### Nano Polymers and Their Effect on Metal Electroplating

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### I. Introduction

A polymer (or polymer) Is a high molecular weight compound composed of repeating subunits, these materials may be organic, inorganic, or mineral-organic, and may be natural or synthetic in origin. [1]. Nanopolymers have come to play an essential and holistic role in everyday life due to their unique properties<sup>[2]</sup>. Electrodepositionis the process of producing a coating, usually metallic on a surface by the action of electric current as a thin layer. [1] Electroplating is often called "Electrodeposition". [3] Electroplating can be considered to occur by the process of electrodeposition and is a specific type of surface finishing. [4] The benefits of such process is to Improve appearance, Slow or prevent corrosion (rust) and Increase strength and resistance to wear for engineering finishes. [5] The kinetics of the electrodeposition of metals involves mainly two steps, Mass transfer step where the metal ions reach the cathode surface from the bulk solution and Charge transfer step where the metal ions combine with electrons at the cathode surface to form metal atoms. [6] The mass transfer of copper ions occurs through the following primary mechanisms, an electric migration under a potential gradient, adiffusion by ion density difference. A convective motion of solution. Different organic substances are used as additives in electrodeposition to modify the physical properties of the deposit. [7] In this study Nano Polymers are used as additives and Nano Polymers are a high molecular weight substances some of them perform important functions in livening organisms and some of them have an important function in industry. [8] Nano Polymers are classified to natural Nano Polymers for example (Protiens and Carbohydrates) and synthetic Nano Polymers for example (Poly ethylene glycol and Poly vinyl alcohol). [9] Adsorption of such organic compounds on the cathode surface may block a part of the active electrode area and therefore, reduce the mass transfer coefficient and the limiting current. [10] Additives inhibit electrode reactions, primary inhibition, caused by substances whose molecular composition doesn't change during electrodeposition process and secondary inhibition, caused by substances whose generated during electrode reaction by chemical reaction which competes with the electrodeposition of metals. [11] Additives accelerate electrode reactions, they accelerate the copper electrodeposition through the formation of ion-complex in solution. [12]. Electrodeposition of metals is used widely in industry to perform the following technical processes, electroplating, electroforming electrorefining of metals, electrowinning of metals from their natural ores, recovery of heavy metals e.g.: Cu, Cd, Hg, Pb, Ag from waste solutions discharged from metal finishing industries ,Effect of Inhibitors on electrodeposition kinetics, An inhibitoris a chemical substance which, when added in small concentrations to an environment,

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decreases or prevents the reaction of the metal with the environment.  $^{[13]}$ It is added to many systems including, cleaning path, cooling systems, pipelines, chemical operations, steam generators oil and gas production, Organic Inhibitors can be classified as cathodic, anodic, or mixed inhibitors. This classification depends on its reaction at the metal surface and how the potential of the metal is affected.  $^{[14]}$  Thefactors contributing the effectiveness of inhibitor ,The size of the Organic Nano Polymers , The aromaticity and/ or conjugated bonding , Bonding strength to metal substrate , The type and the number of bonding atoms or groups in the molecule and Ability for layer to become compact, or cross linked.  $^{[15]}$ 

### II. The aim of the work

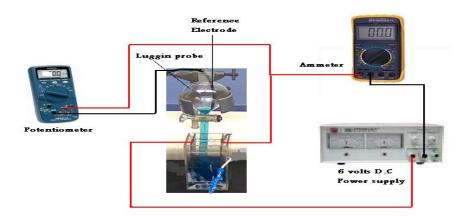
- 1-The rate of copper deposition using (Cu-Cu) cell and in case of (Cu-Stainless steel) cell in absence and presence of organic Nano Polymers .
- 2-Effect of temperature on the rate of electroplating in presence of different organic Nano Polymers .
- 3-Determination of the type of adsorption isotherm.
- 4-Calculation of the thermodynamic parameters.
- 5- Effect of rotation on the rate of electroplating using RCE in presence of those organic Nano Polymers in case of cupper at 25°C.

