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# Thermodynamic Interaction Parameters for Bulk and Nano CuSO<sub>4</sub> with Cresol Red in Mixed DMF-H<sub>2</sub>O Solvents

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**Citation**

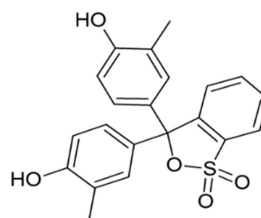
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**Abstract**

Characterization of nano CuSO<sub>4</sub> is done by transmission electron microscopy (TEM) and the thermodynamic parameters were evaluated by using conductometric measurements. The association and complex formation parameters of both bulk (normal) and nano-Cu SO<sub>4</sub> salts in DMF + H<sub>2</sub>O were calculated in presence of cresol red, non isothermally at different temperatures, 292.15, 303.15, 308.15 and 313.15K. The thermodynamics of the complexation reactions for bulk and nano CuSO<sub>4</sub> salts were compared.

**1. Introduction**

Nanoparticles are very important for medicine and environment, because of their increased behaviours. The increasing in the physical and chemical properties of nanomaterials are due to their high surface area and small volume, makes them very reactive, catalytic and able to pass through cell membranes. Copper sulphate is used in the treatment of some bacteria, algae, fungus and some fish parasites such as Ich. It can also be used to kill snails, accidentally or on purpose. The disadvantage of copper sulphate is that it is extremely toxic in water of low alkalinity [1].

**2. Experimental****2.1. Chemicals and Ligand**

Formula: C<sub>21</sub>H<sub>17</sub>NaO<sub>5</sub>S  
Molecular weight: 404.41 g.mol<sup>-1</sup>

Copper Sulfate (CuSO<sub>4</sub>.5H<sub>2</sub>O) was provided from Al Nasr chemicals Co. while nano-CuSO<sub>4</sub> salt was prepared by ball milling method using copper sulfate salt. The ball milling was a Retsch MM 2000 Swing mill with 10 cm<sup>3</sup> stainless steel, double-walled

tube. Two balls (stainless steel) with diameter of 12 mm were used. Ball milling was performed at 20225 Hz and shaking were used, usually at room temperature without circulating liquid and the temperature did not rise above 30 °C.

## 2.2. Experimental

### 2.2.1. Transmission Electron Microscopy (TEM) Images

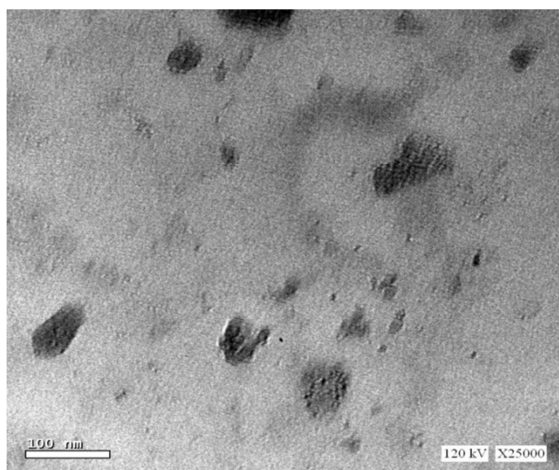
Transmission electron microscope is a special kind of electron microscope for imaging of different objects. The electrons interact with atoms of the sample. Due to this interaction the electrons are being scattered. The final image is very complicated interference pattern of incident and diffracted beams. The images were measured by using JEOL HRTEM – JEM 2100 (JAPAN) show that TEM of CuSO<sub>4</sub> obtained in water are diffused ones.

