



## Inhibition Action of Aprotic Solvents on the Electropolishing of Copper in Ortho Phosphoric Acid

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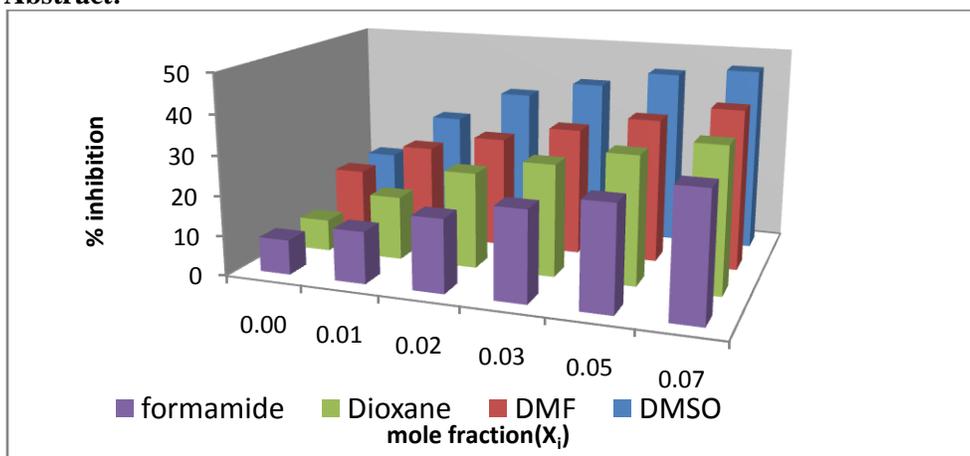
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### ABSTRACT

The inhibition action of four aprotic solvents [Dimethyl sulphoxide (DMSO), Dimethyl formamide (DMF), Dioxane and Formamide] on the electropolishing of copper in  $H_3PO_4$  acid has been investigated by Galvanostatic polarization measurements. Aprotic solvents have great dielectric constant and great dipole moment. These solvents not play as hydrogen donors. Then again the focus of negative charge density is typically on extremely basic atoms N or O, which is greatly showing for intermolecular interaction with acidic or positive site. The inhibitive effect was found to range from 8 to 46%. Thermodynamic parameters of adsorption were calculated using some adsorption isotherms. The activation energy and in addition parameters of thermodynamic for protection procedure was measured and discussed. The surface was analyzed after electropolishing (EP) treatment by AFM (Atomic force microscope) and SEM (Scanning electron microscope).

### Graphical Abstract:



**Keywords:** Electropolishing of copper, Aprotic solvents, Galvanostatic

## INTRODUCTION

Physical, mechanical and chemical characteristics of Cu give it the third world most broadly utilized metal subsequently Al and Fe. It is utilized in very significant part such as electronically uses, consumer products, manufacturing machinery, transportation, equipment, and building manufacture. The metals corrosion is communal singularity in industry, and it has established a considerable quantity of attention. The uses of chemical corrosion protection are common in production and processing operations [1, 2]. To progress the protecting efficiency for corrosion of Cu, many exertions have been put into the examination and numerous technologies have been utilized. [3-5]. Cu/H<sub>3</sub>PO<sub>4</sub> system was commonly used in Cu electropolishing. Even though the polishing electrolytic mechanism is not yet unstated in all aspects, the procedure is normally deliberated to contain both brightening and a leveling action. Throughout the procedure yields of anodic metal liquefaction react with electrolyte to give form at the surface of metal, a coating that can be liquid or gaseous, in the latter situation generally a layer of solution including a great dose of salts of anode material [6, 7]. Polar Aprotic solvents have great dielectric constants (>20) and maximum dipole moments, but hydrogen bonding not participate (no N-H bonds or O-H bonds). Their extraordinary polarity give them to liquefy charged species. The hydrogen bonding lack in solvent revenues that these nucleophiles are comparatively free in solution, production them high reactive [8, 9]. The electropolishing procedure is dispersal controlled; rely on the dose grade forming a selective electrochemical liquefaction of copper. The restrictive current data that measure the protection efficiency rely on the rate of weight transfer of Cu<sup>+2</sup> ions from the dispersal layer to the solution bulk. The mass transfer rate relies on the geometry of anode, comparative ionic transfer, temperature, physical characteristics and kind of the electrolyte [10, 11]. This study targeted to evaluate some aprotic solvents (DMSO, DMF, Dioxane and Formamide) as corrosion protection for Cu from destructive action of 8 M H<sub>3</sub>PO<sub>4</sub>. The protection effect of aprotic solvents on the Cu corrosion was studied by polarization method and the surface of Cu was examined by SEM and AFM. The adsorption parameters and thermodynamic of inhibitor on the surface of copper and copper liquefaction were research [12].

## MATERIALS AND METHODS

**Materials and solutions:** Chemical composition of Cu electrode (wt %): Cd 0.003, Ag 0.003, Pd 0.006, Sn 0.008 and Cu is the balance, AnalaR grades of Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>, 98% w/w) and the aprotic solvents, (DMSO), (DMF), Dioxane and Formamide were used in this work are used as received. second water distilled utilized to solutions readying. Cell, electrical circuit used and experimental conditions were made as in the previous work [13].

**Surface characterization:** SEM portrait were reserved utilized (JEOL, Electron microscope analytical scanning, JSM-6360 LA). Coins for SEM tests where Cu act as anode was size (1cm × 1cm). The roughness of surface (Ra) and geometric data were given by utilized (AFM) model – VEECO.