### III. Materials and Method

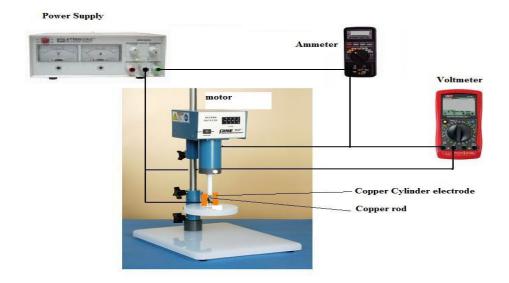
### **Experimental Techniques, Chemicals were used:**

- ✓ Analar grade CuSO4 and H2SO4 (98% w/w) were used for preparation of the electrolyte. <sup>[16]</sup>
- ✓ Organic additives(Poly ethylene glycol with different molecular weights,(400-4000-6000) andPoly vinyl alcohol(7000) [17]

### **Vertical Parallel Plates cell**<sup>[18]</sup>



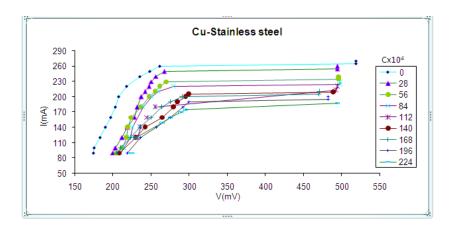
The Rotating Cylinder Electrode (RCE). [19]



IV. Results and Discussions

### **Polarization Curves**

 The Following Fig. shows the cathodic polarization curve for copper electrodeposition from sulphate solution under the influence of adding different concentrations of PEG-400 in case of Cu-Stainless steel cell at 303 K.



The electrodeposition of Cu<sup>2+</sup> ions takes place through two steps;

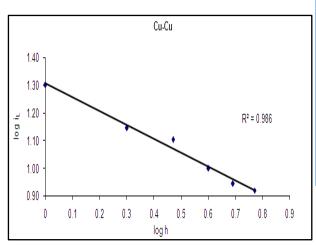
- 2. Thisprocess controlled by rate of Cu<sup>2+</sup> diffusion to the electrode surface.
- 3. Addition of organic Nano Polymers to sulphate solution increases the cathodic polarization and decrease the value of limiting current denisty.
- 4. This organic Nano Polymers acting as inhibitors because the current density for copper deposition from solutions containing Organic Nano Polymers is lower than that found for corresponding Organic Nano Polymers free solution. This inhibition due to adsorption of Organic Nano Polymers on the cathode surface.

The relation between the limiting current and concentration of Organic Nano Polymers at different temperature.

			Cu	ı-Cu		Cu-Stainless steel			
OrganicnNanoPolymer	C×10°	25° C	30° C	35° C	40° C	25° C	30° C	35° C	40° C
	mol/l								
	0	260	293	368	415	255	265	280	305
	28	260	290	361	410	240	255	260	256
	56	257	286	359	406	227	234	245	250
	84	254	286	355	401	225	225	240	247
PEG-400	112	250	284	355	396	210	213	233	235
	140	246	280	350	392	205	209	226	230
	168	243	277	346	390	198	205	215	226
	196	240	274	344	387	187	195	209	218
	224	240	270	340	387	186	187	196	215
	C×10°	25° C	30° C	35° C	40° C	25° C	30° C	35° C	40° C
	mol/l								
	0	260	293	368	415	255	265	280	305
	2.5	255	286	360	405	225	230	240	255
	5	250	280	356	400	220	221	238	241
PEG-4000	7.5	243	278	350	396	210	218	235	235
	10	243	275	347	392	192	215	229	231
	12.5	240	270	343	390	190	210	223	225
	15	236	270	340	387	185	207	218	220
	17.5	232	265	338	384	182	200	210	215
	20	232	263	334	384	180	195	205	210
	C×10°	25° C	30° C	35° C	40° C	25° C	30° C	35° C	40° C
	mol/l								
	0	260	293	368	415	255	265	280	305
	2.5	250	284	356	405	220	225	240	245
	5	246	277	350	400	215	220	229	231
PEG-6000	7.5	241	275	345	397	205	213	218	225
	10	238	271	341	395	190	212	216	223
	12.5	234	271	340	395	189	210	215	220
	15	232	266	335	391	183	202	210	213
	17.5	230	260	335	386	180	193	196	200
	20	226	255	330	382	179	190	195	198
	C×10°	25° C	30° C	35° C	40° C	25° C	30° C	35° C	40° C
	mol/l								
	0	260	293	368	415	255	265	280	305
	3.33	254	283	355	409	225	250	268	295
	6.6	250	267	342	400	215	244	264	293
Polyvinyl alcohol	9.9	239	260	336	396	209	236	259	288
	13.2	234	253	328	386	205	228	254	286
	16.5	224	227	323	382	200	217	252	286
	19.8	209	223	318	379	197	210	250	284
	23.1	204	218	310	376	195	205	247	280
	26.4	202	213	308	374	192	203	245	277