### 2.2.2. Conductometric Measurements

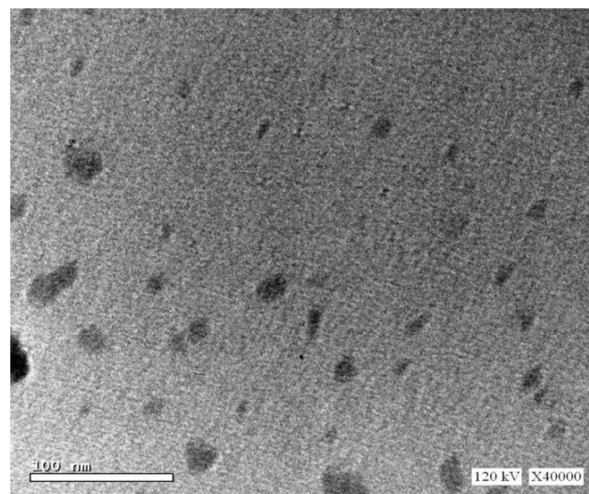
A solution of bulk and nano-CuSO<sub>4</sub> ( $1 \times 10^{-4}$  N) was placed in a titration cell, thermostated at a given temperature and the conductance of the solution was measured [2-21]. The ligand (Cresol red) ( $1 \times 10^{-3}$  N) was transferred step by step to the titration cell using a precalibrated micropipette and the conductance of the solution was measured after each transfer. The addition of the ligand solution was continued until the total concentration of the ligand was approximately four times higher than that of the metal ions. The conductance of the solution was measured by titration of ligand (cresol red) with metal salt solution. The complex formation constant  $K_f$ , and the molar conductance of the complex ML, were evaluated by computer fitting to the molar conductance mole ratio data. The temperatures used are (292.15, 303.15, 308.15 and 313.15K). The specific conductivity  $K_s$  was achieved by using a conductivity bridge of the type (JENCO – 3173 COND).

## 3. Data Results and Discussion

### 3.1. Transmission Electron Microscopy (TEM) Images



(a)



(b)

Fig. (1). TEM images of nano CuSO<sub>4</sub>

The photographs from (TEM) are presented for nano-CuSO<sub>4</sub> salt. The images show that the nano CuSO<sub>4</sub> in the form irregular spheres or deformed spheres, little diffusion with water solvent, the boundaries were seen and the sizes ranging from 20-40 nm.

## 3.2. Conductometric Results

The stability of a transition metal complex with ligand (Cresol red) depends on the number and the type of the donor atoms presented the number and the size of chelate rings formed on the complexation [21-25]. In addition, the stability and the selectivity of the complexities are strongly depend on the donor ability and the dielectric constant of the solvent [25-36], the shape, the size of the solvent molecules [37-45].

### 3.2.1. Calculation of Thermodynamic Parameters of Association for Bulk and Nano-CuSO<sub>4</sub> in DMF

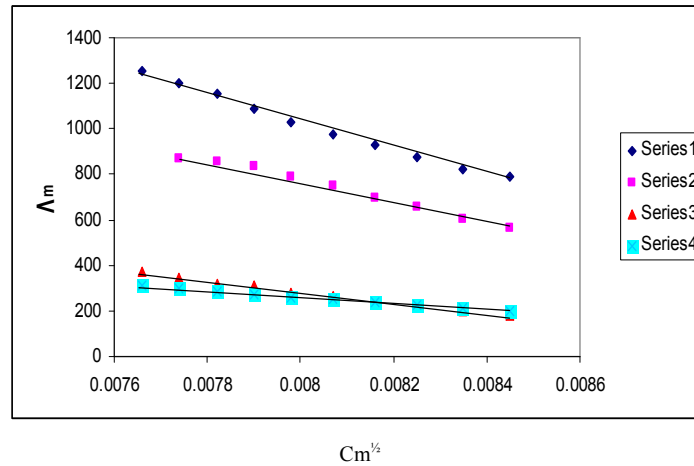
The specific conductance values ( $K_s$ ) of different concentrations for bulk and nano-CuSO<sub>4</sub> in DMF-H<sub>2</sub>O were measured experimentally in absence of ligand (cresol red) at different temperatures (292.15, 303.15, 308.15 and 313.15K). The molar conductance ( $\Lambda_m$ ) values were calculated [46-59] using equation (1):

$$\Lambda_m = \frac{(K_s - K_{solv}) \times K_{cell} \times 1000}{C} \quad (1)$$

Where  $K_s$  and  $K_{solv}$  are the specific conductance of the solution and the solvent, respectively;  $K_{cell}$  is the cell constant and  $C$  is the molar concentration of the bulk and nano-CuSO<sub>4</sub> solutions.

