogenation of organic compounds than platinum.

The compounds selected for the study presented here are aliphatic amines and their derivatives. They have found application in different branches of industry, agriculture and pharmacology [22, 23]. Most amines are toxic, thus their monitoring and detection are of great significance in environmental protection [22]. Up to date, the literature gives rather scarce experimental evidence of electrochemical behaviour of aliphatic amines. On gold, it has been established that oxidation of ethylamine in alkaline media occurs concomitantly with gold oxide formation [24–27]. The relationships between the adsorption properties and electrocatalytic oxidation of a homologous series of aliphatic amines ranging from methylamine [CH_3NH_2] to butylamine [$C_4H_9NH_2$] have been evaluated and discussed [28–33]. The effect of molecular structure on isomeric butylamines has been analysed [34]. More recently, the electrochemical behaviour of benzylamine [$C_6H_5CH_2NH_2$], 2-phenylethylamine [$C_6H_5CH_2CH_2NH_2$] and 2-(4-hydroxyphenyl)ethylamine (tyramine) [$C_6H_4(OH)CH_2CH_2NH_2$] has been studied [35]. Derivatives of phenylethylamine exist in biological systems as alkaloids and hormones [22, 23]. They play an important role in brain chemistry because some are neurotransmitters in the mammalian central nervous system [36]. Thus better understanding of different properties of these amines is of particular importance from both fundamental and practical points of view.

This paper deals with electrooxidation of phenylethanolamine [$C_6H_5CH(OH)CH_2NH_2$]. Additional information about adsorption of the molecules under investigation was obtained from the measurements of differential capacitance

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of the electric double layer versus potential. Comprehensive recognition of adsorption phenomena at electrified interfaces is of the utmost importance for understanding the mechanisms of reactions in electrocatalysis.

2 Experimental

The apparatus used to record cyclic voltammograms (*j–E* curves) and differential capacitance of the electric double layer versus electrode potential curves (*C–E*) was described earlier [3].

The solutions were prepared using water purified in a Millipore Milli-Q system, phenylethanolamine of Fluka, NaOH and $NaClO_4$ of Merck. All chemicals were analytical reagent grade and were used without further purification. All solutions under investigation were deaerated with high purity argon. The electrochemical measurements were carried out at 295 K.

Measurements were performed in a conventional three-electrode cell using a gold bead (99.999% purity) of 0.3 cm^2 geometric area as a working electrode and a gold sheet as a counter-electrode. A hydrogen electrode (RHE) in the supporting electrolyte solution as proposed by Will et al. [37, 38] served as a reference electrode. For comparison with published data all the potentials are given versus the saturated calomel electrode (SCE).

Prior to each series of measurements with amine, the working electrode was electrochemically activated by cycling ($dE/dt = 0.1$ V s^{-1}) in the potential range between $E = -1.1$ V and $E = 0.1$ V in 0.1 mol dm^{-3} NaOH and between $E = -1.0$ V and $E = 0.6$ V in 0.02 mol dm^{-3} $NaClO_4$, where no faradaic reactions occur on the gold surface. This procedure allows the avoidance of structural changes on the gold surface.

The *C–E* curves were obtained by superimposing an ac sinusoidal voltage signal (15 s^{-1}, 0.005 V) on a slow linear electrode potential scan ($dE/dt = 0.005$ V s^{-1}). A slower scan rate of 0.002 V s^{-1} did not affect the *C–E* curves observed. Stirring of the solutions had no effect on the course of the *C–E* curves, which indicated that the mass transport did not limit the rate of adsorption–desorption processes of amine at the bulk concentrations studied. The equilibrium of the adsorption–desorption was checked at different frequencies. For frequencies in the range 5–40 s^{-1} no frequency dispersion of the differential capacity was observed in the potential range $-0.95\ \text{V} < E < 0.1$ V and $-0.95\ \text{V} < E < 0.05$ V in 0.1 mol dm^{-3} NaOH and 0.02 mol dm^{-3} $NaClO_4$, respectively. These potential ranges narrow in the presence of phenylethanolamine in the supporting electrolyte to $-0.4\ \text{V} < E < 0.1$ V in NaOH and to $-0.45\ \text{V} < E < 0.05$ V in $NaClO_4$ at all the adsorbate concentrations studied. These findings confirm that the differential capacity data in the above-mentioned potential range can be considered at equilibrium.

3 Results and discussion

The cyclic voltammetric responses of a gold electrode in 0.1 mol dm^{-3} NaOH supporting electrolyte without and with phenylethanolamine at increased concentration are shown in Fig. 1. The *j–E* curve for a bare electrode reveals two peaks: an anodic peak at $E = 0.33$ V and a cathodic one at $E = 0.15$ V attributed to a gold oxide film formation and to its reduction, respectively [39]. As follows from the cyclic voltammograms obtained the oxidation of phenylethanolamine at gold proceeds irreversibly in two electrode potential regions, Fig. 1. Starting from the negative potential limit during the positive sweep as the electrode potential becomes increasingly positive, a well defined anodic peak with a maximum at $E = -0.05$ V (peak I) appears in the first cycle on the cyclic voltammogram