## RESULTS AND DISCUSSION

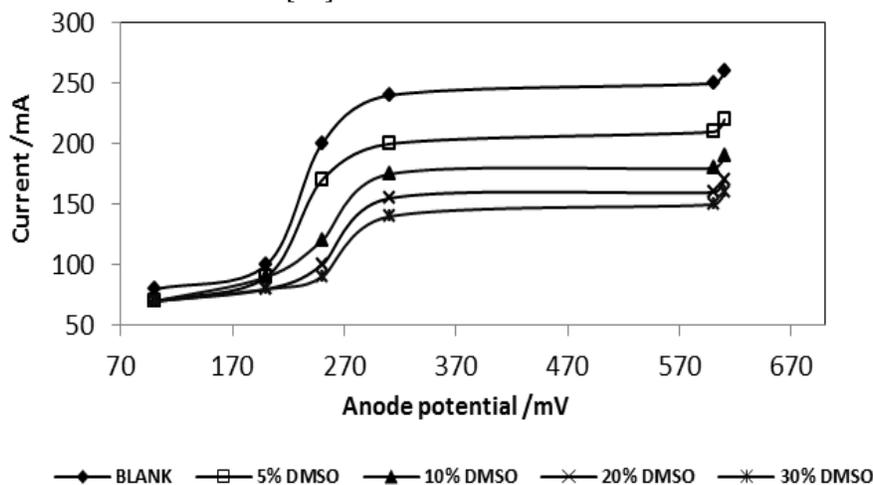
**Cu Electropolishing in H<sub>3</sub>PO<sub>4</sub> with aprotic solvents:** The polarization data of Cu in 8 M H<sub>3</sub>PO<sub>4</sub> existence and nonexistence of unlike doses of DMSO below this research is plotted in Fig. 1. Alike diagrams were given for additional solvents but not plotted. Examination of fig. 1 revealed that, polarization curves are change to less limiting current data in the attendance of DMSO. This behavior suggested the inhibitive action of DMSO. The extent of shift is in lowering current increases with rise of DMSO dose. Inspection of table 1 revealed that the observed limiting currents are found to decrease in the attendance of aprotic solvents and with improving the dose of inhibitor. It is suggested to utilize comparatively great ratio of aprotic solvents to protect corrosion of Cu in 8 M H<sub>3</sub>PO<sub>4</sub>. The IE% was calculated from the I<sub>L</sub> values obtained from the polarization curves by using the formula,

$$IE\% = \frac{I_{L(\text{blank})} - I_{L(\text{aprotic solvent})}}{I_{L(\text{blank})}} \times 100$$

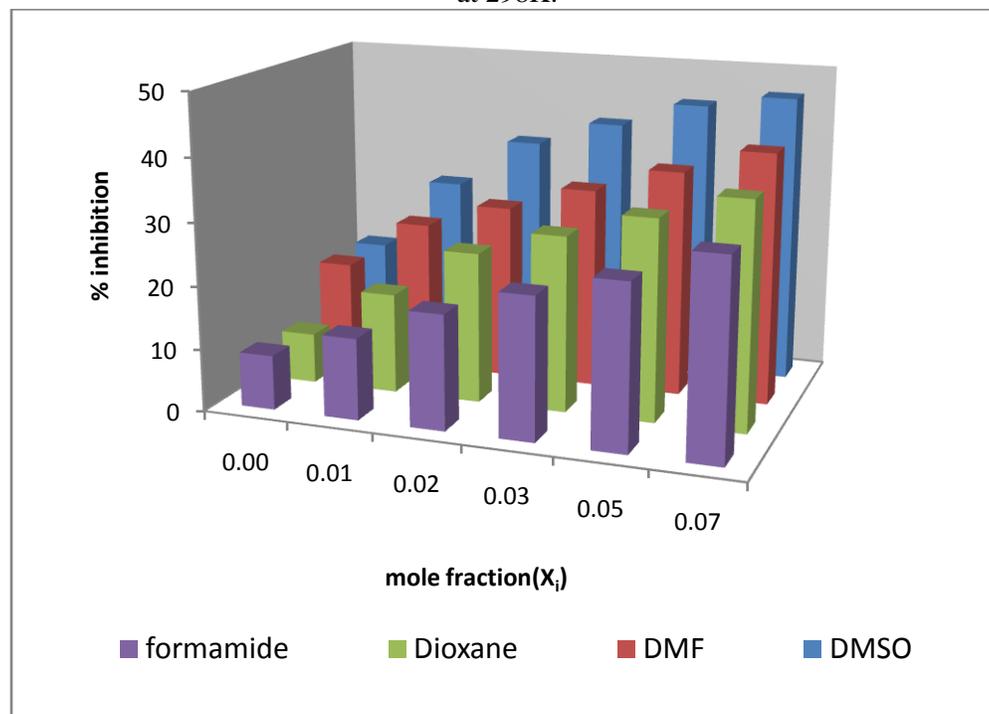
Where  $I_{L(\text{blank})}$  is the restrictive current in the attendance of aprotic solvent,  $I_{L(\text{aprotic solvent})}$  is the limiting current in the presence of aprotic solvent. The IE rises with increasing aprotic solvent dose. It could be recognized in fig. 2 and table 1 that the IE of the four aprotic solvents decreases in the following order:

$$\text{DMSO} > \text{DMF} \geq \text{Dioxane} > \text{Formamide}$$

This order results from the extent of adsorption of the solvent on the Cu surface which makes Cu surface efficiently, separated from the medium [14].