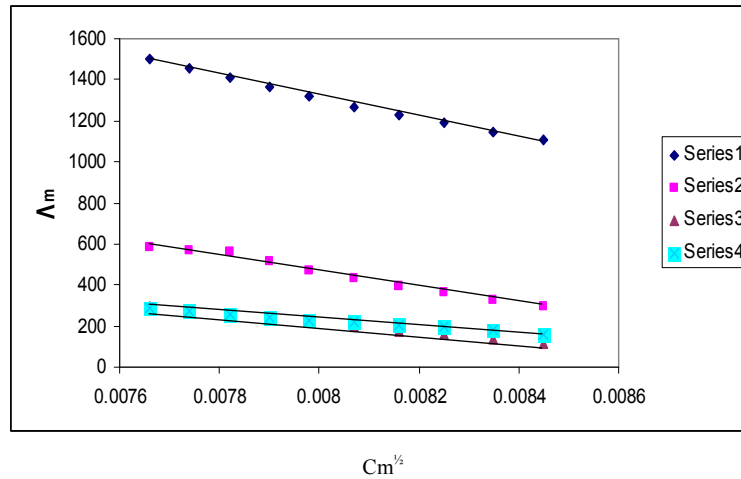
The limiting molar conductance ( $\Lambda_0$ ) at infinite dilutions was estimated for bulk and nano-CuSO<sub>4</sub> in DMF in absence of ligand at different temperatures by extrapolating the relation between  $\Lambda_m$  and  $C_m^{1/2}$  to zero concentration. The relation between  $\Lambda_m$  and  $C_m^{1/2}$  at 313.15K is shown in

Fig.(2)and Fig. (3).



**Fig. (2).** The relation between  $A_m$  and  $C_m^{1/2}$  for nano copper sulfate in presence of cresol red at 313.15K

Series 1: 20%DMF + 80% H<sub>2</sub>O  
 Series 2: 40%DMF + 60% H<sub>2</sub>O  
 Series 3: 60%DMF + 40% H<sub>2</sub>O  
 Series 4: 80%DMF + 20% H<sub>2</sub>O



**Fig. (3).** The relation between  $A_m$  and  $C_m^{1/2}$  for bulk copper sulfate in presence of cresol red at 313.15K

Series 1: 20%DMF + 80% H<sub>2</sub>O  
 Series 2: 40%DMF + 60% H<sub>2</sub>O  
 Series 3: 60%DMF + 40% H<sub>2</sub>O  
 Series 4: 80%DMF + 20% H<sub>2</sub>O

The values of association constants for nano and bulk CuSO<sub>4</sub> were calculated from equation (2) in presence of cresol red and tabulated in Table (1) and (2).

$$K_A = \frac{\Lambda_0 (\Lambda_0 - S(Z) \Lambda_m)}{C_m \Lambda_m^2 S(Z)^2 \gamma_{\pm}^2} \quad (2)$$

The values of free energy of association ( $\Delta G_A$ ) for bulk and nano-CuSO<sub>4</sub> in DMF at 313.15K were calculated [60-78] from the association constant ( $K_A$ ) values by using equation (3) and reported in Table (1) and (2).

$$\Delta G_A = -2.303 RT \log K_A \quad (3)$$

Where  $R$  is the gas constant and  $T$  is the absolute temperature.

The values of Gibbs free energies for the solutions of bulk and nano-CuSO<sub>4</sub> in DMF – H<sub>2</sub>O at 313.15K were calculated and reported in Table (1) and (2).

The dissociation degree ( $\alpha$ ) for bulk and nano-CuSO<sub>4</sub> in DMF in presence of cresol red at 313.15K were calculated from equation 4 and reported in Table (1) and (2).

$$\alpha = \Lambda_m / \Lambda_0 \quad (4)$$

**Table (1).** The association constant, free energies and degree of dissociation for bulk CuSO<sub>4</sub> at different concentration of DMF- H<sub>2</sub>O in presence of cresol red at 313.15K.

X <sub>s</sub> (DMF mole fraction)	C	C <sub>M</sub> <sup>1/2</sup>	Λ <sub>m</sub>	Λ <sub>0</sub>	α	log γ <sub>±</sub>	γ <sub>±</sub>	K <sub>A</sub>	ΔG <sub>A</sub> (kJ/mole)
0.0621	6.122 x 10 <sup>-5</sup>	0.0078	1412.937	4650	0.303	-0.0039	0.9911	1.253 x 10 <sup>5</sup>	-30.546
0.1346	6.122 x 10 <sup>-5</sup>	0.0078	560.274	3750	0.149	-0.0039	0.9911	6.336 x 10 <sup>5</sup>	-34.764
0.2595	6.122 x 10 <sup>-5</sup>	0.0078	261.352	1800	0.145	-0.0039	0.9911	6.742 x 10 <sup>5</sup>	-34.926
0.4831	6.122 x 10 <sup>-5</sup>	0.0078	259.719	1300	0.199	-0.0039	0.9911	3.333 x 10 <sup>5</sup>	-33.093

**Table (2).** The association constant, free energies and degree of dissociation for nano CuSO<sub>4</sub> at different concentration of DMF- H<sub>2</sub>O in presence of cresol red at 313.15K.

