

An Investigation of the Inhibition of the Oxygen Reduction Reaction at a Rotating Cylinder Electrode under Isothermal and Controlled Conditions of Heat and Mass Transfer

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Abstract:

The inhibition of the oxygen reduction reaction on a carbon steel rotating cylinder electrode in naturally aerated 600 ppm Cl⁻ solution was studied using an optimum inhibitor blend, i.e., Sodium Nitrite (SN): Sodium Hexametaphosphate (SHMP) = 500:100 obtained via a weight loss technique. Potentiostatic technique, then, was applied at different bulk temperatures and various flow rates using un-inhibited and inhibited solutions under isothermal and controlled conditions of heat transfer.

In an un-inhibited solution and under isothermal conditions, with limiting conditions of concentration polarization, the limiting current density of oxygen reduction reaction is flow and temperature dependent. The charge transfer of the oxygen reduction reaction is a 4 electron process in the range of bulk temperature employed from 303 to 323 K. Under heat transfer conditions, the charge transfer is still 4 electron process up to 336 K interfacial temperature, above which the contribution of the 2 electron process appeared. Moreover, the limiting current density values of the oxygen reduction reaction in inhibited solutions is much lower than those under identical conditions in un-inhibited solutions. This confirms the inhibition of the cathodic reaction, i.e., the oxygen reduction reaction under isothermal and heat transfer conditions, due to SHMP.

Keywords: Corrosion, inhibitor, carbon steel, heat transfer, mass transfer, oxygen reduction reaction.

دراسة تثبيط تفاعل اختزال الاوكسجين على سطح القطب الاسطواني الدوار عند ظروف انتقال الحرارة والكتلة

الخلاصة

تم دراسة الحد من تفاعل اختزال الاوكسجين لقطب اسطواني دوار من الفولاذ الكربوني في ماء ملحي متعادل يحتوي على 600 جزء بالمليون من ايونات الكلوريد، باستخدام تركيز امثل لخليط مثبط التآكل من نترات الصوديوم (SN) و فوسفات الصوديوم السداسية (SHMP) بنسبة 100:500 = SHMP: SN تم التوصل اليها بطريقة فقدان الوزن.

اجريت تجارب الاستقطاب بالمجهاد الساكن في ظروف ثبات درجة الحرارة باستخدام محلول خال من خليط مثبط التآكل في درجات حرارة مختلفة للمحلول و معدلات جريان اضطرابي مختلفة لكل درجة حرارة. ثم اجريت تجارب مماثلة تحت ظروف انتقال حرارة مسيطر عليه من سطح المعدن الى المحلول. علاوة على ذلك، اجريت تجارب مماثلة لاعلاه بوجود مثبط التآكل موضوع البحث.

اظهرت النتائج، عند ثبوت درجة حرارة المحلول الخال من مثبط التآكل و تحت ظروف الاستقطاب التريزي، زيادة كثافة التيار المحدد لتفاعل اختزال الاوكسجين مع زيادة درجة حرارة المحلول و كذلك مع زيادة معدل الجريان. ان تفاعل اختزال الاوكسجين في مدى درجات حرارة بين 303 و 323 كلفن هو عملية استهلاك 4 الكترونات. اما تحت ظروف انتقال الحرارة، فان تفاعل اختزال الاوكسجين هو عملية استهلاك 4 الكترونات و لحد درجة حرارة سطح المعدن 336 كلفن اما في درجات حرارة اعلى من 336 كلفن لسطح المعدن فتظهر مشاركة عملية استهلاك 2 الكترون مع عملية استهلاك 4 الكترونات.

كما بينت النتائج ان اقيام كثافة التيار المحدد لتفاعل اختزال الاوكسجين بوجود مثبط التآكل هي اقل بكثير من نظيراتها في محلول خال من مثبط التآكل، و هذا يؤكد الحد من التفاعل الكاثودي، اي تفاعل اختزال الاوكسجين تحت ظروف ثبات درجة الحرارة و كذلك ظروف انتقال الحرارة، بسبب تأثير فوسفات الصوديوم السداسية.

