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Thermodynamics, Mechanism and Rate of Corrosion of Copper in Presence of Phosphoric Acid- N-Propanol Mixtures

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Abstract: The corrosion behavior of copper in the presence of phosphoric acid-n-propanol has been studied by using different techniques, including limiting current density measurements and rotating disk electrode method. The influence of organic solvent addition on the rate of corrosion of copper was achieved using the rotating disk electrode technique. Dimensionless correlation between different parameters such as, temperature, speed of rotation of copper disk and viscosity measurements are obtained. The corrosion rate is affected by electrode height, mole fraction of alcohol and phosphoric acid concentration. Physical properties of solution and Thermodynamic parameters are calculated. The data can be correlated by the following equations: Sh = 1.835 $(Sc)^{0.33}$ $(Re)^{0.36}$ for n-propanol A laminar flow mechanism is known from exponent.

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1. Introduction

The phenomenon of electropolishing since it was discovered by Jaquet (1,2-11). In the last two decades the electropolishing process seems to have been rediscovered mainly due to the demand for super clean, homogenous, corrosion resistant, biocompatible surfaces, which do not interfere in processes utilized by semiconductor, biotechnology and body implants industries (1,2).

A great deal of work has been directed to study the optimum polishing conditions for different metals and alloys (12).

Previos studies (13-16) have revealed that electropolishing is a diffusion controlled reaction which takes place at the limiting current; the limiting current can be approached by increasing electric potential or decreasing the rate of mass transfer to the electrode. The value of the limiting current which determines the polishing rate depends on the rate of mass transfer of Cu^{2+} from the diffusion layer to the bulk of the solution. Mass transfer is used for physical processes that involve diffusive and convective transport of chemical species within physical systems. Many factors are affecting the rate of mass transfer such as the relative movement of the anode and the electrolyte, physical properties of the electrolyte, temperature and geometry of the anode. The present work is concerned with the study of the factors affecting the limiting current of the electropolishing of vertical plates in unstirred (H_3PO_4) solution (17-20). Previous investigations overlooked the stirring effect of the cathodic hydrogen which arrives at the anode during electropolishing and enhances the rate of mass transfer and the limiting current; the degree of enhancement depends on the geometry of the cell, electrode separation and physical properties of the solution (21,22).

The aim of the present work is generalization the extent to which n-propanol affects the rate of corrosion of copper., obtaining a dimensionless correlation between different reaction conditions such as temperature, speed of rotation of rotating copper disk, and the physical properties of solutions such as density, viscosity and diffusion coefficient.

Experimental

2. Materials and Methods

n-propanol (E Merk) and phosphoric acid (BDH) were used as received.

Viscosity measurements were performed with an Ubbelohde viscometer, it was first clean with dilute chromic acid then rinsed with distilled water and finally with the solvent. The viscometer with the solution was kept in a water thermostat for 30 minutes before measuring the flow time. The temperature was controlled within 0.01°C.

Apparatus and procedure

Figure (1) shows the cell and electrical circuit used in the present work. The two electrodes were copper sheets of 10 cm height and 5 cm width. The distance between the two electrodes was 5 cm; stirring effect is prevented by using a porous PVC diaphragm.

The electrical circuit consisted of: 1) 6 volt d.c. power supply, 2) variable resistance,3) multirange ammeter connected in a series with the cell,4) high impedance voltammeter was connected in parallel with the cell to measure its potential. Four concentrations of H_3PO_4 were used: 6, 8, 10, 12 M; all were prepared from AR grade H_3PO_4.

The steady state anode potential was measured against a reference electrode consisted of copper wire immersed in a cup of luggin tube filled with phosphoric acid-alcohol solution similar to that in the cell, the tip of the luggin tube was placed 0.5-1 mm apart from anode wall.