Effect of Electrode Height on Limiting Current Density.

h (am)	Cu-Cu cell	Cu-Stainless		
h (cm)	Cu-Cu cen	steel cell		

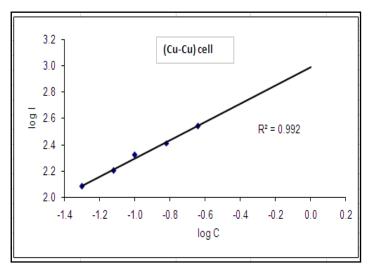


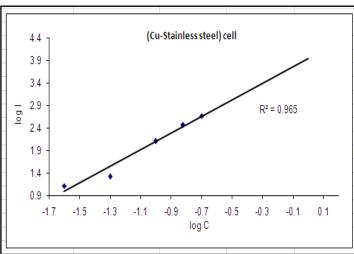
	I(mA)	$Log \ i_L$	I(mA)	$Log \ i_L$
1	100	1.30	106	1.33
2	140	1.15	188	1.27
3	190	1.10	200	1.12
4	200	1.00	250	1.00
5	220	0.94	320	1.03
6	250	0.92		

height.

The limiting current density decreases with increase of

### Effect of Electrolyte Concentration on the Deposit Current:





### The increases in the limiting current with CuSO<sub>4</sub> concentration is due to:

- 1. The solubility of dissolved copper sulphate in sulphuic acid, which is responsible for the limiting current, increases with increasing copper sulphate concentration.
- 2. The viscosity of the solution increases with increasing a concentration with a consequence decrease in the mobility of  $Cu^{+2}$  according to

**Slokes-Einsiein equation:** 

### Effect of organic Nano Polymers concentrations on the limiting current density:

1. The percentage inhibition can be calculated from the following equation:

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### % inhibition= $(I - I_l / I) \times 100$

2. The rate of electrodeposition decreases in case of Cu-Stainless steel cell more than in case of Cu-Cu cell in case of all Nano Polymers added except in case of Poly vinyl alcohol.

### Values of % inhibition for all organic compounds at 303 K.

Organic			Cu-Cu	Cu	Cu-Stainless steel		
NanoPolymer	C×10° mol/l	I(mA)	% Inhibition	I(mA)	% Inhibition		
	0.0	293	0.00	265	0.00		
	28.0	290	1.02	255	3.77		
	56.0	286	2.39	234	11.70		
	84.0	286	2.39	225	15.09		
PEG-400	112.0	284	3.07	213	19.52		
123 400	140.0	280	4.44	209	21.13		
	168.0	277	5.46	205	22.64		
	196.0	274	6.48	195	26.42		
	224.0	270	7.85	187	29.43		
PEG-4000	0.0	293	0.00	265	0.00		
	2.5	286	2.39	230	13.21		
	5.0	280	4.44	221	16.60		
	7.5	278	5.12	218	17.74		
	10.0	275	6.14	215	18.87		
120 1000	12.5	270	7.85	210	20.75		
	15.0	270	7.85	207	21.89		
	17.5	265	9.56	200	24.53		
	20.0	263	10.24	195	26.42		
	0.0	293	0.00	265	0.00		
	2.5	284	3.07	225	15.09		
PEG-5000	5.0	280	5.46	220	16.98		
	7.5	278	6.14	213	19.62		
	10.0	275	7.51	212	20.00		