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Nomenclatures

C_b	Concentration (bulk), mol/l
D	Diffusion coefficient, m ² /s
d	Cylinder diameter, m
F	Faraday's constant, (=96.500 kJ/V.mol)
i_{lim}	Limiting current density, A/m ²
k	Mass transfer coefficient, m/s
ppm	Parts per million
RCE	Rotating cylinder electrode
Re	Reynolds number, dimensionless
RPM	Rotations per minute
Sc	Schmidt number, dimensionless
Sh	Sherwood number, dimensionless
SHMP	Sodium hexametaphosphate
SN	Sodium nitrite
T_b	Temperature (bulk), K
T_i	Temperature (interface), K
V	Velocity of fluid, m/s
Z	Charge transfer
δ_d	Diffusion boundary layer thickness, m
ν	Kinematic viscosity, m ² /s

1. Introduction

In the absence of added oxidants the corrosion of virtually all metals in neutral and alkaline aqueous environments depends upon oxygen reduction reaction. It is now well established that oxygen reduction reaction may occur in a 2 or 4 electron process, the ultimate reaction product being hydrogen peroxide or water, respectively.

The published work treating the oxygen reduction reaction relates almost to experiments at temperatures about 298 K. In the last three decades, several workers reported their results at temperatures higher than the ambient and under heat transfer conditions [1-8].

Alwash [1] and Alwash et al. [2] reported that under isothermal conditions and constant angular velocity, the limiting rate of oxygen

mass transfer to a nickel rotating disc electrode increases with temperature. On a nickel electrode the limiting rate of charge transfer does not increase correspondingly since the oxygen reduction reaction moves progressively from a 4 to 2 electron process as the temperature is increased from 303 to 348 K. The application of heat transfer under these conditions leads to a stimulation of the limiting rate of oxygen mass transfer that cannot be explained solely in terms of an increased interfacial temperature; it appears that assistance from thermal convection, deriving from thermal eddies occurs. This latter finding was confirmed by Parshin et al. [3] and later by Tal [7] and Tal et al. [8].

Jaralla [4] reported that the charge transfer associated with oxygen reduction reaction on an iron rotating electrode in acid solution represents a 4 electron process at 303 K. This decreases as the temperature is increased due to contribution of the 2 electron process which is in agreement with the previous findings [1, 2]. However, Al-Mossawe [5], using a carbon steel rotating cylinder electrode in 1275 ppm Cl⁻ solution under controlled conditions of heat and mass transfers, reported that the charge transfer of oxygen reduction reaction is a 4 electron process up to 323 K (bulk or effective) above which the contribution of the 2 electron process appears.

Moreover, Tal [7] and Tal et al. [8] found that the charge transfer of the oxygen reduction reaction is a 4 electron process, under isothermal and controlled conditions of heat transfer in un-inhibited and inhibited Cl⁻ +SO₄²⁻ solutions, in the range of bulk temperatures used (303 to 323 K) and interfacial temperatures obtained (323 to 347 K). They added that the i_{lim} values of the oxygen

reduction reaction in inhibited solution are lower than those under identical conditions in un-inhibited solution.

It is well known that the essential feature of heat transfer is that there is a temperature gradient between the metal/electrolyte interface and the bulk electrolyte. One point of importance in the corrosion context is the direction of the temperature gradient at the metal/electrolyte interface, i.e., whether the solid is hotter or colder than the surrounding environment [9, 10]. It is also well known that many applications of inhibitors are for cooling water systems, where by definition, a temperature difference obviously exists between the metal/electrolyte interface and the electrolyte bulk giving rise to a heat flux flow. Such heat flux significantly affects the performance of an inhibitor; either by altering the rates of electrodes reactions or by encouraging the deposition of insoluble scales or adsorbed film on the metal surface [11].

The current approach to cooling water treatment is to use a multi-component inhibitors. Cooling water formulations containing mixtures of inhibitors usually offer better and increased protection to ferrous metals than similar concentrations of either of the individual components. Such mixtures are synergistic in their action [6-8, 12-20]. But, the synergistic effects between nitrites, molybdates and inorganic phosphates were not investigated until recently [6-8, 15-19].

Sodium nitrite (SN) is classified as oxidizing anodic inhibitor. It can be effectively used for corrosion control of carbon steel in water when the weight ratio of SN: total aggressive ions (Cl^- or $\text{Cl}^- + \text{SO}_4^{2-}$) equals one or less [6-8, 12, 21-23].