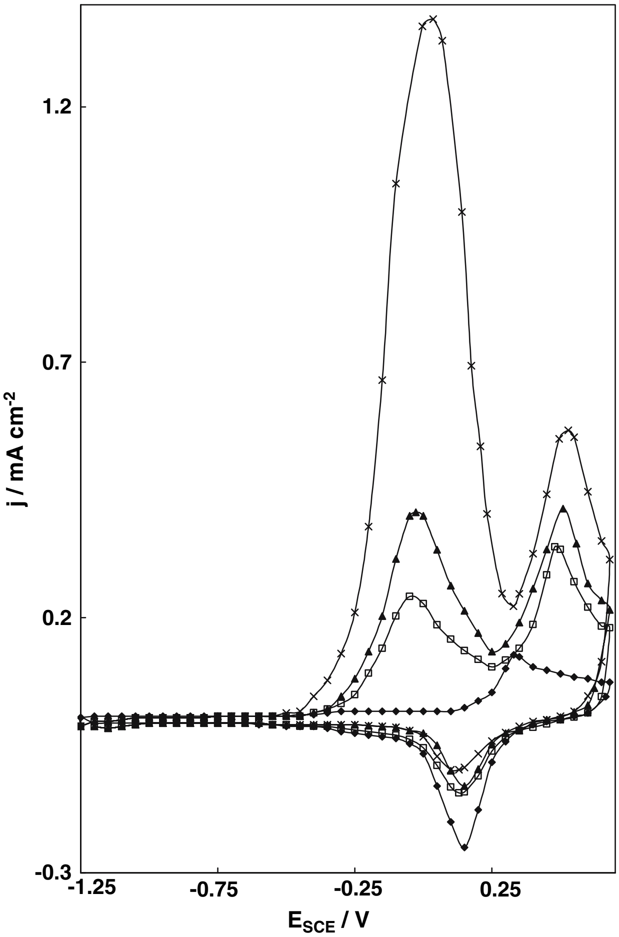


Fig. 1 Cyclic voltammograms of gold electrode in 0.1 mol dm^{-3} NaOH (■) and with phenylethanolamine: (□) 0.0001 mol dm^{-3}; (▲) 0.001 mol dm^{-3}; (×) 0.005 mol dm^{-3}. $dE/dt = 0.1$ V s^{-1}

obtained for phenylethanolamine of 0.0001 mol dm^{-3} (C_A). The potential range in which the first peak appears overlaps with that in which the adsorption of hydroxide ions on gold electrode occurs [39]. This suggests that the OH^- ions play an important role in the mechanism. In the opinion of a number of authors the adsorbed hydroxide ions participating in oxidation of organics on the gold electrode are partly discharged according to the equation [2, 5, 6, 8–11, 15–17] and the interface does not behave as the ideal capacitor:

$$Au + OH^- - \delta e^- \rightarrow AuOH_{ad}^{(1-\delta)^-} + e^- \quad (1)$$

Phenylethanolamine is also oxidized in the potential region of gold oxide formation [39]. The second anodic peak appears on the cyclic voltammograms at $E = 0.48$ V (peak II). When comparing the response of the gold electrode recorded in the supporting electrolyte solution with those obtained in the presence of alcoholamine, it is seen that the charge used for the reduction of gold oxide in the presence of alcoholamine is smaller than that in the solution without the solute. This observation points to a participation of gold oxide in the oxidation of phenylethanolamine, as for aliphatic amines without aromatic substituent [32–34] and for methanol oxidation on polycrystalline gold [40, 41]. Since the potential range in which the first peak appears on the *j*–*E* curve overlaps that in which monohydric aliphatic alcohols are oxidized on gold [2, 6] and this peak appears in the first anodic cycle starting from the negative potential limit, there is no doubt that the $-OH$ group in alcoholamine molecule is oxidized first and is followed by the oxidation of the $-NH_2$ group.

For the alcoholamine studied a characteristic feature is an increase in the anodic current density with increasing solute concentration, Fig. 1. In the positive scan the maxima in the oxidation peaks shift positively as the bulk alcoholamine concentration increases from 0.0001 mol dm^{-3} to 0.005 mol dm^{-3}. Such a shift is typical of reactions involving molecules adsorbed on the electrode surface. An increase in the adsorbate coverage may generate a decrease in the hydroxide ions and gold oxide coverage. This conclusion is supported by the fact that an increase in the anodic peak is accompanied by a decrease in the cathodic peak.