**Fig.1:** Characteristic polarization curve for the copper electropolishing existence of unlike dose of DMSO at 298K.



**Fig. 2** Relation between the percent inhibitions for different doses of aprotic solvents at 298 K in 8 M  $\text{H}_3\text{PO}_4$

Table 1: The mitigation current and % protection for all solvents at unlike temperatures

DMSO			DMF			Dioxane			Formamide			
T(K)	$X_i$	$I_l$	% IE	$X_i$	$I_l$	% IE	$X_i$	$I_l$	% IE	$X_i$	$I_l$	% IE
298K	0	250	0	0	250	0	0	250	0	0	250	0
	0.003	210	16.0	0.008	210	16.0	0.003	230	8.0	0.011	228	8.8
	0.006	180	28.0	0.020	190	24.0	0.006	200	16.0	0.029	217	13.0
	0.016	160	36.0	0.042	180	28.0	0.016	190	24.0	0.059	190	18.3
	0.032	150	40.0	0.065	170	32.0	0.032	180	28.0	0.090	187	22.6
	0.050	130	44.0	0.089	160	36.0	0.050	170	32.0	0.123	158	26.1
303K	0	280	0	0	280	0	0	280	0	0	280	0
	0.003	230	17.9	0.008	230	17.9	0.003	230	17.9	0.011	215	14.0
	0.006	205	26.8	0.020	210	25.0	0.006	210	25.0	0.029	200	20.0
	0.016	220	21.4	0.042	200	28.6	0.016	200	28.6	0.059	204	24.0
	0.032	175	37.5	0.065	180	35.7	0.032	185	33.9	0.090	194	33.2
	0.050	160	42.9	0.089	170	39.3	0.050	175	37.5	0.123	180	35.7
308K	0	310	0	0	310	0	0	310	0	0	310	0
	0.003	250	21.9	0.008	240	22.6	0.003	250	19.4	0.011	230	23.3
	0.006	220	31.3	0.020	220	29.0	0.006	230	25.8	0.029	215	28.3
	0.016	235	26.6	0.042	210	32.3	0.016	210	32.3	0.059	200	33.3
	0.032	200	37.5	0.065	190	38.7	0.032	190	38.7	0.090	190	36.7
	0.050	180	43.8	0.089	180	41.9	0.050	180	41.9	0.123	185	40.3
313K	0	350	0	0	350	0	0	350	0	0	350	0
	0.003	290	17.1	0.008	260	25.7	0.003	280	20.0	0.011	250	28.6
	0.006	240	31.4	0.020	230	34.3	0.006	250	28.6	0.029	230	34.3
	0.016	250	28.6	0.042	230	34.3	0.016	230	34.3	0.059	210	40.0
	0.032	220	37.1	0.065	200	42.9	0.032	200	42.9	0.090	200	42.9
	0.050	190	45.7	0.089	190	45.7	0.050	190	45.7	0.123	190	45.7
0.070	170	51.4	0.115	180	48.6	0.070	185	47.2	0.158	187	46.5	

**Temperature characteristic and activation electropolishing parameters:** The apparent activation energy,  $E_a^*$  for electropolishing of Cu coins in 8 M  $H_3PO_4$  solution in the existence and lake of aprotic solvents were determined by the least squares method and values are given in Table 2. The +ve sign of  $\Delta H^*$  reproduces the endothermic Cu liquefaction procedure, which means that dissolution of Cu is difficult in aprotic solvents presence [15-18].

The change in  $\Delta S^*$  in the existence and nonexistence of the aprotic solvents is -ve sign. This suggests that the actuated complex in the rate determining stage signifies an association somewhat than dissociation signifying that more order occurs accepted from reactant to activated complex [19]. The plot of  $\Delta H^\ddagger$  versus  $\Delta S^\ddagger$ , Fig. 3, for different aprotic solvents at 298K was found to be linear and this suggests that, all of aprotic solvents used have the same mechanism during the electropolishing process. The isokinetic temperature  $\beta$  obtain from the plot slope is 289 K, which is lesser than that of the investigational temperatures, demonstrating that the reaction rate is entropy control [20].

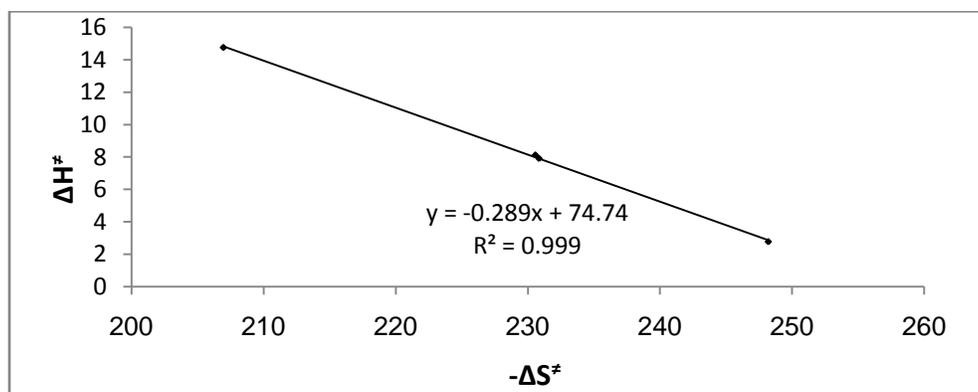
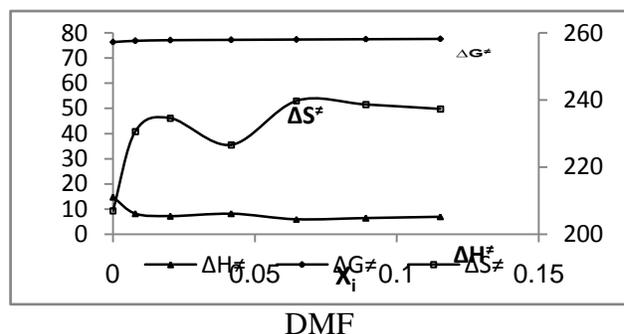
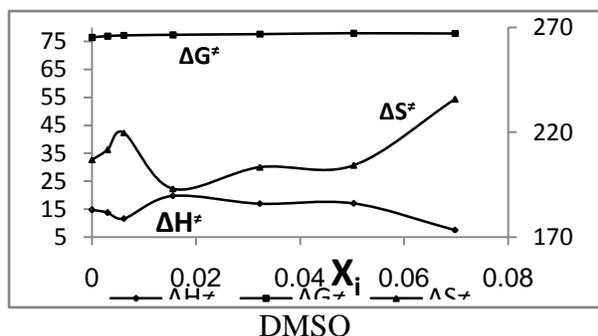