X <sub>s</sub> DMF mole fraction	C	C <sub>M</sub> <sup>1/2</sup>	Λ <sub>m</sub>	Λ <sub>0</sub>	α	log γ <sub>±</sub>	γ <sub>±</sub>	K <sub>A</sub>	ΔG <sub>A</sub> (kJ/mole)
0.0621	6.122 x 10 <sup>-5</sup>	0.0078	1153.218	4850	0.238	-0.0039	0.9911	2.241x 10 <sup>5</sup>	-32.060
0.1346	6.122 x 10 <sup>-5</sup>	0.0078	854.296	4000	0.213	-0.0039	0.9911	2.867x 10 <sup>5</sup>	-32.700
0.2595	6.122 x 10 <sup>-5</sup>	0.0078	321.790	1700	0.189	-0.0039	0.9911	3.762x 10 <sup>5</sup>	-33.408
0.4831	6.122 x 10 <sup>-5</sup>	0.0078	282.587	1050	0.269	-0.0039	0.9911	1.677x 10 <sup>5</sup>	-31.306

### 3.2.2. Calculation of Thermodynamic Parameters of Complex Formation for Bulk and Nano-CuSO<sub>4</sub> with Cresol red in DMF

The specific conductance values (K<sub>s</sub>) of different concentrations of bulk and nano-CuSO<sub>4</sub> in DMF were measured experimentally in the presence of ligand at different temperatures (292.15, 303.15, 308.15 and 313.15 K). The molar conductance (Λ<sub>m</sub>) values were calculated [21-70] using equation (1).

By drawing the relation between molar conductance (Λ<sub>m</sub>) for bulk and nano-CuSO<sub>4</sub> in presence of ligand at different temperatures and the molar ratio of metal to ligand [M]/[L] concentrations, Fig.(3) different lines are obtained with breaks indicating the formation of 1:2 and 1:1 (M:L) stoichiometric complexes ,as done in previous works [7-75].

The formation constants (K<sub>f</sub>) for bulk and nano-CuSO<sub>4</sub> complexes were calculated for each type of complexes (1:2)

and (1:1) (M: L) by using equation (5) [75-78]:

$$K_f = \frac{\Lambda_m - \Lambda_{obs}}{(\Lambda_{obs} - \Lambda_{ML})[L]} \quad (5)$$

Where Λ<sub>m</sub> is the limiting molar conductance of the bulk and nano-Cu SO<sub>4</sub> alone, Λ<sub>obs</sub> is the molar conductance of solution during titration, Λ<sub>ML</sub> is the molar conductance of the complex and [L] is the ligand concentration.

The Gibbs free energies of formation for each stoichiometry complex (ΔG<sub>f</sub>) were calculated by using the equation (6) [10-25]:

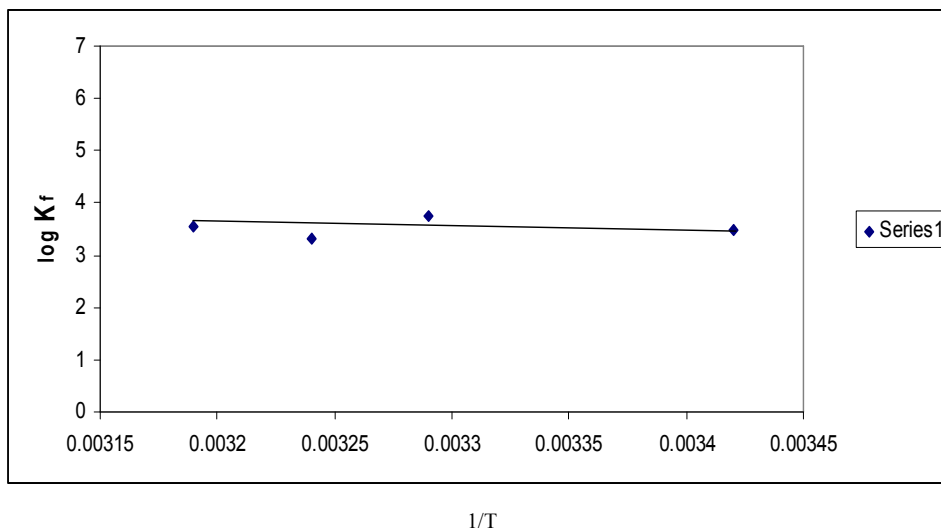
$$\Delta G_f = - 2.303 RT \log K_f \quad (6)$$