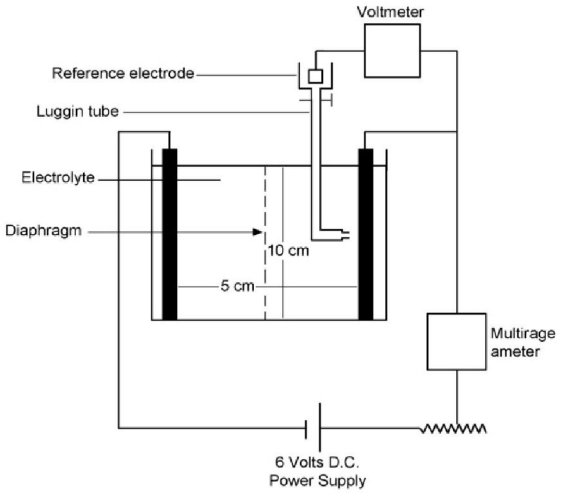

Fig (1) The electrolytic cell and Electrical circuit

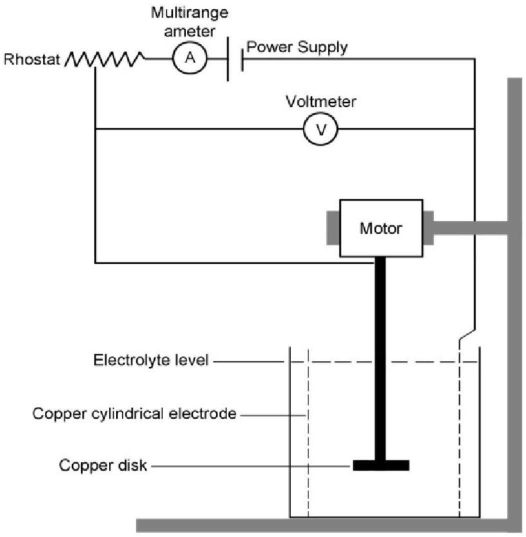

Fig (2) Cell and Electrical circuit

Three electrodes setup the potentiostate controls the voltage difference between a working electrode and a reference electrode by injecting a current through a counter electrode. The limiting current were plotted by increasing the applied current stepwise and measuring the corresponding steady state potential, the steady state potential is obtained in two minutes. The back of the anode was insulated with polystyrene lacquer and the active surface was polished with a series of emery papers with different grades, degreased with trichloroethylene, washed thoroughly with distilled water and dried with ethanol. The temperature was regulated by placing the cell in a thermostat. Viscosity, density and diffusion coefficient were measured using general techniques.

Figure (2) is a block diagram of apparatus which permits the rotation of disk electrode at accurately controlled angular velocities. The shaft is driven by variable speed motor. The frequency of rotation, recorded as revolution per minute, was counted by an optical tachometer.

The anode consists of copper metal disk of 2cm diameter and 2mm thickness. The sides and back of the disk, as well as the derive shaft were insulated by epoxy resin. The cathode is made of a cylindrical copper electrode of 12cm diameter; it also acts as a reference electrode by changing its surface area compared to that of the anode (23).

Results and discussion

Figure (3) shows a set of typical current potential curves obtained at different H_3PO_4 alcohol concentrations. It is obvious that the limiting current decreases with increasing H_3PO_4 concentration within the range studied (6-12M), this is in agreement with the finding of other authors who worked within the same range of concentration using other anode geometries (17-20). The effect of H_3PO_4 concentration on the value of the limiting current can be explained on the basis of the mass transfer equation (1) (24):

$$I_L = \frac{ZFD}{\delta} Cu^{++} \qquad (1)$$

Increasing H_3PO_4 concentration Fig (3) and Fig (4) decreases the saturation solubility of copper phosphate (11) (Cu^{2+}) with a consequent decrease in the limiting current according to equation (1). Also, increasing H_3PO_4 concentration increases the viscosity of the solution, these results in a decrease in the diffusivity of copper ions (D) and an increase in the diffusion layer thickness (δ) with a consequent decrease in the limiting current value according to equation (1).