Sodium hexametaphosphate (SHMP) is classified as a cathodic inhibitor. Divalent metal ions and

Ca^{2+} in particular are needed with the polyphosphate for effective inhibition of steel. A protective film develops through the formation of a positively charged colloidal complex that migrates to the cathode, forming an amorphous protective barrier film [14]. Moreover, SHMP is considered the most effective for the prevention of scale formation and the precipitation of carbonate of calcium and iron that interferes with the heat transfer [12]. The concentration of SHMP needed to prevent the corrosion of low carbon steel depends on the composition of the water and on its rate of movement. Usually, from 0.5 to 100 ppm of SHMP are used for the treatment of fresh water and water containing chlorides [12].

The purpose of the present work is to investigate the oxygen reduction reaction on iron rotating cylinder electrode (RCE) in un-inhibited and inhibited aerated neutral 600 ppm Cl^- solution [24] under isothermal and controlled heat and mass transfer conditions. A turbulent flow was employed, which is of industrial importance. In the inhibited solution, a formulated inhibitor blend was used, i. e., sodium nitrite (SN): sodium hexametaphosphate (SHMP) = 500:100 ppm [6].

2. Experimental Work

The apparatus used in this work consisted of a supporting framework, a RCE assembly, a polarization cell, a constant temperature bath and a potentiostat. The detailed designs of the apparatus and the experimental work were described elsewhere [6, 25]. In Brief, the inhibition of the oxygen reduction reaction on a carbon steel RCE in naturally aerated 600 ppm Cl^- solution was studied using an optimum inhibitor blend, i.e., SN:SHMP=500:100. A potentiostatic technique was applied at different bulk temperatures, i.e., 303, 313 and 323 K and various turbulent flow rates corresponding to 50, 200, 300 and

400 rpm using un-inhibited and inhibited solutions under isothermal and controlled conditions of heat transfer.

The carbon steel RCE composition is given below in wt. % [26].

C	Mn	Si	Ni	S	Cr
0.16	0.36	0.04	0.02	0.01	0.00
11	70	30	25	62	33

The design of the RCE assembly, in addition to satisfying the fundamental requirements of the hydrodynamic theory, also required the provision of a heat transfer system with a means of measuring temperature at various distances from the center of the electrode shaft in order to determine the heat flux and to calculate the electrode interfacial temperature.

3. Experimental Results and Discussion

Without exception, on a typical cathodic polarization curve for the iron electrode in aerated Cl⁻ solutions, a definite limiting current due to oxygen reduction was apparent. This confirms that an iron electrode in neutral solution can support a mass transfer controlled oxygen reduction reaction as demonstrated by Fontana and Greene [27]. The body of the results obtained in the present study, are most readily discussed by reference to Figures 1 to 31 for uninhibited experiments and Figures 4 to 62 for inhibited ones. These figures show that *i_{lim}* is related to *V*^{0.7} at a particular test solution bulk temperature.

Un-inhibited solution

Figures 1 to 3 show that at each bulk temperature of uninhibited experiments (under isothermal and heat transfer conditions), there is almost a linear relationship between *i_{lim}* and *V*^{0.7} which, when extrapolated, passes through the origin. Thus, the experimental data shown in Figures 1 to 3 are in qualitative agreement with Eisenberg et al. [28] theory for mass

transfer to a rotating cylinder electrode and represented by the expression:

$$i_{lim} = 0.079 Z F C_b V^{0.7} d^{-0.3} u^{-0.344} D^{0.644} \dots(1)$$

Or:

$$Sh = 0.079 Re^{0.7} Sc^{0.356} \dots(2)$$

This can be attributed to the increase in the mass transfer coefficient (*k*) which will lead to the increase of the oxygen flux arriving at the metal surface and the decrease in the resistance that hinders the transfer of oxygen especially the diffusion boundary layer thickness (*δ_d*) [1, 4, 7].