The influence of the competitive adsorption of phenylethanolamine and hydroxide ions was verified in experiments in which the concentration of one component was kept constant while that of the other was varied. The plots of the logarithm of current density versus the logarithm of substrate and supporting electrolyte concentration at constant electrode potentials are depicted in Figs. 2 and 3, respectively. The slopes of the straight lines of the appropriate log *j*–log *c* relationships give the reaction order with respect to the substrate concentration, $z_A = \mathrm{dlog}\, j/\mathrm{dlog}\, c_A$ and to the hydroxide anion concentration, $z_{OH}- = \mathrm{dlog}\, j/\mathrm{dlog}\, c_{OH}-$, respectively. For constant hydroxide ions concentration, $c_{OH}- = 0.1$ mol dm^3 and for $c_A \leq 0.01$ mol dm^3 the resulting z_A is 0.67 ± 0.02 ($R^2 = 0.993$) and 0.26 ± 0.01 ($R^2 = 0.993$) for the first and the second peak, respectively. For $c_A = 0.001$ mol dm^3 and for $c_{OH}- \leq 0.1$ mol dm^{-3} the $z_{OH}-$ is 0.69 ± 0.02 ($R^2 = 0.993$) and 0.24 ± 0.01 ($R^2 = 0.998$) for the first and the second peak, respectively. The reaction orders, z_A and $z_{OH}-$ decrease somewhat with decreasing potential sweep rate from 0.1 V s^{-1} to 0.01 V s^{-1}. The fractional reaction order shows that the kinetics of phenylethanolamine oxidation is influenced by the relative surface concentration of organic species and hydroxide anions. The reaction rate decreases after passing a maximum when the amount of either organic molecules or OH^- prevails at the electrode-solution interface (see Figs. 2, 3).

It has been observed that the scan rate influences the oxidation of alcoholamine on gold. An increase of *v* is

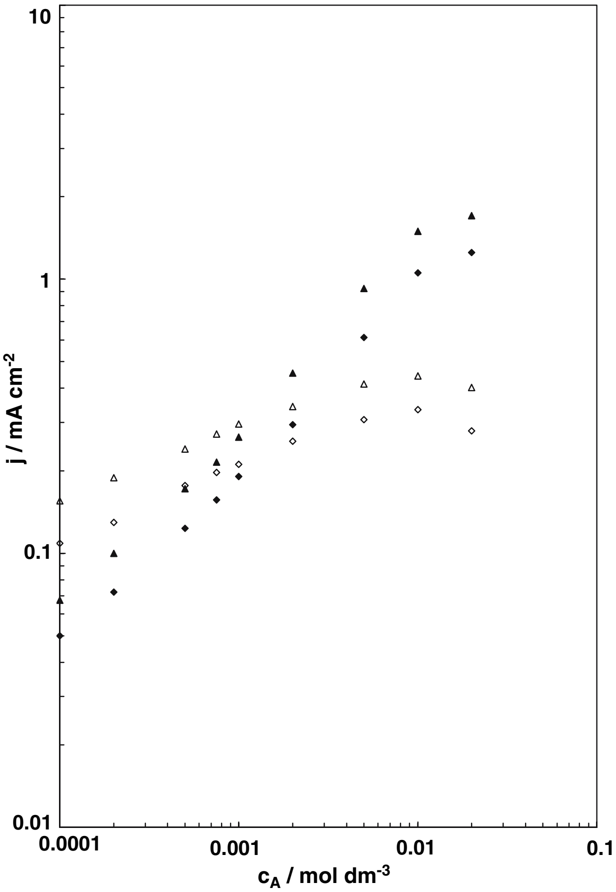


Fig. 2 Dependence of the current density on phenylethanolamine concentrations in 0.1 mol dm^{-3} NaOH at: (◆) $E = -0.15$ V; (▲) $E = -0.1$ V for peak I and at: (◇) $E = 0.4$ V; (Δ) $E = 0.45$ V for peak II. d*E*/d*t* = 0.1 V s^{-1}

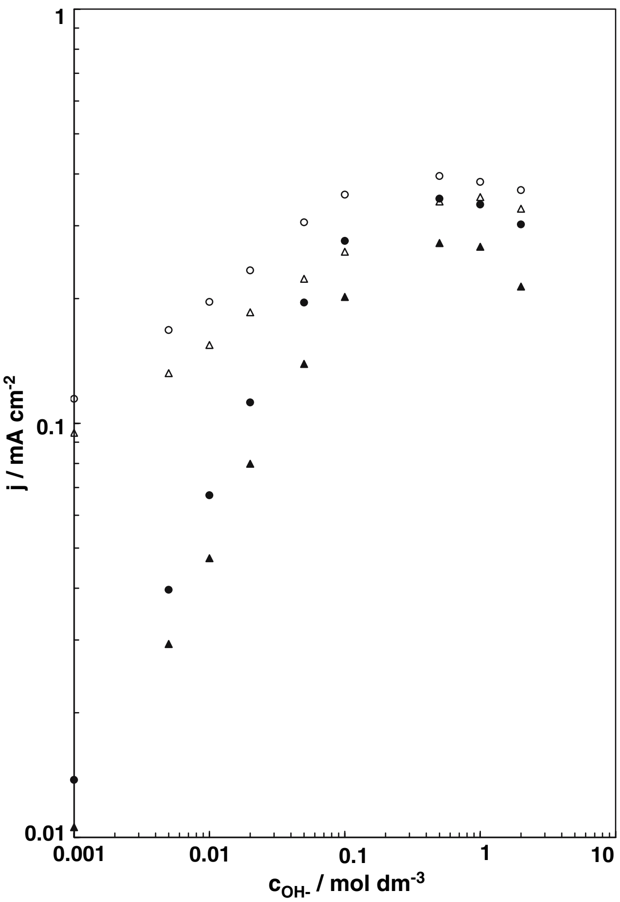


Fig. 3 Dependence of the current density on NaOH concentration in 0.001 mol dm^{-3} of phenylethanolamine at: (▲) $E = -0.15$ V; (●) $E = -0.1$ V for peak I and at: (Δ) $E = 0.4$ V; (○) $E = 0.45$ V for peak II. dE/dt = 0.1 V s^{-1}