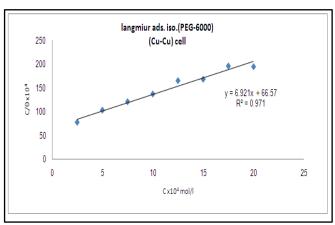
12.5	271	7.51	210	20.75
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Organic	Cu-Cu cell	Cu-Stainless steel
I(mA)	Slope	Slope
1) PEG-400	7.81	2.33
2) PEG-4000	5.51	3.44
3) PEG-6000	6.92	2.76
4) Poly vinyl alcohol	3.06	5.48

	15.0	266	9.22	202	23.77
	17.5	260	11.26	193	27.17
	20.0	332	11.97	190	28.30
	C×10° mol/l	I(mA)	% Inhibition	I(mA)	% Inhibition
	0.0	293	0.00	265	0.00
	3.3	283	3.41	250	5.55
	6.6	267	8.87	244	7.92
Dolor of worl	9.9	260	11.26	236	10.94
Poly vinyl alcohol	13.2	253	13.65	228	13.95
	16.5	227	22.53	217	18.11
	19.8	223	23.89	210	20.75
	23.1	218	25.50	205	22.64
	26.4	213	27.30	203	23.40

### Adsorption isotherm

The most used isotherms are Langmuir, Timken, Flory Huggins and Kinetic adsorption.



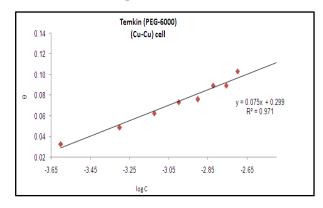
isotherm.

### Langmuir adsorption isotherm

### Langmuir adsorption isotherm is given by: $C/\theta {=} 1/K + C \label{eq:condition}$

Where C is the concentration of polymer,  $\theta$  fractional surface coverage and K is the adsorption equilibrium constant. So that a linear — relationship can be obtained on plotting  $C/\theta$  as a function of C for organic Nano Polymers . It is observed that although this plot gives a good straight line, the gradients are never unity and not obey the Langmuir adsorption

### Temkin adsorption isotherm



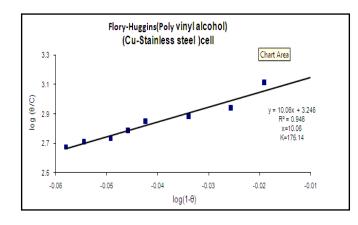
<b>Organic</b> Nano <b>Polymer</b>	Cu-C	'u cell	Cu-Sta	ninless l cell
	a	k	a	k
1) PEG-400	-13.85	311.17	-4.93	587.99
2) PEG-4000	-14.74	6408.63	-5.35	8174.82
3) PEG-6000	-15.33	9678.32	-6.76	2492.45
4) Poly vinyl alcohol	-8.10	5070.14	-12.23	74255.8 1

The characteristic of the **Temkin adsorption** isotherm is given by

$$Exp(-2a\theta) = KC$$

"a" is the lateral interaction parameter describing the molecular interaction in the adsorption layer and metal surface. Negative values of "a" indicates repulsion exists in the adsorption layer. It is generally known that  $\mathbf{K}$  denotes the strength between the adsorbate and adsorbent. Large values of  $\mathbf{K}$  refer to more efficient adsorption.

Florry-Huggins adsorption isotherm



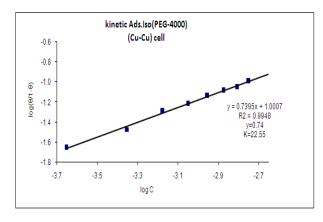
Organic Nano <b>Polyme</b> r	Cu-Cu cell		Cu-Stainless steel cell		
	X	k	X	k	
1) PEG-400	-	-	3.2	9.51	
2) PEG- 4000	7.82	12	3.6	105.87	
3) PEG- 6000	12.08	14.76	5.82	213.45	
4) Poly vinyl alcohol	4.03	320.63	10.06	175.14	

The equation of **Flory – Huggins** Isotherm

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

Where x is the number of active sites occupied by one inhibitor molecule. The values of x > 1 implied that one inhibitor molecule replace more than one water molecule (adsorbate).