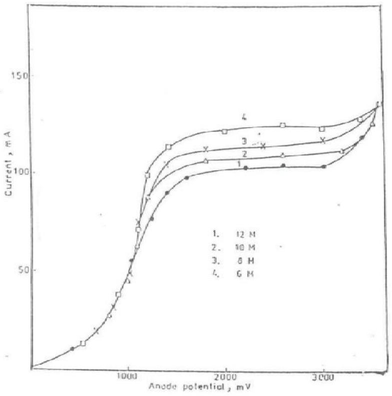

Fig. (3) Typical polarization curves obtained using n-propanol-water mixtures at different concentration of phosphoric acid x = 0.3106

1. Effect of electrolyte concentration on the polishing current

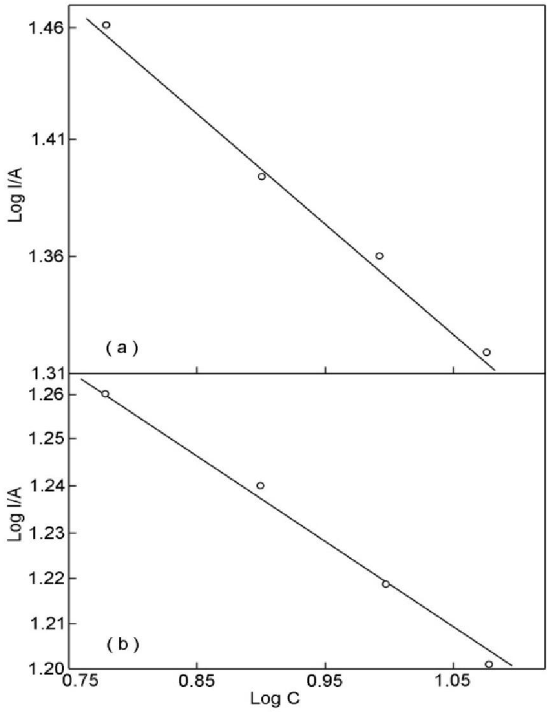

Fig. (4) Effect of phosphoric acid concentration on the limiting current density in presence of n-propanol at 25˚C.

Fig. (4) shows the dependence of the polishing current on the bulk concentration of orthophosphoric acid. The limiting current density decreases with increase phosphoric acid concentration but the values of limiting current in acid-(alcohol-water) mixtures is less than in water-acid mixtures. From the practical point of view, we can recommend on the basis of above result that it is preferable to use relatively high organic solvent percentage to inhibit polishing or dissolution of metal at the same phosphoric acid concentration.

(a) x = 0.3103 (b) x = 1

2. Effect of electrode height on polishing current

Fig. (5) shows that the limiting current density decreases with the increase in height. In electropolishing and generally for anodic dissolution of metals, the direction of flow of the hydrodynamic boundary layer and the diffusion layer increase in the downward direction. Accordingly, the local limiting current density increases in the upward direction of the anode. This explains why polishing is attained at the upper parts of the electrode before the lower part at the limiting current region. This was confirmed by the visual observation during electropolishing. The average limiting current density decreases with the increase in the height according to the equation (2):

$$\mathbf{I}_L = \frac{\mathbf{C}}{(\mathbf{H})^{\mathbf{0.31}}} \qquad (2)$$

Where C is constant.

The above results were given in the presence of alcohol- phosphoric acid mixture as well as in the absence of alcohol.

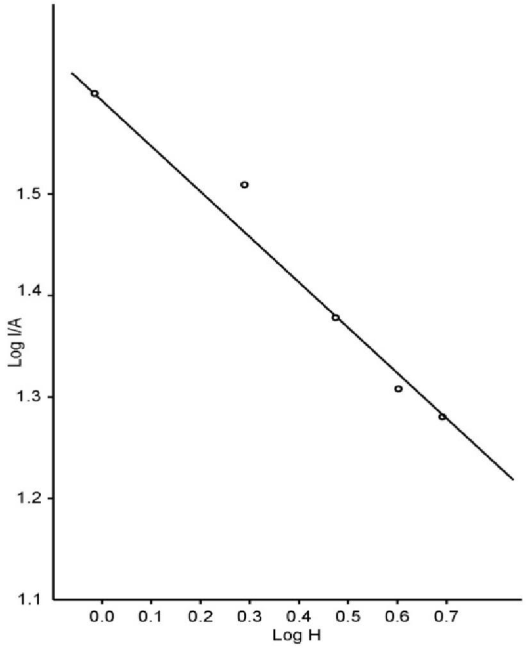

Fig. (5) Effect of electrode height on the limiting current density in 10M H_3PO_4 in presence of n-propanol at 25˚C.