Previously, it was noted that oxygen reduction is found to proceed by a 2 or 4 electron process, i.e., *Z* in the Eisenberg et al. [28] equation is 2 or 4. Thus, the theoretical values of the limiting current density for both the two electron (2e) and four electron (4e) in the test solutions are also shown in Figures 1 to 6 (solid lines)³

Figures 1 to 3 show that, under isothermal conditions, between 303 and 323 K, oxygen is reduced quantitatively to water, i.e., 4e process. This finding is in agreement with other workers results [5, 7, 8]. However, under heat transfer conditions and at 303 and 313 K bulk temperatures, the oxygen reduction is still controlled by a 4e process despite the high interfacial temperatures encountered, i.e., greater than 323 K (see Table 2). Moreover, at 323 K bulk temperature and up to interfacial temperature equals 335.6 K, the charge transfer of the oxygen reduction reaction is again a 4e process, above which the contribution of the 2e process appeared (see Figure 3 and Table 2). To

³ The theoretical values of the limiting current density of the oxygen reduction reaction at any value of RPM can be calculated from the kinematic viscosity [29], the diffusion coefficient [1] and concentration of oxygen [30], using the Eisenberg et al [28] equation, see Table 5.

¹ See Tables 1 and 2

² See Tables 3 and 4

summarize, under heat transfer conditions, the oxygen reduction is controlled by a 4e process in the range of interfacial temperatures 323 to 336 K, which is rather close to that reported by Tal [7] and Tal et al. [8].

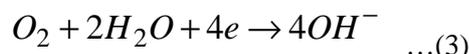
Moreover, Figures 1 to 3 show that the experimental data deviate above the 4e theoretical line. This may be due to the surface roughness which causes the data to lie above the correlation for a smooth cylinder. The surface roughness enhances the mass transfer of oxygen to the surface of the electrode and shifts the correlation to higher Sherwood number than that for smooth surface [31, 32].

The limiting rate of mass transfer of oxygen to a rotating cylinder electrode, i.e. i_{lim} , increases with an increase in bulk temperature, under isothermal conditions, despite the marked fall in oxygen concentration (see Table 1). This is due to an increase in the oxygen diffusion coefficient (D) and a lowering of the kinematic viscosity (ν) [1, 6]. Similarly, in the case of heat transfer and because of the interfacial temperature (T_i), where $T_i > T_b$, oxygen mass transfer is stimulated due to the increase in D of oxygen and the decrease in ν of the solution that is adjacent to the metal surface. Furthermore, the heat transfer data, especially at bulk temperature 303 K, fall above the isothermal data (see Figure 1). The presence of heat transfer, from the metal surface to the solution, leads to form thermal convection derived from thermal eddies adjacent to the metal surface. Hence, under these conditions, stimulation of the limiting current density (i_{lim}) occurs, i.e., enhancement of the oxygen transfer to the surface, as stated by Alwash [1] and confirmed by Parshin et al [3] and later by Tal [7] and Tal et al. [8].

Inhibited solution

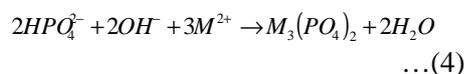
Figures 4 to 6 show that at each bulk temperature, for inhibited experiments under isothermal and heat transfer conditions, there is, firstly, no linear relationship between i_{lim} and $\nu^{0.7}$. Secondly, the experimental data fall below the 2e theoretical line, and thirdly,

they have much lower values than those under identical conditions in un-inhibited solutions. The results confirm the inhibition of the cathodic reaction, i.e., the oxygen reduction reaction which is represented by the following equation in neutral solution:



The inhibitor blend contains SN and SHMP. The first is a well known anodic inhibitor, while the second is a cathodic inhibitor. Therefore, very low values of i_{lim} of the oxygen reduction reaction were obtained (see Figures 4 to 6) indicating the good inhibitive characteristics of SHMP.

The inhibitive action of SHMP takes place in two steps. The first being stimulation of the anodic dissolution process [33]. The free Fe^{2+} ions stimulate the hydrolysis of SHMP mainly to HPO_4^{2-} and then to PO_4^{3-} as the second step, i.e., the formation of the protective layer, as follows:



Where M is a divalent metal ion, Ca^{2+} in particular, Fe^{2+} and Zn^{2+} [34]. This reaction proceeds at the metal surface. The source of hydroxyl ions (OH^-) is reaction 3.

It is generally believed that the flux of inhibitor to the metal surface is considerably enhanced with increasing flow rate [9]. Furthermore, as the flow rate is increased, the inhibitor concentration profiles are modified since the mass transfer boundary layer is thinned causing an increase of the interfacial concentration of the inhibitor. Consequently, the retarding effect of the inhibitor on the cathodic reaction is considerably enhanced [9]. The results in Tables 1 and 3 are in agreement with Ross works [9]. On the other hand, an increased flow rate causes also an increase of oxygen flux to the metal surface and a consequent enhancement in the rate of oxygen reduction reaction (equation 3), which provides OH^- to

reaction 4. Hence, it is most certain here that the retarding effect of the inhibitor on the cathodic process is much greater than the enhancement in the rate of the oxygen reduction reaction.