Fig. 3: The relation among  $-\Delta S^*$  and  $\Delta H^*$  for unlike aprotic solvents at 298 K

Table 2. The data of activated in existence and lack aprotic solvents in 8 M  $H_3PO_4$  at 298

DMSO	$E_a^*$ kJ mol <sup>-1</sup>	$H^*\Delta$ kJ mol <sup>-1</sup>	$-\Delta S^*$ J mol <sup>-1</sup> .K <sup>-1</sup>	$\Delta G^*$ kJmol <sup>-1</sup>	DMF	$E_a^*$ kJmol <sup>-1</sup>	$H^*\Delta$ kJ mol <sup>-1</sup>	$-\Delta S^*$ Jmol <sup>-1</sup> .K <sup>-1</sup>	$G^*\Delta$ kJmol <sup>-1</sup>
$X_i$					$X_i$				
0	17.23	14.75	206.95	76.42	0	17.23	14.75	206.95	76.42
0.003	16.28	13.80	211.66	76.88	0.008	10.60	8.12	230.57	76.83
0.006	14.13	11.66	219.84	77.17	0.020	9.64	7.16	234.56	77.06
0.016	22.25	19.77	193.16	77.33	0.042	12.16	8.12	226.59	77.21
0.032	19.50	17.00	203.39	77.63	0.065	8.40	5.92	239.75	77.37
0.050	19.55	17.08	204.24	77.94	0.089	8.88	6.41	238.67	77.53
0.070	10.04	7.56	235.88	77.85	0.115	9.42	6.95	237.36	77.68
Dioxane	$E_a^*$ kJ mol <sup>-1</sup>	$\Delta H^*$ kJ mol <sup>-1</sup>	$-\Delta S^*$ J mol <sup>-1</sup> .K <sup>-1</sup>	$G^* \Delta$ kJ mol <sup>-1</sup>	Formami de	$E_a^*$ kJ mol <sup>-1</sup>	$\Delta H^*$ kJ mol <sup>-1</sup>	$-\Delta S^*$ J mol <sup>-1</sup> .K <sup>-1</sup>	$\Delta G^*$ kJ mol <sup>-1</sup>
$X_i$					$X_i$				
0	17.23	14.75	206.95	76.42	0	17.22	14.75	206.95	76.42
0.003	10.39	7.91	230.85	76.70	0.011	5.26	2.78	248.21	76.75
0.006	11.77	9.30	227.19	77.00	0.029	3.75	1.27	252.74	76.59
0.016	9.62	7.15	234.80	77.12	0.059	4.66	2.18	251.36	77.80
0.032	5.30	2.82	249.72	77.24	0.090	2.80	0.32	248.69	74.42
0.050	5.98	3.12	249.21	77.39	0.123	8.33	5.85	237.92	76.75
0.070	7.38	4.91	243.69	77.53	0.158	9.06	6.58	237.92	77.48

**Relation between mole fraction and thermodynamic parameters:** The estimated values of  $\Delta G^*$ ,  $\Delta H^*$  and  $\Delta S^*$  at 298 K as showed in fig. 4 and table 2 expresses the variation of  $\Delta G^*$ ,  $\Delta H^*$  and  $\Delta S^*$  with mole fractions of the aprotic solvents. The slow rise of both  $\Delta S^*$  and  $\Delta H^*$  gives excellent sign of special solvation of surface in attendance of aprotic dose solvent, this is a gauge of exact solution. Also the weak rely of  $\Delta G^*$  on the arrangement of the protic solvent can be qualified to recompense effect among  $\Delta H^*$  and  $\Delta S^*$ .



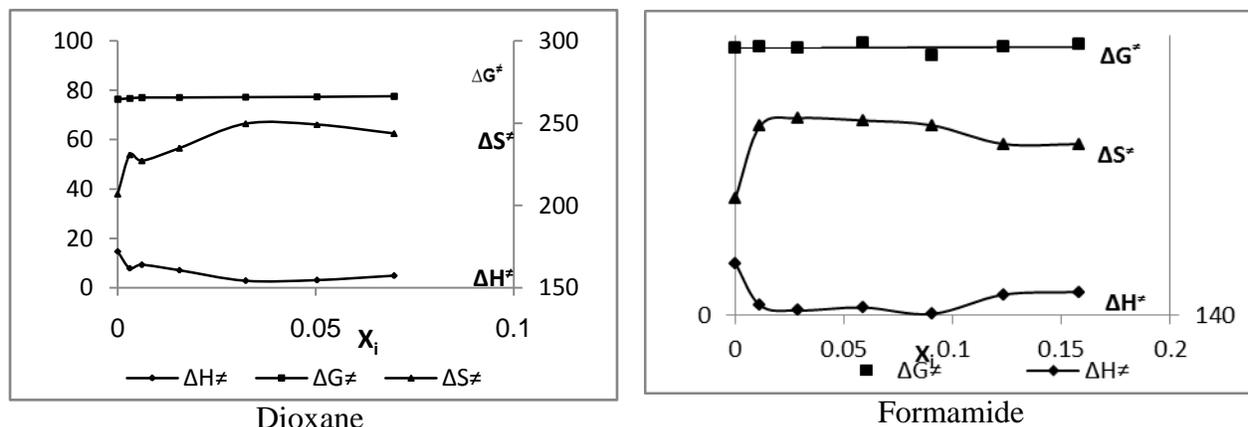


Fig. 4: The relation between thermodynamic parameters for electropolishing of Cu at different aprotic solvents at 298 K