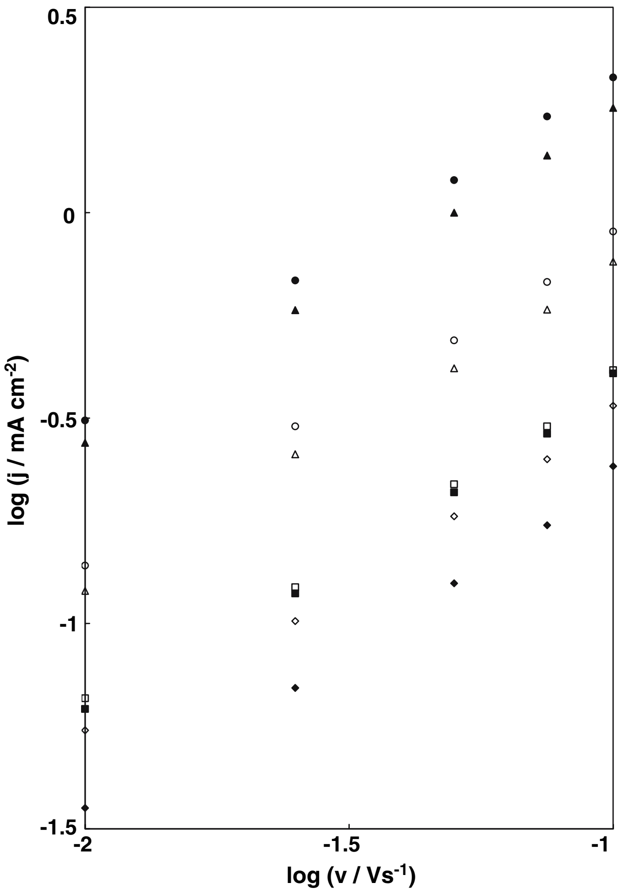


Fig. 4 The logarithm of peak current density oxidation versus the logarithm of potential sweep rate for peak I: (◆) 0.0001 mol dm^{-3}; (■) 0.001 mol dm^{-3}; (▲) 0.01 mol dm^{-3}; (●) 0.1 mol dm^{-3} and for peak II: (◇) 0.0001 mol dm^{-3}; (□) 0.001 mol dm^{-3}; (Δ) 0.01 mol dm^{-3} and (○) 0.1 mol dm^{-3} of phenylethanolamine on gold electrode in 0.1 mol dm^{-3} NaOH

accompanied by an increase in current density and the positive movement of the oxidation peak potential. This confirms the irreversibility of the electrocatalytic process. The relationship of the logarithm of the peak current density versus the logarithm of the potential sweep rate depicted in Fig. 4 can be expressed by the following linear regression equation: $\log j_p = 0.8\log v + 0.18$ with a correlation coefficient $R^2 = 0.996$ for 0.0001 mol dm^3 of phenylethanolamine. A slope of $\mathrm{d}\log j_p/\mathrm{d}\log v = 0.8 \pm 0.02$ suggests complicated kinetics of phenylethanolamine oxidation on gold and points to a mixed, i.e. diffusion–adsorption rate control [42] as observed for the oxidation of aliphatic amines without aromatic substituent [32–34].

In order to gain a greater insight into the rate determining step of phenylethanolamine oxidation, Tafel analysis of voltammograms was made. In the case of 0.001 mol dm^3 of phenylethanolamine, the slopes $\mathrm{d}E/\mathrm{d}\log j = 2.303RT/\alpha nF$ for the linear parts of the plots in $\log j$–E coordinates (Fig. 5) give values of 0.22 ± 0.01 V ($R^2 = 0.998$) and 0.44 ± 0.03 V ($R^2 = 0.995$) per decade of current density for the first and the second peak, respectively. The changes in the dE/log j slopes with increasing alcoholamine concentration can be neglected. The electron transfer coefficient, αn derived from the Tafel slopes, in every case lower than 0.5, is compatible with the assumption that the transfer of the first electron from substrate molecule in the adsorbed state to the electrode is the step determining the overall reaction rate. Further steps proceed so fast that they have no influence on the rate. This conclusion is in accordance with the results of the sweep rate study. As follows from the kinetic theory of electrode reactions, in the case of the simple rate determining step with no contribution of adsorption, a Tafel slope of only 0.12 V decade^{-1} should be found [42].