To summarize, SHMP decelerates the cathodic process due to protective film formation and this effect is increased as the flow rate increases.

Figure 7 shows a plot of i_{lim} , at constant flow rate, versus the bulk temperature. It shows clearly that i_{lim} increases with temperature up to 313 K, thereafter, it falls to a low value at 323 K. This is due to the increased hydrolysis of SHMP with temperature. At 323 K, the hydrolysis of SHMP to PO_4^{3-} is rapid and forming a protective film rapidly on steel and consequently blocking the cathodic sites [6]. Therefore, the values of i_{lim} at 323 K are lower than those at 303 and 313 K (see Table 3). The effect of flow rate at 323 K is very small (see Figure 6). The results indicate that a better inhibition of the oxygen reduction reaction was obtained at 323 K rather than at lower temperatures.

4. Conclusions

Under isothermal and heat transfer conditions and in un-inhibited solutions, the i_{lim} of the oxygen reduction reaction on carbon steel RCE is controlled by a 4e process in the range of bulk temperatures employed between 303 and 323 K and interfacial temperature obtained from 323 to 336 K. Above 336 K, the contribution of the 2e process appeared. The i_{lim} , under isothermal conditions and un-inhibited solutions, is flow and temperature dependent.

Moreover, the i_{lim} values of the oxygen reduction reaction in inhibited solutions are much lower than those under identical conditions in un-inhibited solutions. This confirms the inhibition of the cathodic reaction, i.e., the oxygen reduction reaction under isothermal and heat transfer conditions, due to SHMP.

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Table (1) The limiting current density of the oxygen reduction reaction on iron surface in the salt solution

RPM	$V^{0.7}, (m/s)^{0.7}$	$i_{lim}, A/m^2$ at bulk temperature		
		303 K	313 K	323 K
0	0	0.146	0.341	0.487
50	0.3	0.97	1.8	2.38
200	0.79	4.87	5.23	5.36
300	1.05	5.40	6.82	7.30
400	1.28	8.10	8.3	8.53

Table (2) The limiting current density of the oxygen reduction reaction on iron surface in the salt solution under 20 kW/m² heat transfer

RPM	$V^{0.7}, (m/s)^{0.7}$	$i_{lim}, A/m^2$ (obtained temperature K) at bulk temperature		
		303 K	313 K	323 K
50	0.3	1.88 (329.0)	2.20 (333.0)	1.30 (341.2)
200	0.79	4.97 (327.3)	5.10 (329.0)	3.30 (339.8)
300	1.05	6.60 (323.3)	6.70 (325.7)	5.80 (335.6)
400	1.28	8.10 (322.2)	8.00 (324.8)	7.40 (333.8)

Table(3) The limiting current density of the oxygen reduction reaction on iron surface in the inhibited solution

RPM	$V^{0.7}, (m/s)^{0.7}$	$i_{lim}, A/m^2$ at bulk temperature		
		303 K	313 K	323 K
0	0	0.293	0.832	0.35
50	0.3	0.66	0.98	0.43
200	0.79	0.88	1.43	0.39
300	1.05	1.04	1.828	0.47
400	1.28	1.597	2.04	0.47

Table (4) The limiting current density of the oxygen reduction reaction on iron surface in the inhibited solution under 20 kW/m² heat transfer

RPM	$V^{0.7}, (m/s)^{0.7}$	$i_{lim}, A/m^2$ (obtained temperature K) at bulk temperature		
		303 K	313 K	323 K
50	0.3	0.414 (329.0)	0.58 (333.0)	0.687 (341.2)
200	0.79	0.56 (327.3)	0.67 (329.0)	0.76 (339.8)
300	1.05	0.82 (323.3)	0.86 (325.7)	0.92 (335.6)
400	1.28	1.082 (322.2)	1.05 (324.8)	1.19 (333.8)

Table (5) The theoretical values of the limiting current density of the oxygen reduction reaction for 4e process under isothermal conditions