**Effect of dielectric constant of polar aprotic solvents:** From Fig. 5 give the relative among the lower current and dielectric constants (D) of the aprotic solvents at 298 K. It is obvious that the electropolishing rate is rise by improvement the dielectric constant of solvent. It is found that DMSO, DMF and dioxane, which possess a high dielectric constant compared to formamide, are the strongest inhibitors for electropolishing of Cu. This can be explained on the basis that the ionic mobility of  $\text{Cu}^{2+}$  increases as the dielectric constant increases and the chance to form ion-pair decreases. The plot of  $\log I_L$  versus  $1/D$ , completed the whole series of solvent arrangement give linear for all the aprotic solvent systems, with high correlation coefficient ( $R^2 > 0.95$ ) according to Born's equation [21].

$$\log I_L = \frac{-\Delta G_{n.e.s}^\circ}{2.303 RT} - \frac{Ne^2}{4.606 RT} \left( \frac{1}{r^+} - \frac{1}{r^-} \right) \frac{1}{D}$$

The linear plot obtained in fig. 5 indicates that the change of the  $I_L$  with D is governed by electrostatic effects. The intercept of the line represents the nonelectrostatic part of the free energy of association, conforming that ( $\Delta G_{n.e.s}^\circ$ ) has no contribution during reaction mechanism.

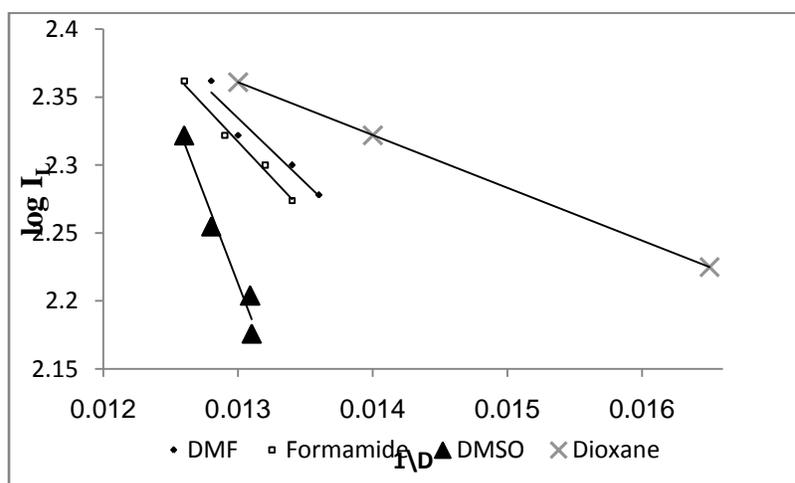


Fig 5. The relation between dielectric constant of aprotic solvents and limiting current at 298 K

**Adsorption isotherms:** Adsorption isotherms provide knowledge on adsorption procedure such as adsorption equilibrium constant, coverage of surface and a knowledge on the contact among the aprotic solvents and the surface of electrode. The ( $\theta$ ) data for unlike doses of the solvents in 8 M  $\text{H}_3\text{PO}_4$  were measured utilized the next equation:

$$\theta = \frac{I_{blank} - I_{aprotic\ solvent}}{I_{blank}}$$

The adsorption mechanism was given by appropriate surface coverage ( $\theta$ ) data to various adsorption isotherms for example Kinetics-adsorption Flory-Huggins and Langmuir [22, 23].

**Langmuir isotherm:** Inhibitor adsorption appearances can be assessed from the following equation:

$$\frac{C}{\theta} = \frac{1}{K} + C$$

Where C = dose of inhibitor and K = equilibrium constant, demonstrating the degree of adsorption (i.e., the greater data of K led to that the inhibitor is powerfully adsorbed on the Cu).

Regressions among  $\frac{C}{\theta}$  and C were measured and the parameters are planned in fig. 6 and Table 3. These outcome data give that all the coefficients linear relationship ( $R^2$ ) are close to unity, and all the line slopes are not equal to one, which suggests that the adsorption of aprotic solvents in 8 M  $H_3PO_4$  on Cu not follow adsorption Langmuir [24].

**Flory-Huggins isotherm:** The equation Flory-Huggins adsorption is represented by:

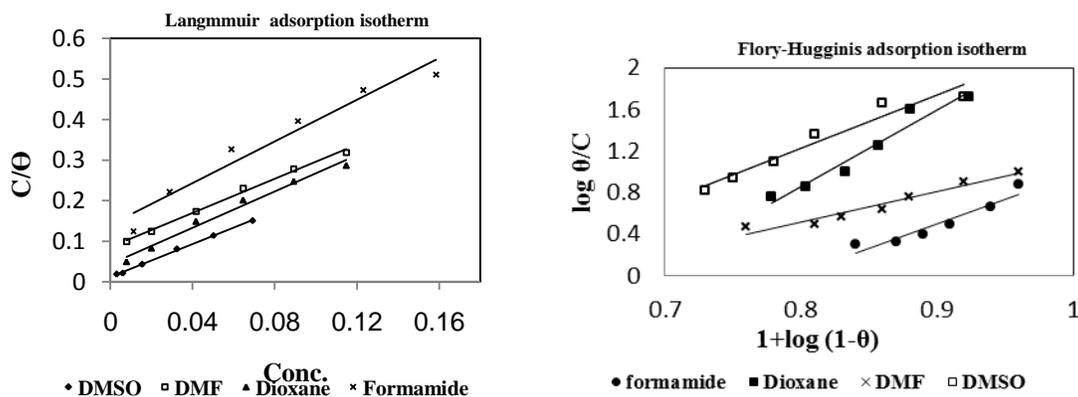
$$\log \theta / C = x \log (1 - \theta) + \log x K$$

Fig. 6 by plotting  $\log \theta / C$  against  $\log (1 - \theta)$  at 298 K. Straightforward lines are received with intercept  $\log x K$  and a slope x, where x = water atom number exchanged by one solvent molecule. The inhibitors adsorb at interface solution-Cu may be due to the founding of covalent connection or electrostatic among the adsorbents and the surface of Cu [25].