X = 0.1165

3. Structural effects

In water-organic solvent mixtures, the variation of physicochemical properties of medium with the percentage composition Table (1) has an important role in controlling electropolishing. It has been found that corrosion is inhibited by the organic alcohol and percentage inhibition has been found to increase with increasing concentration of organic solvents Table (1). These results were interpreted on the basis of the fact that these types of corrosion are controlled by diffusion of Cu^{2+} from metal surface to bulk solution. The increase in the viscosity of the medium with increase in the percentage composition leads to a marked decrease in diffusion coefficient.

Table (1) The relation between percentage inhibitor of corrosion of copper in 10M phosphoric acid in the presence of different composition of n-propanol at 25°C.

Dielectric constant	**Composition V/V**	**I_L (mA Cm^3)**	**% inhibition**
80.37	0	250	--
66.54	20	200	20
51.68	40	175	30
37.51	60	155	38
26.83	80	130	48
20.81	100	110	56

In this study, the aspect of the dependence of the rate of corrosion on the concept of water interaction is considered. It is found that addition of alcohol to H_3PO_4 has a marked effect on reducing the acidity of the medium. This has been attributed to the change in the state of solvation of Cu^{2+} and to increase the proton affinity of water (25, 26). On addition of alcohol to water, the acidity decreases strongly due to breakdown of the open tetrahedral structure of water (27). This concept explains the decrease in the rate of electropolishing with the addition of alcohol to the purely aqueous medium, especially in alcohol which is more basic, as indicated in Table (2). Furthermore addition of alcohol would not influence the tetrahedral structure of water to the same extent. It has been reported that (28-30) the dielectric constants of alcohol-water mixtures decrease with increase in alcohol percentage. Table (3) shows that the limiting current decrease with decreasing dielectric constant of mixtures.

According to electrostatic theory, the association of an electrolyte in a solvent involve both steric and columbic effects. On the basis of this approach the structure modification of the alcoholic polymers generated by added solvents should result in a variable influence of alcohol molecules on ion-pair association of Cu^{2+}.

When a higher alcohol is chosen as the solvent system, the pattern of ionic association of hydroxyl solvent may be investigated without such complication as three dimensional structural effects (31-33).

4. Effect of temperature

The activation energy of the process is an important parameter for determining the rate controlling step (26). Table (3) give the values of E. It is obvious that, the values of $E < 40$ K Joule/mole. This indicates that electropolishing reaction is controlled by diffusion process.

Table (2) Limiting current – temperature for n-propanol mixture at different mole fraction of n-propanol.

Mole fraction	**Limiting current**				
T° C	**20**	**25**	**30**	**35**	**40**
0.0475	180	200	220	245	270
0.1665	160	175	210	230	250
0.3106	140	155	175	195	215
0.5456	115	130	150	180	200
1.000	95	110	130	150	170

5. Thermodynamic treatment of the results:

The values for enthalpy of activation, ΔH*, entropy of activation ΔS*, and free energy of activation ΔG*, can be obtained by using equations:

$$\Delta H^* = E - RT \quad (3)$$

$$\frac{\Delta S^*}{R} = Ln\,A - Ln\frac{\alpha Te}{h} \quad (4)$$

$$\Delta G^* = \Delta H^* - T\Delta S^* \quad (5)$$

Where α is the Boltzman constant, e is 2.7183 and h is Plank's constant.

Thermodynamic functions of electropolishing of copper in n-propanol gives evidence of structural change occurring in the solution as the type of solvents are changed. The effect can arise from the solvent properties and (or) from the solvation properties of the ion in different solvents.

The calculated values of ΔG*, ΔH* at different temperatures are shown in Table (3). Figure (6) shows the variations of ΔG*, ΔH* and ΔS* with the mole fraction of the organic solvents. The gradual increases of both ΔG* and ΔH* give a good indication of preferential solvation of metal surface in presence of aprotic solvents which is a criterion of specific solvation. Also the weak dependence of ΔG* on the composition of the organic solvent can be attributed largely to the general linear compensation between ΔH* and ΔS* for the given temperature.