RPM	$V^{0.7}, (m/s)^{0.7}$	$i_{lim}, A/m^2$ at bulk temperature		
		303 K	313 K	323 K
50	0.3	1.34	1.62	1.66
200	0.79	3.55	4.27	4.37
300	1.05	4.72	5.68	5.81
400	1.28	5.80	6.95	7.11

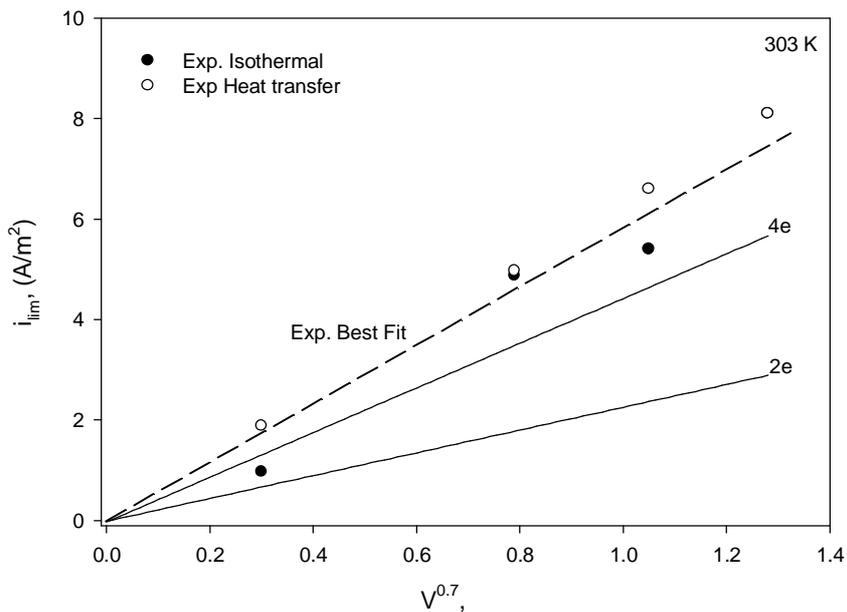


Figure (1) Limiting current density of oxygen reduction reaction on iron surface in the salt solution and 303 K bulk temperature

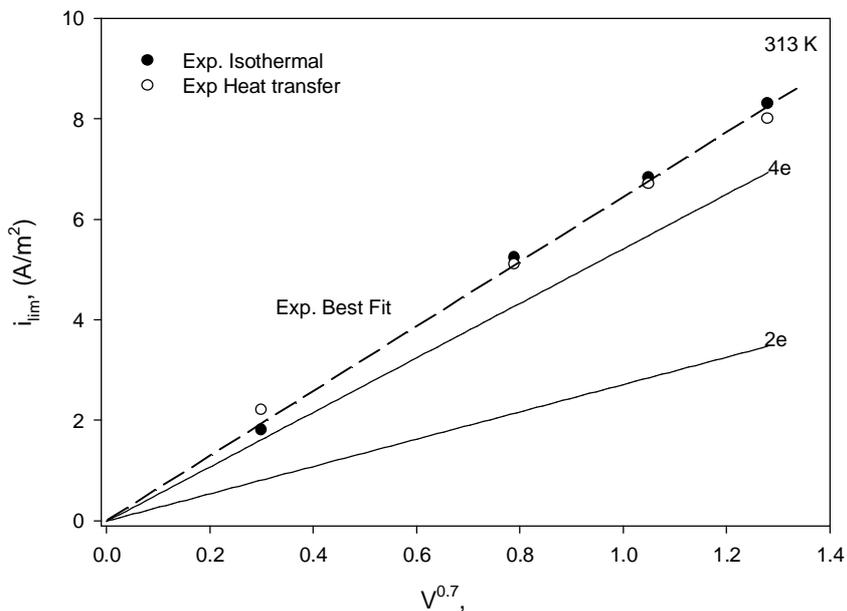


Figure (2) Limiting current density of oxygen reduction reaction on iron surface in the salt solution and 313 K bulk temperature