**The kinetic isotherm:** Kinetic isotherm may be typing as follow:

$$(KC)^y = \left(\frac{\theta}{1-\theta}\right)$$

Where K = equilibrium constant and y = extract atom number inhabiting an active center. It is obvious from Fig. 6 that, the 1/y data rely on the inhibitors kinds, which are calculated in Table 3, the 1/y data given were extra than unity which lead to that each atom of the aprotic solvents include in the adsorption procedure was committed to more than one active center on Cu surface. K characterizes the strong point among adsorbent and adsorbate. Greater K data give good  $\theta$  and more adsorption efficient [26, 27].



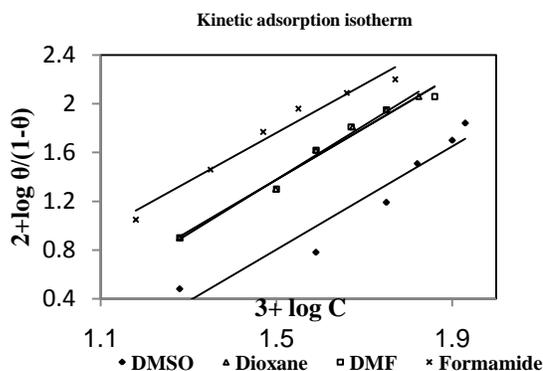


Fig 6. Adsorption figure for aprotic solvents in 8 M  $H_3PO_4$  on the Cu surface at 298 K

Table 3. Parameters of adsorption and data measured of  $\Delta G_{ads}$  for 8 M  $H_3PO_4$  and aprotic solvents at 298 K

Aprotic solvents	Flory-Huggins isotherm		Kinetic adsorption isotherm				Langmuir isotherm		
	X	K	$-\Delta G_{ads}$	K	1/Y	K	$-\Delta G_{ads}$	$R^2$	Slope
DMSO	5.11	34.33	18.72	13.4 1	2.28	12.36	16.19	0.9984	2.02
DMF	2.95	28.35	18.25	4.09	2.18	3.47	13.04	0.9947	2.11
Dioxane	7.33	1.347	13.59	1.21	2.26	3.14	12.80	0.9839	2.24
Formamide	4.76	1.997	11.67	1.70	2.27	1.16	10.33	0.9515	2.57

**Structural effects of the aprotic solvents:** In this work the order of aprotic solvent inhibition effect for corrosion of Cu electrode are:



This effect of inhibition for corrosion appear due to the adsorption of these solvents on the Cu electrode through lone pair of (O or N) atoms in solvents under investigation and their ability to coordinate to the electrode depending on the atmosphere for each lone pair.

In case of Formamide there are conjugations between lone pair of electron of atom N and O in formamide which enhance the ability of lone pairs of electron for the coordination to the electrode. The

effect increase in case of DMF due to the presence of two methyl groups, and in case of

Dioxane there are two centers of Oxygen atoms each one have their lone pairs. Finally the effect

reach the highest in DMSO due to the high polarity of the S = O than (C = O)

### Surface characteristics

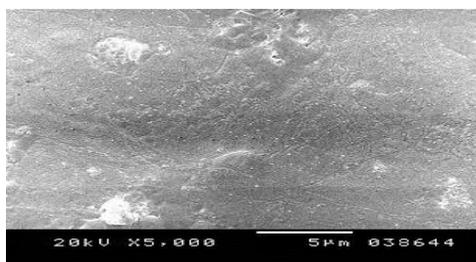
**SEM micrographs of electropolished samples of Cu in 8 M  $H_3PO_4$ :** The polished surfaces were analyzed by (SEM) to examine the morphology of surface and presented in Fig.7 at different conditions. Fig.7(A-G) shows a comparison between samples after electropolishing with and without appending of DMSO, DMF, dioxane and formamide, they exposed the improvement of surface uniformity [28]. With the presence of aprotic solvents, films are able to protect Cu from corrosion according to the outcomes of the electropolishing measurements.

Fig. 7(A) give the morphology of the electropolished Cu in 8 M  $H_3PO_4$  in absence of aprotic solvents (blank) at temperatures 298 K. The surface of specimen shows small scratches and deep cavities with large number of pits.

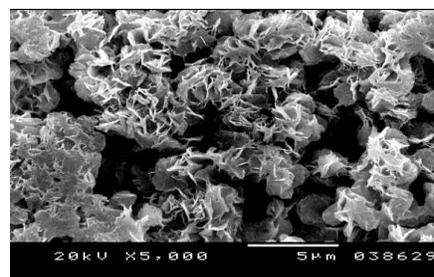
In Fig. 7(E, G) which represent the morphology of the electro polished Cu in 8 M  $H_3PO_4$  and DMSO 10% at 298 K and 308 K, respectively, it is clear that as the temperature increases, the surface roughness increases. At high temperatures, the surface was badly damaged as shown from the presence of deep cavities and drawbacks [29].

Fig. 7(B, C, D, E) gives the morphology of the electropolished Cu in 8 M  $H_3PO_4$  with formamide, dioxane, DMF, DMSO, respectively as additives at 10 % and 298 K. In case of addition DMSO, the surface appears to be high brightness, uniformity, smoother and no pits were observed rather than in case of addition of DMF, dioxane and formamide.