The obtained values (K<sub>f</sub>) for bulk and nano-CuSO<sub>4</sub>stoichiometry complexes and their calculated ΔG<sub>f</sub> values at 313.15K are presented in Tables.3 and 4 respectively.

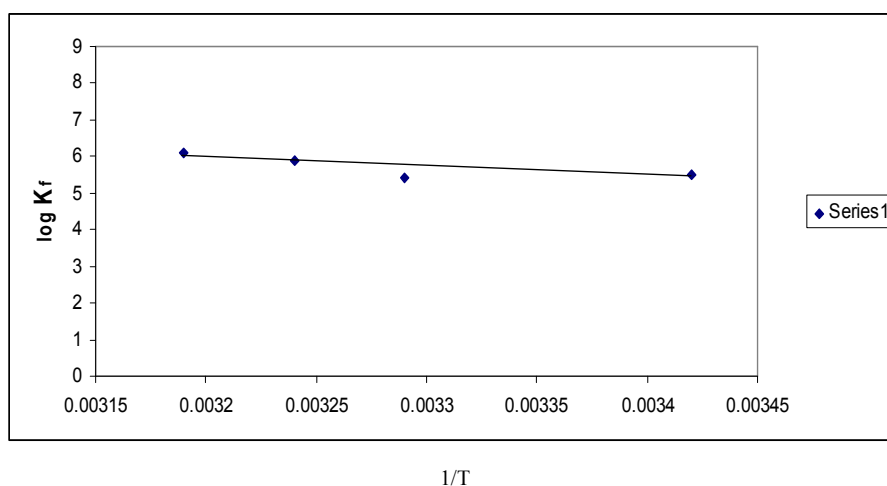
**Table (3).** The formation constants and Gibbs free energies of formation for bulk CuSO<sub>4</sub>in presence of cresol red at different concentration of DMF- H<sub>2</sub>O at 313.15K:

X <sub>s</sub> (Vol % DMF)	M/L	[L]	Λ <sub>obs</sub>	Λ <sub>ML</sub>	Λ <sub>M</sub>	K <sub>f</sub>	ΔG <sub>f</sub> (kJ/mole)
0.0621	1:2	4.000 x 10 <sup>-4</sup>	1453.333	930	2190	3.519x 10 <sup>3</sup>	-21.250
		4.117 x 10 <sup>-4</sup>	1501.190	930	2190	2.929x 10 <sup>3</sup>	-20.772
		4.231 x 10 <sup>-4</sup>	1530.595	930	2190	2.594x 10 <sup>3</sup>	-20.456
		1.176 x 10 <sup>-4</sup>	848.918	840	2190	1.278734x 10 <sup>6</sup>	-36.591
	1:1	1.428 x 10 <sup>-4</sup>	864.543	840	2190	3.78189x 10 <sup>5</sup>	-33.421
0.1346	1:2	1.667 x 10 <sup>-4</sup>	903.636	840	2190	1.21262x 10 <sup>5</sup>	-30.461
		3.617 x 10 <sup>-4</sup>	473.132	130	1050	4.648 x 10 <sup>3</sup>	-21.974
		3.750 x 10 <sup>-4</sup>	513.600	130	1050	3.728 x 10 <sup>3</sup>	-21.400
		3.877 x 10 <sup>-4</sup>	560.274	130	1050	2.935 x 10 <sup>3</sup>	-20.777
	1:1	1.176 x 10 <sup>-4</sup>	69.137	60	1050	9.12845 x 10 <sup>5</sup>	-35.714
		1.428 x 10 <sup>-4</sup>	100.338	60	1050	1.64864 x 10 <sup>5</sup>	-31.260
		1.667 x 10 <sup>-4</sup>	110.404	60	1050	1.11825 x 10 <sup>5</sup>	-30.250
		4.000 x 10 <sup>-4</sup>	280.000	60	680	4.545 x 10 <sup>3</sup>	-21.915
0.2595	1:2	4.117 x 10 <sup>-4</sup>	297.518	60	680	3.911 x 10 <sup>3</sup>	-21.524
		4.231 x 10 <sup>-4</sup>	329.346	60	680	3.076 x 10 <sup>3</sup>	-20.899
		1.176 x 10 <sup>-4</sup>	14.734	12	680	2.069138 x 10 <sup>6</sup>	-37.843
		1.428 x 10 <sup>-4</sup>	18.667	12	680	6.94642 x 10 <sup>5</sup>	-35.003
	1:1	1.667 x 10 <sup>-4</sup>	22.801	12	680	3.65003 x 10 <sup>5</sup>	-33.329
0.4831	1:2	4.000 x 10 <sup>-4</sup>	273.333	125	460	3.146x 10 <sup>3</sup>	-20.958
		4.117 x 10 <sup>-4</sup>	289.017	125	460	2.532x 10 <sup>3</sup>	-20.393
		4.231 x 10 <sup>-4</sup>	294.678	125	460	2.302x 10 <sup>3</sup>	-20.145
		1.176 x 10 <sup>-4</sup>	82.738	68	460	2.17669x 10 <sup>5</sup>	-31.983
	1:1	1.428 x 10 <sup>-4</sup>	89.838	68	460	1.18700x 10 <sup>5</sup>	-30.405
		1.667 x 10 <sup>-4</sup>	100.804	68	460	6.5685x 10 <sup>4</sup>	-28.866

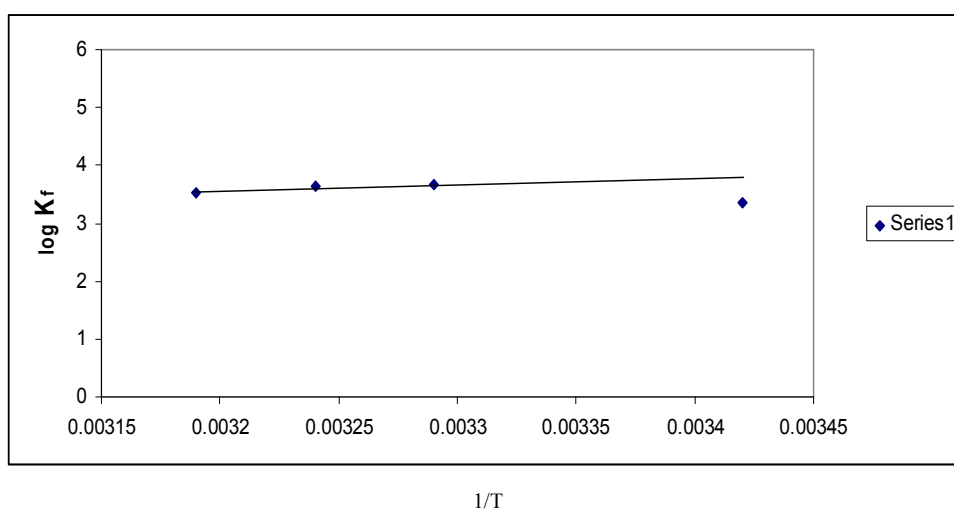
By drawing the relation between  $\log K_f$  and  $1/T$ , different (M:L) stoichiometry complexes Fig.( 4,5,6 and 7) as lines are obtained indicating the formation of 1:2 and 1:1 explained in previous works [26-60].



**Fig. (4).** The relation between  $\log K_f$  and  $1/T$  for 1:2 complex ratio of bulk  $\text{CuSO}_4$  in presence of cresol red.