### Kinetic adsorption isotherm



Organic Nano <b>Polyme</b> r	Cu-Cu cell		Cu-Stainless steel cell		
	y	k	y	k	
1) PEG-400	0.71	1.11	0.77	12.57	
2) PEG- 4000	0.74	22.55	0.76	25.13	
3) PEG- 6000	0.57	10.90	0.44	67.91	
4) Poly vinyl alcohol	0.77	505.09	0.58	133.28	

The kinetic -thermodynamic model is given by:

$$Log [\theta / (1-\theta)] = log K' + y log C$$

"y" is the number of inhibitor molecules occupying one active site. "y" values of the linear relation are less than unity, it means that the given additive molecule occupied more than one active site, The binding constant K is given by

$$\mathbf{K} = \mathbf{K}'^{(1/y)}$$

Large values of K mean better and strong interaction.

### The free energy of adsorption ( $\Delta G_{ads.}$ ) at different concentrations was calculated from the equation: $\Delta G_{ads.} = -RT \ ln \ (55.5 \ K)$

	Cu-Cu cell				Cu-Sta	
Organic	Timken	Flory- Huggins	Kinetic adsorption isotherm	Timken	Flory- Huggins	Kinetic adsorption isotherm
NanoPolymers	$\Delta \mathbf{G}_{\mathrm{ads}}$	$\Delta G_{ads}$				
1) PEG-400	-25.75	-15.53	-16.22	-24.17	-	-10.21
2) PEG-4000	-32.27	-21.50	-18.55	-31.67	-16.11	17.94
3) PEG-6000	-35.03	-23.24	-20.40	-32.69	-16.76	-15.86
4) Poly vinyl alcohol	-37.74	-22.75	-22.06	-36.79	-24.25	-25.37

- 1. The values of  $(\Delta G_{ads.})$  are negative means spontaneous adsorption of the inhibitor.
- 2. The most efficient inhibitor shows the most negative ( $\Delta$  G<sub>ads.</sub>) value. This suggests that they are strongly adsorbed and interacted with the metal surface.
- 3. The values of  $(\Delta G_{ads.})$  more positive than -40kJ/mol indicating physical adsorption that mean the adsorption of organic Nano Polymers take place through electrostatic interaction between the inhibitor molecule and the metal surface.

### **Effect of Temperatures and Thermodynamic Treatment**

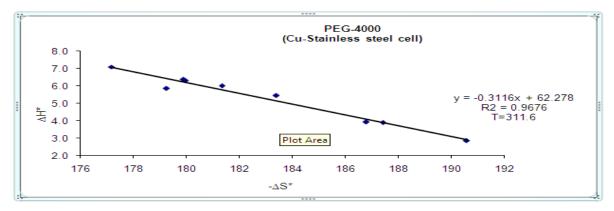
- 1. Positive sign for  $E_a$ , reflecting the endothermic nature of electrodeposition process and less than 43 kJ/mol indicating physical adsorption.
- 2. Positive sign for  $\Delta H^*$ , reflecting the endothermic nature of the adsorption process.

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- 3. The negative values of  $\Delta S^*$  pointed to a greater order produced during the process of activation.
- 4.  $\Delta G^*$  values of the inhibited systems were more positive than that for the uninhibited systems revealing that in cores of inhibitor addition the activated electrodeposition complex becomes less stable as compared to its absence.