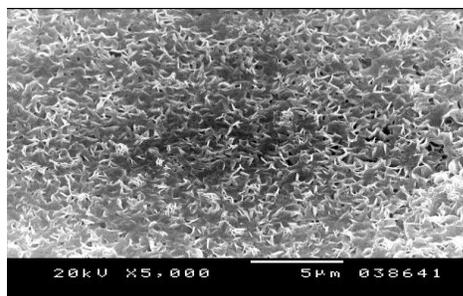
Fig. 7(E, F) gives the morphology of the electropolished Cu in 8 M  $H_3PO_4$  with DMSO as additive at 10 % and 30%, electropolishing treatments showed that surface roughness of Cu decreases with increasing doses of DMSO. Therefore, the surface morphology was smooth, adherent with a little small marks and the surface coverage was quite satisfactory [30].



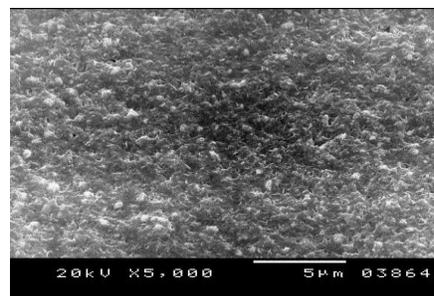
A: Blank at 298 K



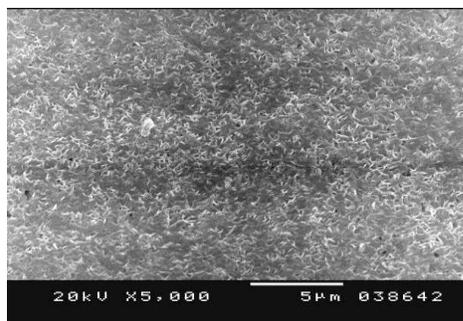
B: Formamide 10%, 298 K



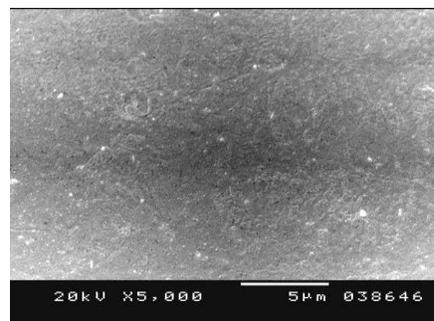
C: Dioxane 10%, 298 K



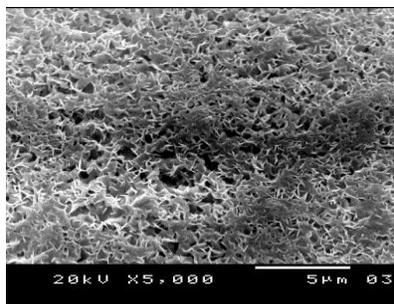
D: DMF 10%, 298 K



E: DMSO 10%, 298 K



F: DMSO 30%, 298 K



G: DMSO 10%, 308 K

**Fig. 7:** Surface morphologies of the Cu after electropolishing in 8 M  $H_3PO_4$  and in presence of aprotic solvents

**AFM analysis:** The AFM offers a commanding means of describing the 3D- images and the 2D- images topography for micro structure of the Cu surface before the electropolishing and after electropolishing in 8 M  $H_3PO_4$  in nonexistence and attendance of polar aprotic solvents, are given in Figs.(8 and 9), correspondingly. Row samples looked non uniformity, non-homogeneity and some parts to a little mound like assembly [31]. After electropolishing in 8 M  $H_3PO_4$  and absence of polar aprotic solvents, AFM image revealed the decrease of roughness, the portrait is moderately identical and some parts had a random hill like structure [32]. Ra was lower by the addition of polar aprotic solvents, surface appear relatively uniform homogenous, random hills structure are reduced and Ra decreased from formamide to DMSO. Thus, it might be concluded that these aprotic solvents make an adsorption coat of the inhibitor on the Cu, which efficiently inhibits the electropolishing Cu. Ra data lead to that EP of Cu surface more effective than in example of presence of DMSO more than the other aprotic solvents [33].

All the above results can be confirmed from Table 4 in which the roughness obviously decreased after electropolishing, where Ra decreased from 251.1 nm to 215.2nm nonexistence appending of any aprotic solvents. Ra was lower from 251.1 nm to 186.0 nm by appending of formamide, but lower to 125.4 nm by appending of dioxane, decreased to 111.9 nm by appending of DMF and lower to 106.9 nm by appending of DMSO. The Ra data designated that electropolishing of Cu surface effective rather in case of appending DMSO than other aprotic solvents (DMF, Dioxane and Formamide). Moreover, the picture statistics give also, that (RMS) root mean square lower from 317.2 to 141.8 nm after electropolishing in the case of DMSO [34].

**Table 4.** Measured roughness (Ra) and RMS roughness (Rq) of Cu coins

Sample	EP conditions	Ra (nm)	RMS (Rq) (nm)
a	Before EP	251.1	317.2
b	After EP without additives	215.2	274.4
c	After EP + Formamide	186.0	242.8
d	After EP +Dioxane	125.4	158.1
e	After EP + DMF	111.9	144.6
f	After EP + DMSO	106.9	141.8