Table (3) shows that the free energy change increases positively with increasing the dielectric constant of the solvent. This shows that as the dielectric constant decrease more work is required to keep the ions apart and, therefore, the dissolution of Cu is decreased as the dielectric constant decreases. There is less spontaneity and less dissolution at lower dielectric constant. Table (3) and Table (4) show that for any solvent, as the temperature increases the dielectric constant decreases and the forces among the ions greater, but the rate of mass transfer increases. This is attributed to the fact that as the temperature increases, the viscosity of solvent decreases Table (5). So, solvation decreases and velocity of Cu^{2+} increases, this leads to increases in the rate of mass transfer.

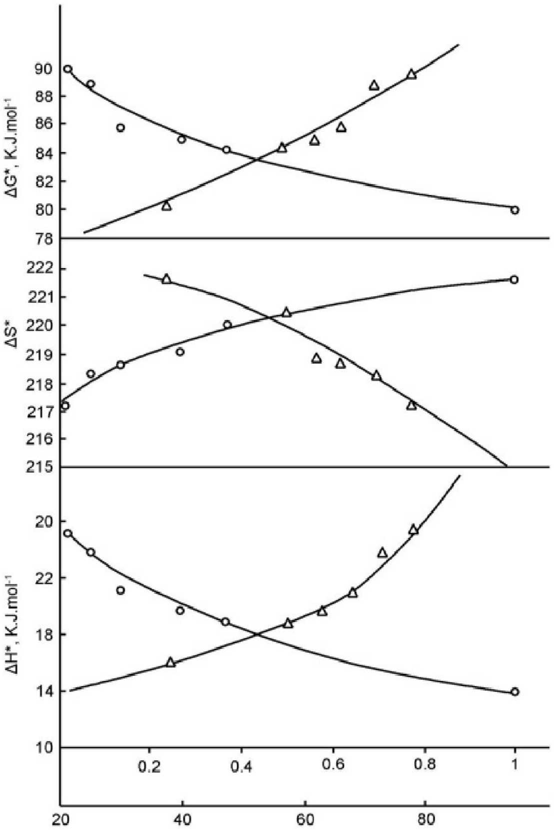

Fig (6) Variation of Thermodynamic parameters ΔH*,ΔS*,ΔG* with D in water-n propanol mixtures

Table (3): Thermodynamic parameters for n-propanol water-phosphoric acid mixture at 25°C.

Mole fraction (X)	**E** **KJ. mol^{-1}**	**ΔH*** **KJ. mol^{-1}**	**ΔG*** **KJ. mol^{-1}**	**-ΔS*** **J. $K^{-1}mol^{-1}$**
0.0475	15.48±0.21	13.00±0.21	59.9±0.41	157±0.8
0.1665	17.83±1.28	15.35±1.38	60.14±2.75	150.25±4.5
0.3106	16.6±0.30	14.13±0.30	60.5±0.584	155.5±9.7
0.5456	21.86±1.01	19.38±1.01	60.91±2.01	139±3.43
1	22.517±0.45	20.04±0.45	61.35±0.91	138±1.5

Table (4) General correlation of free convection mass transfer in presence of n-propanol