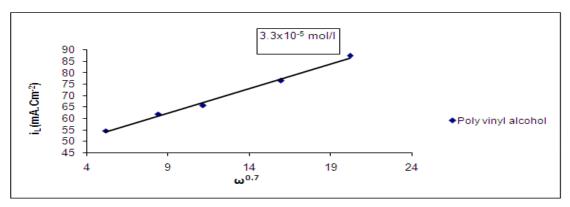
Omannia		Cu-Cu			Cu-Stainless steel				
Organic - NanoPolymer	C×10°	Ea	$\Delta$ H*	$\Delta S^*$	$\Delta G^*$	Ea	$\Delta H^*$	Δ <b>S*</b>	$\Delta G^*$
Namor orymer	mol/l	La				La			
	0	25.29	22.75	122.44	59.24	8.40	5.86	179.24	59.27
	28	24.58	22.04	124.85	59.25	4.93	2.39	191.21	59.37
	56	24.79	22.25	124.23	59.28	5.21	2.67	190.85	59.54
	84	24.59	22.06	124.95	59.29	5.33	2.79	190.63	59.59
PEG-400	112	24.87	22.33	124.13	59.32	6.63	4.09	186.79	59.76
	140	25.14	22.61	123.33	59.36	6.57	4.03	187.19	59.81
	168	25.46	22.93	122.37	59.40	6.89	4.35	186.40	59.89
	196	25.76	23.23	121.47	59.43	8.21	5.67	182.41	60.03
	224	25.80	23.26	121.41	59.44	7.43	4.89	185.24	60.09
	0	25.29	22.75	122.43	59.24	8.40	5.86	179.24	59.27
	2.5	25.09	22.55	123.27	59.29	6.46	3.92	186.79	59.59
	5	25.59	23.05	121.77	59.34	5.39	2.85	190.58	59.64
	7.5	26.30	23.76	119.58	59.40	6.42	3.88	187.42	59.73
PEG-4000	10	25.85	23.32	121.09	59.41	9.64	7.10	177.18	59.90
	12.5	26.29	23.76	119.76	59.45	8.84	6.30	179.95	59.93
	15	26.59	24.05	118.86	59.47	8.92	6.38	179.89	59.99
	17.5	27.22	24.68	116.89	59.52	8.55	6.01	181.34	60.05
	20	25.29	24.60	117.19	59.53	7.98	5.44	183.38	60.08
	0	25.29	22.75	122.43	59.24	8.40	5.86	179.24	59.27
	2.5	25.72	23.18	121.30	59.33	6.01	3.47	188.46	59.63
	5	26.07	23.70	119.74	59.38	3.97	1.43	195.45	59.67
	7.5	26.34	23.96	118.99	59.42	4.69	2.16	193.40	59.79
PEG-6000	10	26.39	24.07	118.75	59.46	7.78	5.24	183.50	59.92
	12.5	26.81	24.27	118.14	59.48	7.47	4.93	184.57	59.94
	15	27.02	24.48	117.56	59.52	7.71	5.17	184.07	60.02
	17.5	27.53	24.99	115.97	59.55	5.17	2.63	192.79	60.08
	20	28.40	25.87	113.21	59.61	5.12	2.58	193.02	60.10
	C×10°	Ea	$\Delta H^*$	$\Delta S^*$	$\Delta G^*$	Ea	$\Delta H^*$	$\Delta S^*$	$\Delta G^*$
	mol/l								
	0	25.29	22.75	122.44	59.24	8.40	5.86	179.24	59.27
Polyvinyl alcohol	3.33	25.66	22.04	121.44	59.31	13.69	11.15	162.44	59.55
	6.6	25.66	22.25	121.72	59.40	15.64	13.09	156.25	59.66
	9.9	25.66	22.06	116.14	59.50	16.37	13.83	154.05	59.74
	13.2	27.43	22.33	113.39	59.63	17.16	14.62	151.62	59.80
	16.5	28.37	22.61	107.56	59.74	18.94	16.40	145.98	59.90
<u> </u>	19.8	30.22	22.93	98.24	59.88	19.69	17.15	143.65	59.95
_	23.1	33.72	23.23	96.51	59.94	19.69	17.14	143.79	59.99
	26.4	34.30	23.26	94.68	59.88	19.94	17.39	143.06	60.03

### The Isokinetic Relationship



- **1.** Variation in the reaction rate within a reaction series may be caused by changes in either, or both, the enthalpy or the entropy of activation.
- 2.  $\delta \Delta H^* = \delta \Delta G^* + T \delta \Delta S^*$
- **3.** When  $\delta \Delta G^*$  equal zero,  $\beta$  equals **T**
- **4.** The slope in a linear plot of  $\Delta \mathbf{H}^*$  versus  $\Delta \mathbf{S}^*$  is the temperature at which all the reactions that confirm to the line occur at the same rate.  $\boldsymbol{\beta}$  is therefore known as the isokinetic temperature.