The adsorption of phenylethanolamine on gold is confirmed by the measurements of the double layer capacitance of the electrode-solution interface as a function of the electrode potential. The representative set of the C–E curves in 0.1 mol dm^{-3} NaOH as a supporting electrolyte

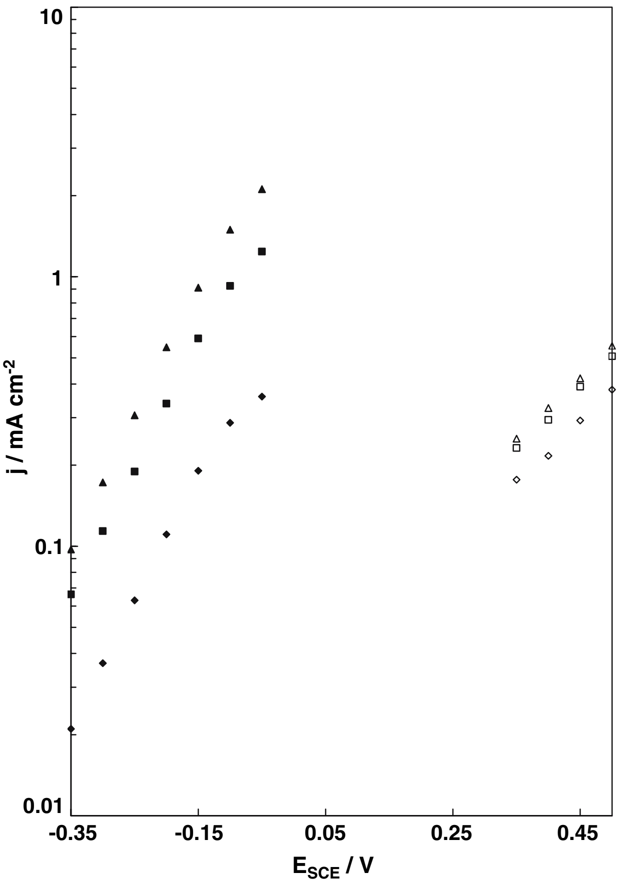


Fig. 5 Dependence of current density on gold electrode potential in 0.1 mol dm^{-3} NaOH for peak I of phenylethanolamine: (◆) 0.001 mol dm^{-3}; (■) 0.005 mol dm^{-3}; (▲) 0.01 mol dm^{-3} and for peak II: (◇) 0.0001 mol dm^{-3}; (□) 0.001 mol dm^{-3}; (Δ) 0.01 mol dm^{-3}. $dE/dt = 0.1$ V s^{-1}

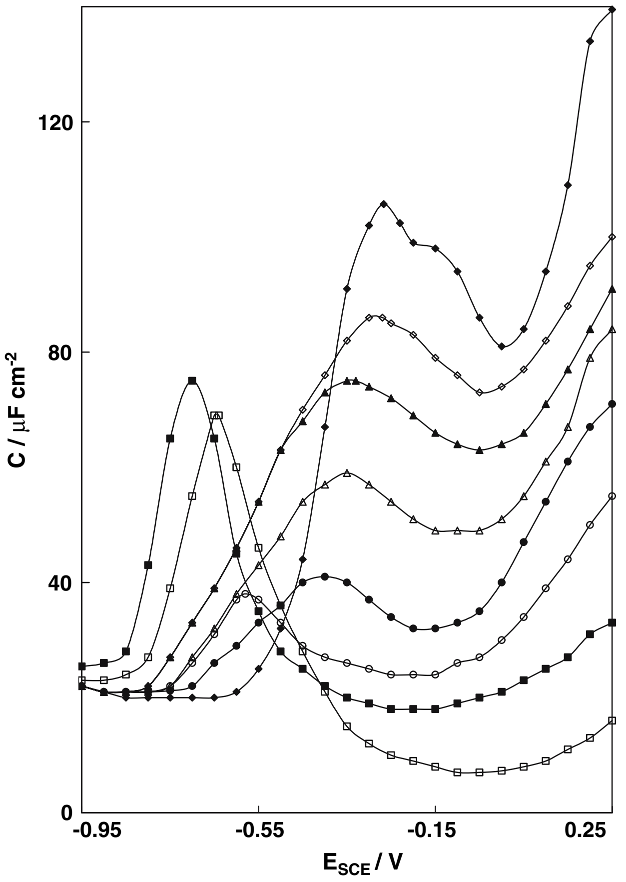


Fig. 6 Differential capacitance versus potential for the gold electrode in 0.1 mol dm^{-3} NaOH (◆) with increasing phenylethanolamine concentration: (◇) 0.00005 mol dm^{-3}; (▲) 0.0001 mol dm^{-3}; (Δ) 0.005 mol dm^{-3}; (●) 0.001 mol dm^{-3}; (○) 0.01 mol dm^{-3}; (■) 0.02 mol dm^{-3}; (□) 0.05 mol dm^{-3}. $dE/dt = 0.005$ V s^{-1}

and at increased concentration of phenylethanolamine is shown in Fig. 6. A gradual suppression of the capacitance curves with increasing bulk adsorbate concentration in the electrolyte takes place in the potential range of the characteristic minimum in the vicinity of the potential of zero charge (E_{pzc}) of the gold electrode [40, 43–45] and in that of the adsorption–desorption maximum on the negatively charged surface. This behaviour proves the substitution of water with alcoholamine molecules at the gold surface. As in the case of other aliphatic amines adsorbed on gold [24–34] a relatively strong adsorption of phenylethanolamine is manifested by the fact that the *C*–*E* curves for the highest solute concentrations do not merge at the negative potential limit with the corresponding *C*–*E* curve for supporting electrolyte solution. This indicates that even at such negative electrode potentials some adsorbate molecules still remain at the gold surface.