**Fig. (5).** The relation between  $\log K_f$  and  $1/T$  for 1:1 complex ratio of bulk  $\text{CuSO}_4$  in presence of cresol red



**Fig. (6).** The relation between  $\log K_f$  and  $1/T$  for 1: 2 complex ratio of nano  $\text{CuSO}_4$

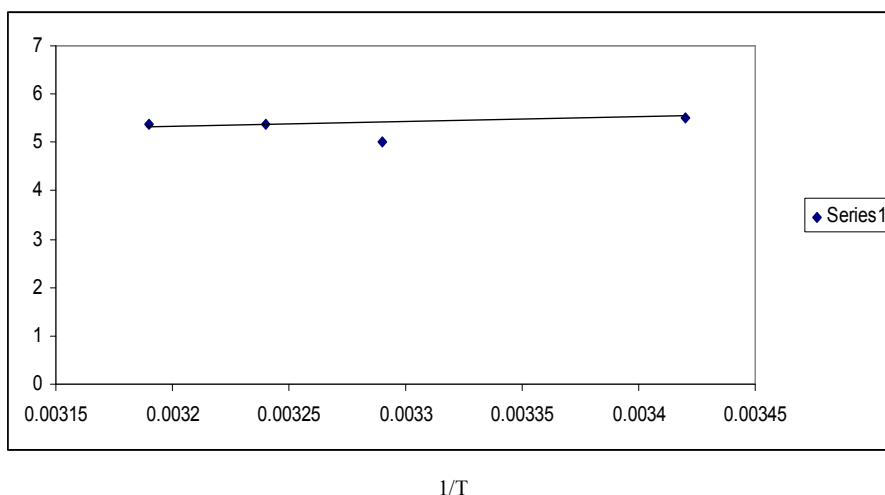


Fig. (7). The relation between  $\log K_f$  and  $1/T$  for 1: 1 complex ratio of nano CuSO<sub>4</sub> in presence of cresol red

From the relation between  $\log K_f$  and  $1/T$ ,  $\Delta H_f$  can be calculated for each type of complexes, from the slope of each line which equal  $(-\Delta H_f/2.303R)$ . The entropy ( $\Delta S_f$ ) for bulk and nano-CuSO<sub>4</sub> stoichiometric complexes were calculated [32, 33] for each type of complexes (1:2) and (1:1) (M:L) by using by using Gibbs-Helmholtz equation (7) [44-50]:

$$\Delta G_f = \Delta H_f - T\Delta S_f \quad (7)$$

The calculated values of ( $\Delta H_f$ ), ( $T\Delta S_f$ ) and ( $\Delta S_f$ ) for bulk and nano-CuSO<sub>4</sub> at 313.15K stoichiometric complexes are presented in Table (4) and (5) respectively.

Table (4). The values of ( $\Delta H_f$ ), ( $T\Delta S_f$ ) and ( $\Delta S_f$ ) in (kJ/mole) for bulk CuSO<sub>4</sub> in presence of Cresol red at 313.15K

Complex ratio	$\Delta H$	$T\Delta S$	$\Delta S$
1:2	6.157	27.407	0.087
1:1	40.083	76.674	0.245

Table (5). The values of ( $\Delta H_f$ ), ( $T\Delta S_f$ ) and ( $\Delta S_f$ ) in (kJ/mole) for nano CuSO<sub>4</sub> at 313.15K

Complex ratio	$\Delta H$	$T\Delta S$	$\Delta S$
1:2	-28.898	-7.790	-0.025
1:1	-14.247	20.799	0.066

It was found that irregular spheres are seen in TEM images for nano CuSO<sub>4</sub> which explain that the association of nano CuSO<sub>4</sub> is small in solution phase. If the association is big the boundaries of the particles between solvent and salt can not be found. Therefore the association thermodynamic parameters for bulk CuSO<sub>4</sub> is bigger than nano CuSO<sub>4</sub> in presence of cresol red.

The association constants and free energies of association for both bulk and nano CuSO<sub>4</sub> are bigger than complex formation in presence of cresol red because both complexation and association are proceeded which seen in association parameter.

The entropies of complex formation and degree of ionization are bigger in case of bulk CuSO<sub>4</sub> than that of nano CuSO<sub>4</sub> which support the above explanation.

## 4. Conclusion

The association and complex formation parameters for bulk CuSO<sub>4</sub> are bigger than that of nano CuSO<sub>4</sub> because of the bigger in both entropies and dissociation degrees for the former. Comparison between the nano and bulk electrolyte thermodynamics are very important to discuss their behaviors in solutions. The aim of this work is to give & study the properties of some nano salts like CuSO<sub>4</sub> that can help in determination easily and fast.

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