### **Effect Rotating Cylinder Electrode**



- 1. The angular velocity,  $\omega$ , is related to speed of rotation by :
- 2.  $\omega = 2\pi \text{ rpm} / 60$
- **3.** Straight lines were obtained and the limiting current density increases by increasing rotation for a cupper RCE, which indicates that the electrodeposition process of copper is **diffusion-controlled reaction.**

### **Diffusion Coefficient**

The diffusion coefficient of  $Cu^{++}$  ions, **D**, in different solutions was determined from the values of limiting current density using:

Eisenberg, Tobias and Wilke equation for rotating cylinder electrode (RCE):

$$i_L = knFC_b d^{\text{-0.3}} v^{\text{-0.344}} \ D^{\text{0.644}}_{\ U}^{\ 0.7}$$

Ī	$i_{L} =$	k	n	F	$C_{\mathbf{b}}$	$d^{-0.3}$	$v^{-0.344}$	$\mathbf{D}^{0.644}$	0.7 U

Limiting	Constnt	Bulk	Diameter of	Kinematic	Diffusion	Peripheral
current		Concentration	cylinder	viscosity	coefficient	velocity
density						

### **Dimensional Analysis**

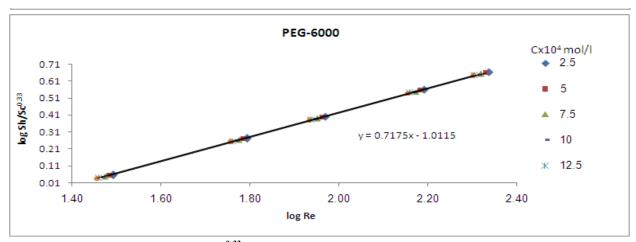
- 1. Dimensional analysis used to obtain an over all mass transfer correlation under the present conditions where a RCE were used.
- 2. To identify the variables, which affect the rate of mass transfer in the electrodeposition reaction, the mechanism of forced convection mass transfer shouldbe recalled first.
- 3. Forced convection takes place as a result of cylinder rotation. The thickness of the hydrodynamic boundary layer (Diffusion layer) at the rotating cylinder is determined by the physical properties of the solution, the geometry of the system (cylinder diameter) and cylinder rotation speed. This picture leads to the equation:

$$k=f(\rho, \eta, D, U, d)$$

4. The mass transport to an inner rotating cylinder electrode may be described by :

$$Sh = a Sc^{c} Re^{b}$$

Sh =	a	Sc	c	Re	ь
Sherwood number	intercept	Schmidt number	0.33	Reynolds number	slope
Sh=kd/D		Sc=v/D		Re=ud/v	



Overall correlation between logSh/Sc  $^{0.33}$  and logRefor PEG-6000 using cylinder electrode at 298 k.

The overall correlation for all additives used which correlated by the equations:

 $\log Sh/(Sc)^{0.33} = \log a + b \log Re$ 

Organic Nano Polymers	Copper cylinder
PEG-400	Sh= $0.0931 \text{ Re}^{0.7196} \text{ Sc}^{0.33}$ with an average deviation $\pm 0.072\%$
PEG-4000	Sh= $0.0966 \text{ Re}^{0.7183} \text{ Sc}^{0.33}$ with an average deviation $\pm 0.061\%$
PEG-6000	Sh= $0.0974 \text{ Re}^{0.7175} \text{ Sc}^{0.33}$ with an average deviation $\pm 0.073\%$

Poly vinyl alcohol	Sh= $0.098 \text{ Re}^{0.718} \text{ Sc}^{0.33}$ with an average deviation $\pm 0.059\%$
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The exponent denote the type of flow:

The exponent of Re is about 0.718 indicating turbulent flow of ions.

#### Conclusions

- 1. The values of inhibition efficiency of Cu deposition increase with increasing inhibitor concentrations.
- 2. Addition of organic Nano Polymers did not change the diffusion-controlled mechanism of Cu deposition to a charge transfer controlled mechanism.
- 3. The adsorption of all inhibitors on copper and Stainless steel cathodes obey Temkin, Flory–Huggins and kinetic adsorption isotherm except PEG-400 in case of Cu-Cu cell did not obey Flory–Huggins isotherm
- 4. The values of both Kads. and  $\Delta$ Gads. indicated that all studied inhibitors are strongly adsorbed on Stainless steel surface than copper surface.
- 5. The activation energy values for copper electrodeposition in the inhibited solutions are higher than that for the uninhibited solutions indicating good inhibitor characteristics associating with physical mechanism.

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