The potential range in which phenylethanolamine adsorbs on the gold-alkaline solution interface coincides with that in which the adsorption of hydroxide ions occurs [2, 6, 11, 39, 40]. Thus a quantitative evaluation of the *C*–*E* curves in this medium is not possible because of the competing adsorption of organic molecules and hydroxide ions at the electrode surface and only qualitative information can be obtained. Therefore, additional measurements of differential capacitance with a non-adsorbing supporting electrolyte, 0.02 mol dm^{-3} $NaClO_4$ [43], were made. The results reveal that the potential range of the alcoholamine adsorption as measured with respect to E_{pzc} does not change with pH of the electrolyte solution. The potential of the maximum adsorption of phenylethanolamine at the gold-solution interface, E_{max} was evaluated from the maximum of the relationships between the $\Delta C = C_{\Theta=0} - C_{\Theta}$ versus *E* for a certain solute concentration, Fig. 7. It has been established that thus determined value of $E_{max} = -0.3$ V does not change with increasing solute concentration.

The dependence of the surface coverage $\Theta = (C_{\Theta=0} - C_{\Theta})/(C_{\Theta=0} - C_{\Theta=1})$ (where: $C_{\Theta=0}$, $C_{\Theta=1}$ and C_{Θ} represent differential capacitance of the double layer at

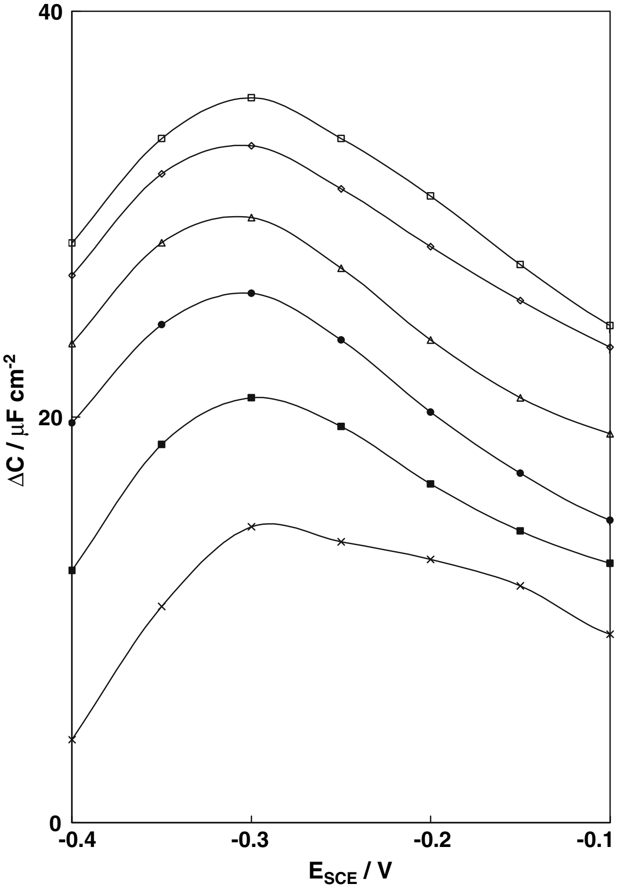


Fig. 7 $\Delta C = C_{\Theta=0} - C_{\Theta}$ versus the gold electrode potential in 0.02 M $NaClO_4$ with increased phenylethanolamine concentration: (□) 0.000001 mol dm^{-3}; (◇) 0.000002 mol dm^{-3}; (Δ) 0.000004 mol dm^{-3}; (●) 0.000006 mol dm^{-3}; (■) 0.00001 mol dm^{-3}; (×) 0.00001 mol dm^{-3}. dE/dt = 0.005 V s^{-1}

$\Theta = 0$, $\Theta = 1$ and at intermediate coverages ($0 < \Theta < 1$), respectively) of the gold electrode on increasing phenylethanolamine concentration in the solution at E_{max}, can be satisfactorily approximated by the Frumkin isotherm [46]: $[\Theta/(1 - \Theta)]\exp^{-2a\Theta} = \beta C$, where: β is the adsorption equilibrium constant and "a" denotes the lateral interaction parameter. Taking as the reference state the unit mole fraction of amine in the bulk of the solution and a monolayer coverage ($\Theta = 1$) of the non-interacting adsorbate molecules at the surface [20, 21, 47–50], β is related to the standard Gibbs energy of adsorption ΔG^0_{ad} by the following relationship: $\beta = [(1/55,5)\exp(-\Delta G^0_{ad}/RT)]$. The magnitude of $\Delta G^0_{ad} = -43.2 \pm 0.05$ kJ mol^{-1} reflects strong interactions between the adsorbate molecule and the electrode surface. The alcoholamine molecules are able to form surface complexes by coordinative interaction between the electrode surface and the electron lone pair at the nitrogen and oxygen atoms.