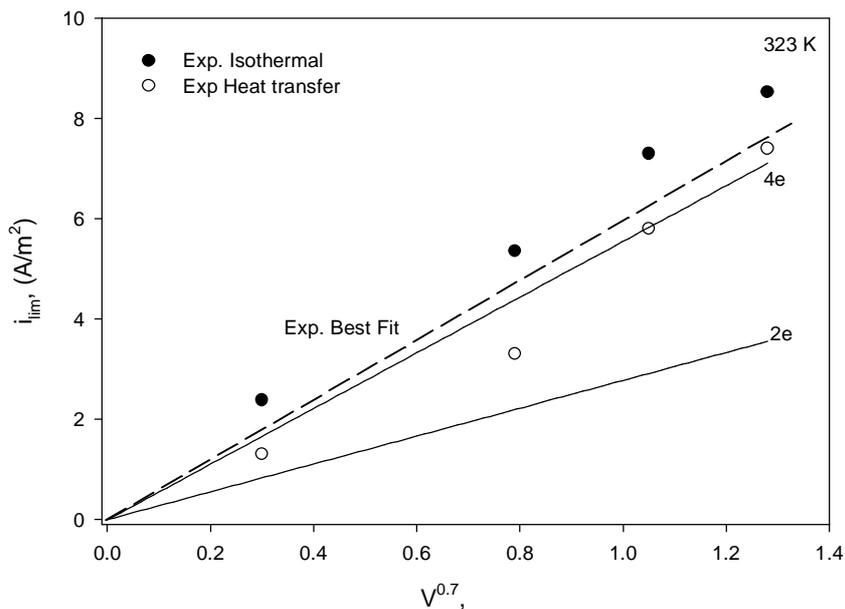


Figure (3) Limiting current density of oxygen reduction reaction on iron surface in the salt solution and 323 K bulk temperature

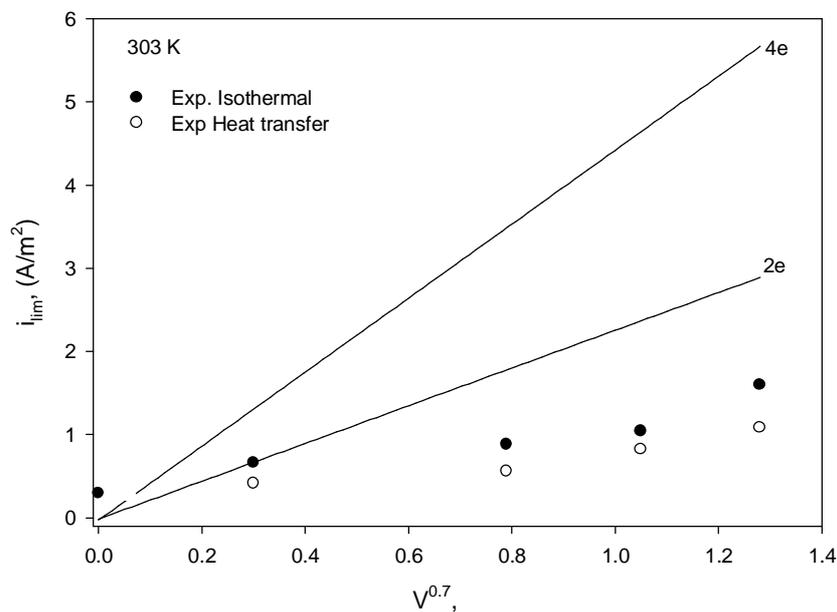


Figure (4) Limiting current density of oxygen reduction reaction on iron surface in the inhibited solution and 303 K bulk temperature