Volume of alcohol (%)	Rpm	I_L (mA cm^{-2})	K x 10^{-3} (cm s^{-1})	γ (cm^2 s^{-1})	D x 10^6	Sh	Sc	Re
7.5	360	280	1.8420	8.52117x10^{-3}	3.2367	1138.196	2632.672	8843.856
	502	295	1.9407		2.7277	1422.957	3123.9396	12331.64
	607	310	2.0394		2.5483	1600.596	3343.864	14911.098
	680	330	2.1709		2.5703	1689.219	3315.243	16705.053
	790	370	2.4341		2.7269	1785.251	3124.856	19407.358
23	360	270	1.8270	8.6526x10^{-3}	3.2096	1138.450	2695.85	8709.521
	502	285	1.9285		2.7125	1421.935	3189.899	12144.327
	607	300	2.0300		2.5405	1958.111	3405.865	14684.603
	680	315	2.1315		2.5102	1696.271	3446.978	16451.309
	790	360	2.4360		2.7407	1777.65	3157.077	19112.567
38	360	220	1.5723	8.73459x10^{-3}	2.5683	1224.3897	3400.923	8627.766
	502	230	1.6437		2.1394	1536.599	4082.729	12030.33
	607	245	1.7509		2.0398	1716.737	7282.082	14546.762
	680	280	2.0011		2.2887	1748.678	3816.398	16296.884
	790	300	2.144		2.2683	1890.402	3850.721	18993.161
54	360	148	1.0761	8.8711x10^{-3}	1.4599	1474.211	6076.512	8495.001
	502	160	1.1634		1.2788	1819.518	6937.050	11845.205
	607	180	1.3088		1.3233	1978.085	6703.771	14322.914
	680	300	1.4542		1.4234	2043.277	6232.331	16046.105
	790	230	1.6723		1.5686	2132.220	5655.524	18641.814
70	360	115	0.8994	8.97486x10^{-3}	1.1187	1607.938	8022.580	8396.788
	502	140	1.0949		1.1711	1869.866	7663.615	11708.261
	607	160	1.2513		1.2408	2016.925	7233.124	14157.324
	680	180	1.4078		1.3597	2070.751	6600.618	15860.593
	790	200	1.5642		1.4231	2198.299	6306.559	18426.293
100	360	100	0.8868	8.08544x10^{-3}	1.0986	1614.345	8270.016	8294.59
	502	120	1.0641		1.1254	1891.061	8073.076	11565.758
	607	135	1.1971		1.1646	2055.813	7801.339	13985.013
	680	150	1.3301		1.2526	2123.743	7253.265	15667.552
	790	168	1.4897		1.3268	2245.553	6847.633	18202.024

6. The isokinetic relationship

Variation in the rate within a reaction series may be caused by changes in either, or both, the enthalpy or entropy of activation. The correlation of ΔH* with ΔS* is a linear relationship which may be stated algebraically:

$$\Delta H^* = \beta\, \Delta S^* + \text{constant} \qquad (6)$$

or

$$\delta \Delta H^* = \beta\, \Delta S^*\, \delta \qquad (7)$$

The operator concerns the difference between any two reactions in the series. Substituting from (7) into the familiar relationship

$$\delta \Delta H^* = \delta \Delta G^* + T\, \delta\, \Delta S^* \qquad (8)$$

We obtain

$$\beta\, \Delta S^*\, \delta = \delta \Delta G^* + T\, \delta\, \Delta S^* \qquad (9)$$

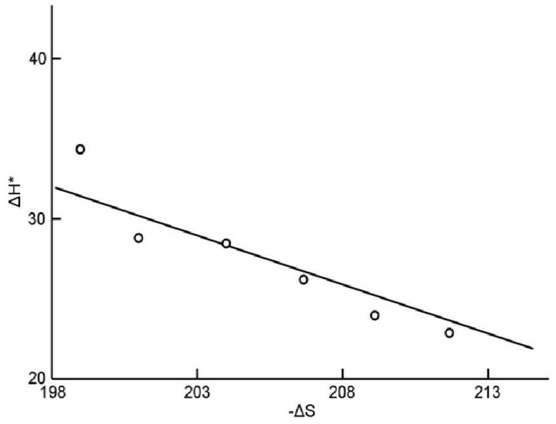

Fig. (7) Variation of ΔH* with ΔS* in water-n propanol mixtures.

It follows that when **$\delta\Delta G^*$** equals zero, β equals T. in Fig (6), other words, the slope in a linear plot of ΔH^* versus ΔS^* is the temperature at which all the reactions that conform to the line occur at the same rate. β is, therefore, known as the isokinetic temperature. Figures (7) and Fig (8) shows the plot of ΔH^* versus ΔS^* for different solvents and compositions; the isokinetic temperature β were estimated as 400, 615 for n-propanol. This value is much higher than that of the experimental temperature (298 K) indicating that the rate of the reaction is enthalpy controlled (32).