Finally, analysis of the variation of the peak potential E_p with pH of supporting electrolyte shows that during the electrode process not only electrons but also protons are released from the organic molecules. The peak potentials shift linearly towards less positive values with increasing pH. For the amine studied in the pH range between 11 and 13 the E_p versus pH plots (Fig. 8) are linear with an average slope of -0.060 ± 0.002 V ($R^2 > 0.992$) per unit pH. The magnitude of the dE_p/dpH slope suggests participation of equal numbers of protons and electrons in the electrooxidation reaction.

In the light of the results and literature data [2, 6, 32] the possible reaction pathways for phenylethanolamine electrooxidation on gold in alkaline solution can be proposed as shown in Scheme 1. The first step in the proposed mechanism is the formation of the catalytic $AuOH_{ad}^{(1-\delta)^-}$ layer at the electrode surface. Next, the adsorption of phenylethanolamine is followed by hydrogen abstraction from the alcoholamine molecule via a transient adsorbate complex formed as a result of the interaction between adsorbed substrate molecules and the $AuOH_{ad}^{(1-\delta)^-}$ catalytic sites.

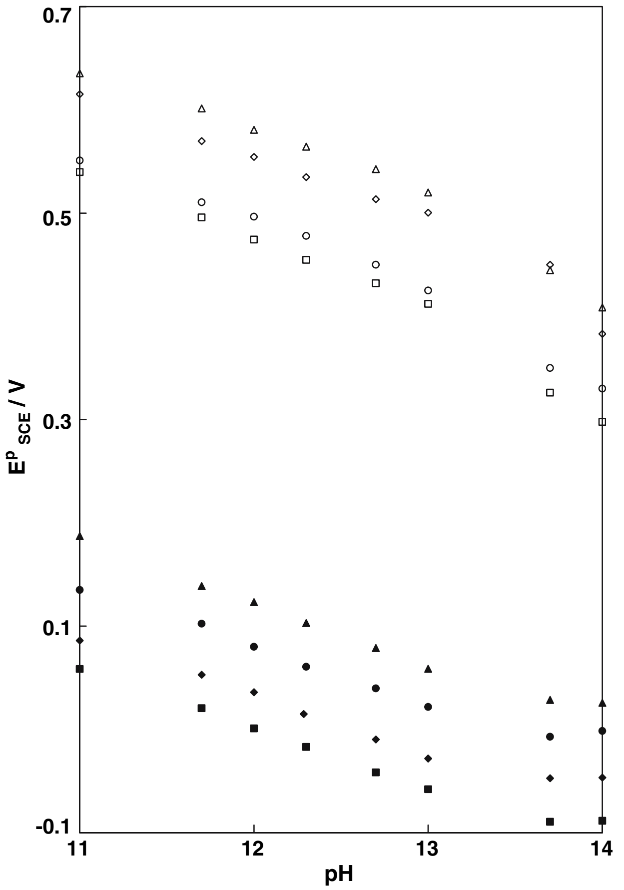
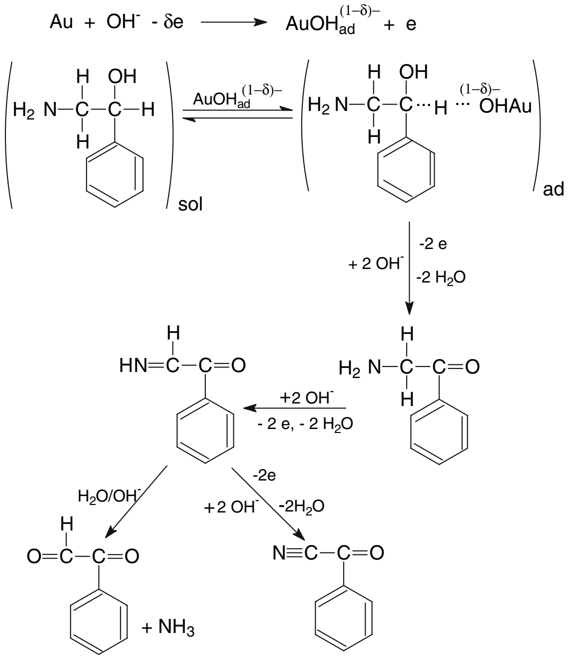


Fig. 8 Dependence of peak potential on pH of the supporting electrolyte for peak I of phenylethanolamine of 0.001 mol dm^{-3}: (◆) $dE/dt = 0.1$ V s^{-1}; (■) $dE/dt = 0.01$ V s^{-1} and of 0.01 mol dm^{-3}: (▲) $dE/dt = 0.1$ V s^{-1}; (●) $dE/dt = 0.01$ V s^{-1}. For peak II of phenylethanolamine of 0.001 mol dm^{-3}: (◇) $dE/dt = 0.1$ V s^{-1}; (□) $dE/dt = 0.01$ V s^{-1} and of 0.01 mol dm^{-3}: (Δ) $dE/dt = 0.1$ V s^{-1}; (○) $dE/dt = 0.01$ V s^{-1}



Scheme 1 Proposition of the reaction pathways for electrooxidation of phenylethanolamine on a gold electrode in an alkaline solution