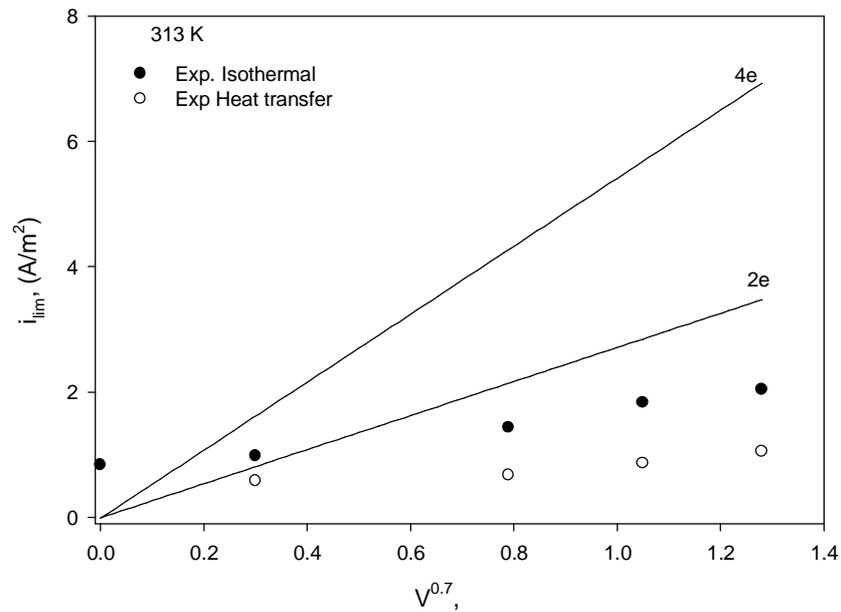


Figure (5) Limiting current density of oxygen reduction reaction on iron surface in the inhibited solution and 313 K bulk temperature

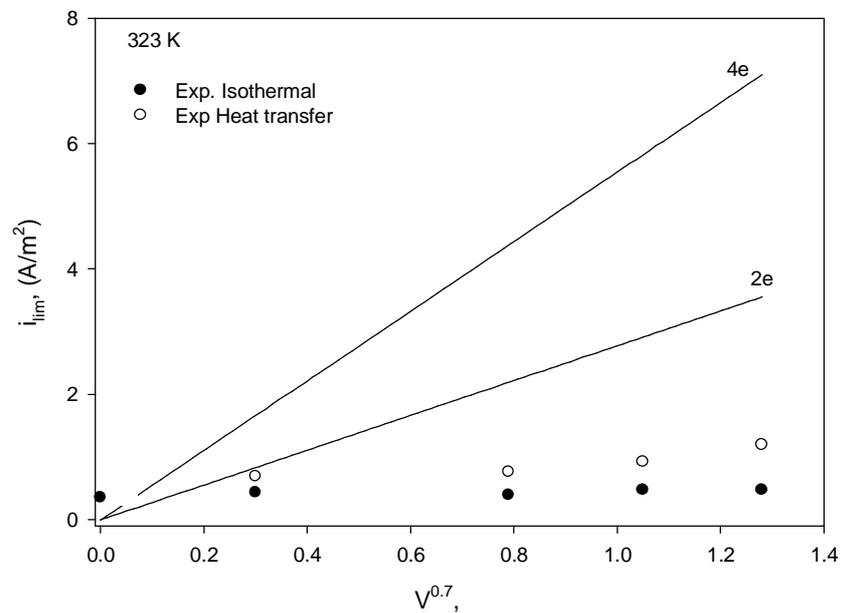


Figure (6) Limiting current density of oxygen reduction reaction on iron surface in the inhibited solution and 323 K bulk temperature

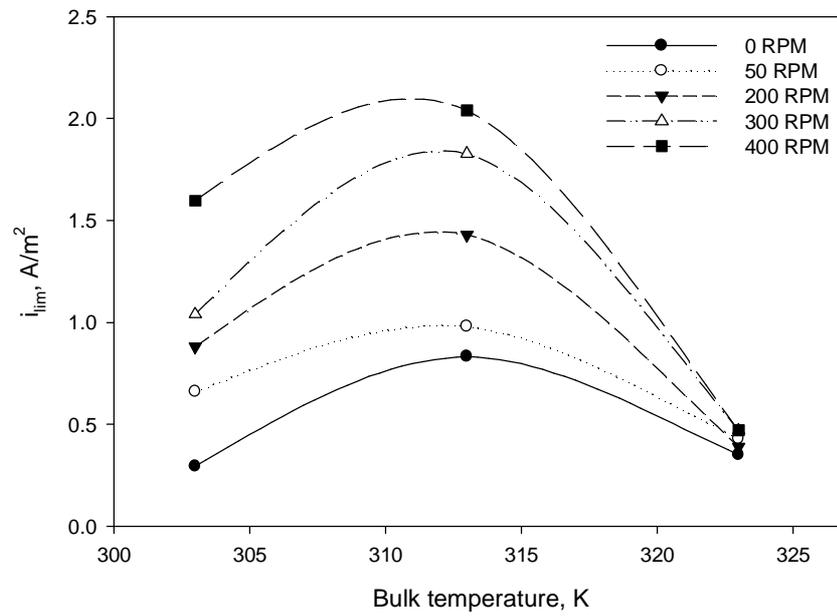


Figure (7) Limiting current density of oxygen reduction reaction versus bulk temperature of inhibited solution under isothermal conditions