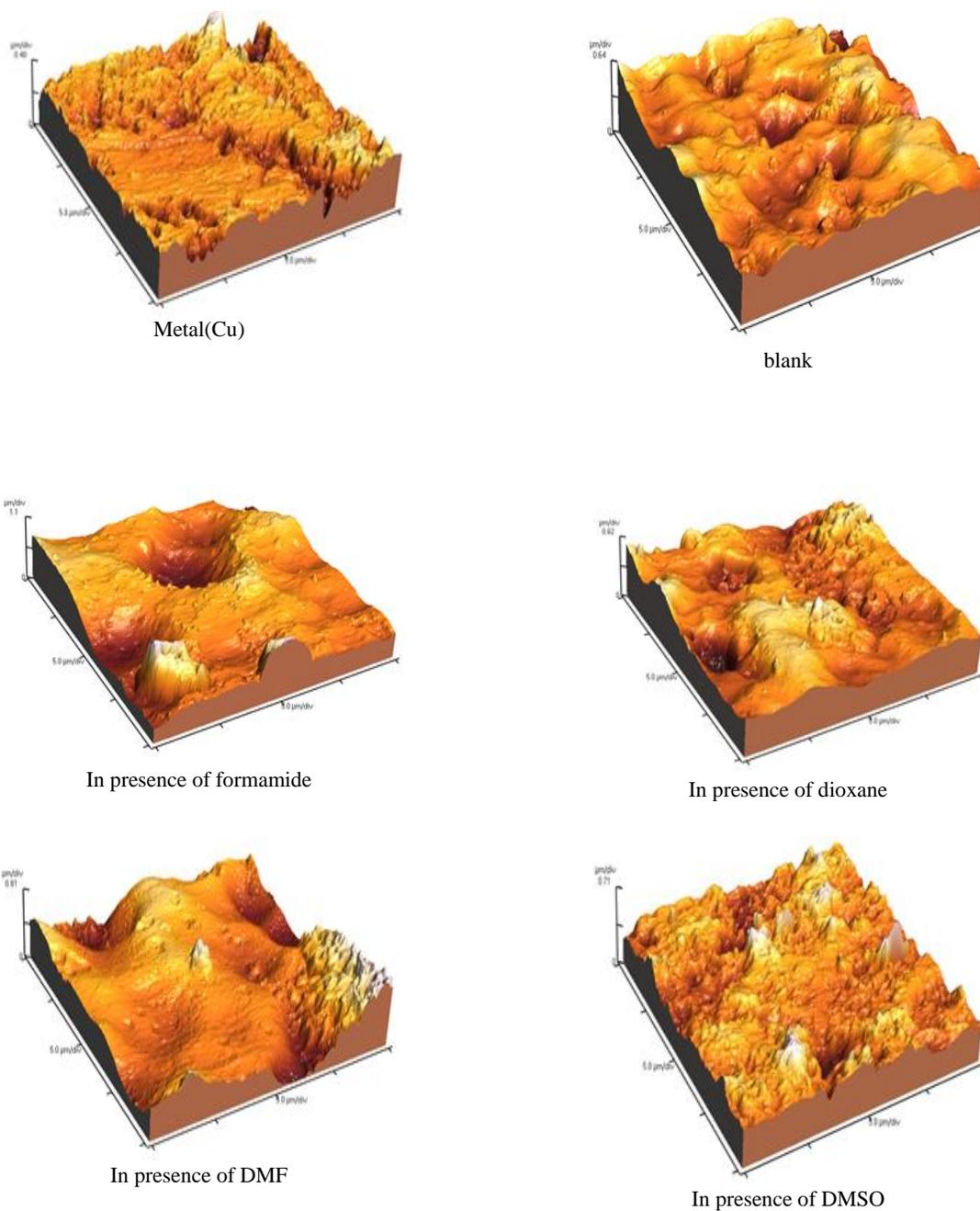
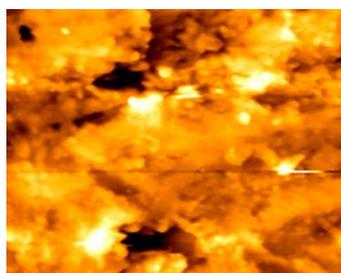
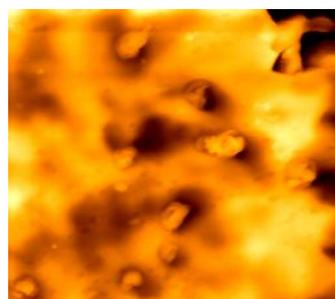


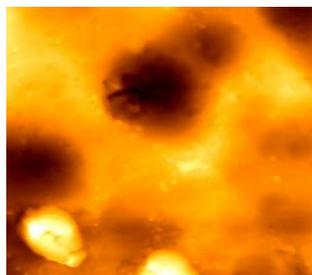
Fig. 8 AFM 3d of the Cu surface in 8 M H<sub>3</sub>PO<sub>4</sub> existence and lack of polar aprotic solvents



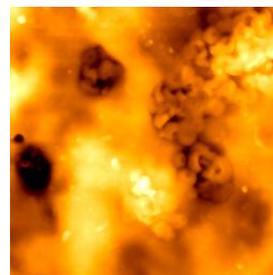
Cu



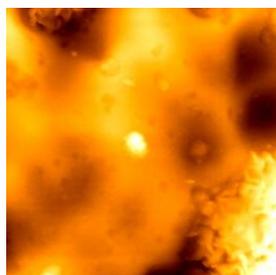
blank



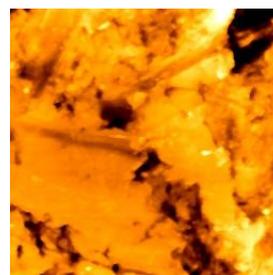
In presence of formamide



In presence of Dioxane



In presence of DMF



In presence of DMSO

Fig 9. AFM 2d of the Cu surface in 8 M H<sub>3</sub>PO<sub>4</sub> existence and lack of polar aprotic solvents

## CONCLUSIONS

Electropolishing behavior of Cu in a bath include the four aprotic solvents could rise the capability of the bath to yield continuous polishing. Improvement produced in electropolishing was outstanding to the adsorption of aprotic solvents on the anode Cu surface. The augmentation of the morphology Cu surface and topography was succeeded by appending of some aprotic solvents for example, DMSO, DMF, dioxane or formamide. These additives act as vital role to decrease pitting and defects formed by evolution of O<sub>2</sub>. From the AFM and SEM tests, the roughness (Ra) data led to that electropolishing of Cu surface is more effective in appending of DMSO (Ra = 106.9 nm) more than DMF (Ra = 111.9 nm), Dioxane (Ra = 125.4 nm) and formamide (Ra = 186.0 nm).

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