The heterogeneous dehydrogenation of phenylethanolamine molecule, involving electron transfer to the gold electrode and the formation of water molecule determines the rate of the whole process. Further steps proceed so fast that they have no influence on the rate of organic oxidation. It should be emphasized that alcoholamine molecules may be adsorbed on the gold catalyst surface without being involved in the oxidation reaction, in particular at small coverages of the metal surface with preadsorbed hydroxide ions at low anodic potentials. They may actually inhibit adsorption of further hydroxide ions by blocking the electrode surface. This would account for the observed decrease in the rate of alcoholamine oxidation, at high concentration ($c_A > 0.01$ mol dm^3, Fig. 2) and together with the analogous decrease in the reaction rate observed at high hydroxide ions concentration ($c_{OH} > 0.1$ mol dm^3, Fig. 3) are indicative of the competitive adsorption of alcoholamine molecules and hydroxide ions at gold. Taking into account the magnitude of the dE_p/dpH slope, the CH_2OH in phenylethanolamine molecule can be oxidized to ketoamine which may be next oxidized to ketonimine. The latter compound may be then oxidized to ketonitrile. Moreover, ketoaldehyde can be formed after hydrolysis of ketoimine. Some further details about the mechanism proposed, especially the existence of the intermediate species should be elucidated by using in situ spectroelectrochemical techniques.

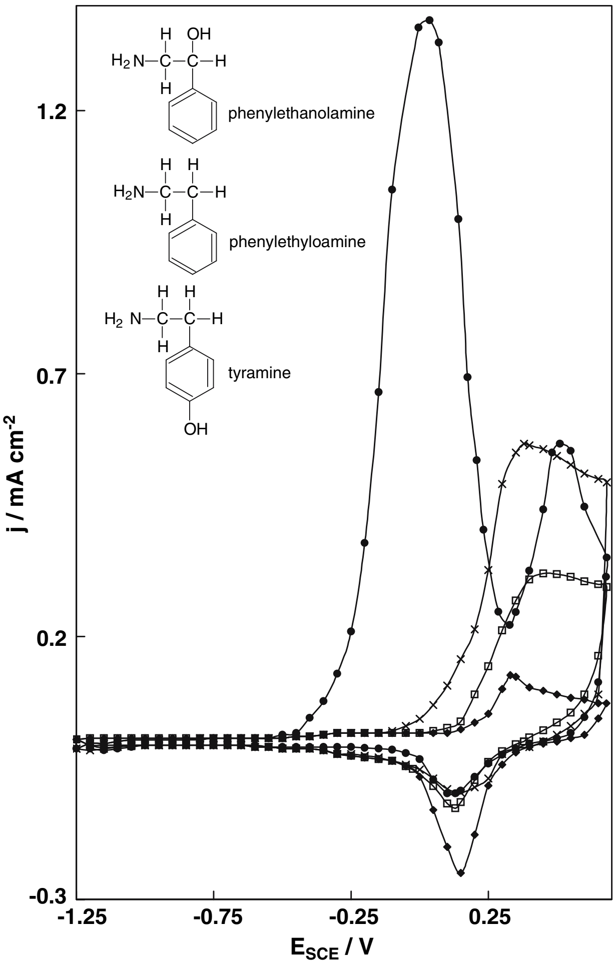


Fig. 9 Cyclic voltammograms of the gold electrode in 0.1 mol dm^{-3} NaOH (◆) and with 0.005 mol dm^{-3} of: (●) phenylethanolamine; (×) fenylethyloamine; (□) tyramine. $dE/dt = 0.1$ V s^{-1}

It is interesting to compare the voltammetric response of a gold electrode in the presence of phenylethanolamine with those obtained for phenylethylamine and tyramine [35]. The carbon backbone in molecules of these compounds has the same length. Phenylethanolamine and tyramine have the same total formulas but the $-OH$ group has the different position in their molecules. Figure 9 reveals clear differences between the electrooxidation activities of above mentioned compounds.

Two major factors are important in evaluation of the catalytic effect of electrodes on electrooxidation process: a decrease in the overpotential of oxidation and an increase in the current density as compared with those of the bare electrode. In this context, the higher catalytic effect of gold is achieved for phenylethanolamine electrooxidation. As follows from Fig. 9, the current density obtained for phenylethanolamine is greater than that observed for fenylethylamine. This is a consequence of greater standard Gibbs energy of adsorption for alcoholamine ($\Delta G^0_{ad} = -43.2 \pm 0.05$ kJ mol^{-1}) as compared with that obtained for phenylethylamine ($\Delta G^0_{ad} = -42.7 \pm 0.04$ kJ mol^{-1}, [35]).

Stronger interactions between the organic molecule and the electrode surface, reflected by the ΔG^0_{ad} values, may facilitate oxidation. The same relation was concluded in the case of alcohols and diols oxidation on gold [6].

Finally, the smaller current density observed for tyramine, must follow from the fact that the hydroxide group in this compound is attached directly to the aromatic ring. Besides the NH_2 group, the OH group linked directly to the aromatic ring is able to interact with the gold surface. This possibility is responsible for blocking the electrode surface for further substrate adsorption and oxidation [35]. As a result, the current density flowing through the electrode during tyramine oxidation was the lowest, in spite of the highest value of ΔG^0_{ad}, for this compound ($\Delta G^0_{\mathrm{ad}} = -46.0 \pm 0.06$ kJ mol^{-1}).

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