7. Data correlation

To obtain an overall mass transfer correlation under the present conditions, where a rotating disk is used, the method of dimensional analysis was used. To identify the variables which affect the rate of mass transfer in the corrosion, the mechanism of forced convection mass transfer should be recalled first. Forced convection takes place as a result of disk rotation. The thickness of this hydrodynamic boundary layer determines the thickness of the diffusion layer across which diffusion of Cu^{2+} from the interface to bulk takes place. The thickness of the hydrodynamic boundary layer at the rotating disk and the diffusion layer are determined by the physical properties of the solution, the geometry of the system (disk diameter) and disk rotation speed. This picture leads to the equation:

$$K = f(\rho, \eta, D, V, d) \qquad (10)$$

where

K = mass transfer coefficient, sec^{-1};
ρ = density of bulk, $g\ cm^{-3}$;
η = viscosity of bulk, $cm^2\ sec^{-1}$;
D = diffusion coefficient, $cm^2\ sec^{-1}$;
V = disk linear velocity; ($V = \omega r$);
d = diameter of disk, cm

By using the method of dimensional analysis, the above equation can be written as

$$Sh = a\ Sc^b\ Re^c \qquad (11)$$

Where Sh is Sherwood number, Sc is Schmidt number and Re is Reynolds number, a, b and c are constants; the other equation used was

$$Sh = a\ Sc^{0.33}\ Re^c \qquad (12)$$

By plotting log $Sh/Sc^{0.33}$ and log Re a straight line was obtained; its slope gives constant c and intercept gives the constant a.

Figure (8) show the overall mass transfer correlation for solvent used. Table (6) summarize the values of dimensionless groups Sh, Sc and Re used in obtaining the correlations shown in figure (6), the physical properties ρ, η and D used in calculating these dimensionless groups were measured above and were given in table (5).

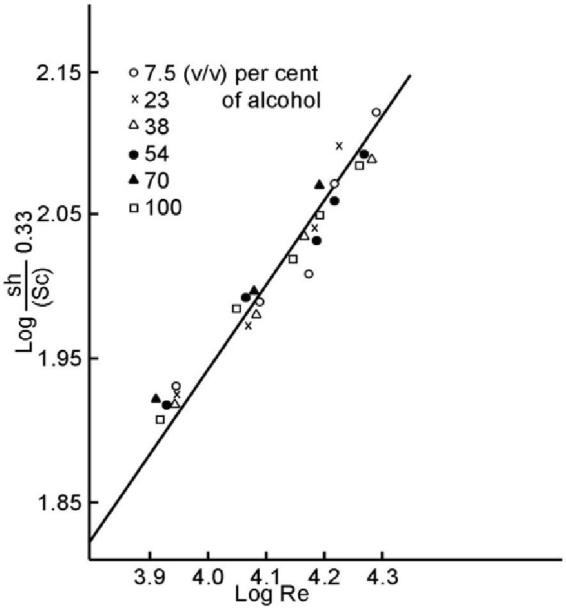

Fig. (8) The overall mass transfer correlation in presence of n-propanol.

Table (5) Viscosities and densities of phosphoric acid-n-propanol mixtures at different temperatures.

Volume % of alcohol	**D 20**	**ξ 20**	**D 25**	**ξ 25**	**D 30**	**ξ 30**	**D 35**	**ξ 35**
100	1.6338	1.5121	1.6314	1.4822	1.6288	1.4510	1.6169	1.4025
70	1.6360	1.4870	1.6349	1.4673	1.6227	1.4180	1.6205	1.3788
54	1.6388	1.4630	1.6370	1.4522	1.6351	1.4008	1.6218	1.3581
38	1.6409	1.4500	1.6390	1.4316	1.6367	1.3880	1.6353	1.3391
23	1.6431	1.4331	1.6402	1.4192	1.6390	1.3591	1.6367	1.3283
7.5	1.6456	1.4119	1.6432	1.4002	1.6410	1.3493	1.6391	1.3190

Figure (4) shows that the data can be correlated for n-propanol by the following equation:

$$Sh = 1.835\ (Sc)^{0.33}\ (Re)^{0.48} \qquad (13)$$

The exponent in the above equation donates a laminar flow which agrees with the previous mass transfer studies [35, 36 and 37] in aqueous media. It should be emphasized that the correlations obtained in our present study forced convection mechanism, which agree very well with the relationship.

$$Sh = 0.62\ (Sc)^{0.33}\ (Re)^{0.5} \qquad (14)$$

Given by Riddford for mass transfer to a rotating disk in laminar flow systems.

Conclusion

The rate of copper dissolution in presence of phosphoric acid-n-propanol mixtures was found to be inhibited by alcohol.

The rate of corrosion was found to decrease by increasing concentration of alcohol and depends on concentration of phosphoric acid, electrode height and temperatures.

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