Engineering and Technology
2015; 2(2): 74-80
Published online April 10, 2015 (http://www.aascit.org/journal/et)

American Association for Science and Technology

Keywords
Thermodynamics,
Molar Conductance,
Nano Copper Sulfate and Bulk Copper Sulfate,
Bromophenol Blue and Mixed Solvents

Received: March 14, 2015
Revised: April 1, 2015
Accepted: April 2, 2015

Non Isothermal Thermodynamic Interaction Parameters of Bulk and Nano $CuSO_4$ with Bromophenol Blue (BPB)

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Citation
Mohamed N. H. Hamed, Esam A. Gomaa, Sameh G. Sanad. Non Isothermal Thermodynamic Interaction Parameters of Bulk and Nano $CuSO_4$ with Bromophenol Blue (BPB). *Engineering and Technology.* Vol. 2, No. 2, 2015, pp. 74-80.

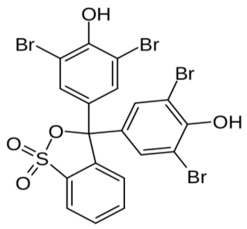
Abstract
Characterization of the prepared nano$CuSO_4$ is done by using transmission electron microscopy (TEM) and its thermodynamic parameters were determined using conductometric measurements. The association and complex formation parameters for both bulk (normal) and nano-$CuSO_4$ salts in dimethylformamide (DMF) and water mixtures were calculated in presence of bromophenol blue (BPB), non isothermally at different temperatures, 292.15 , 303.15 , 308.15 and 313.15K.The thermodynamics of the complexation reactions for bulk and nano $CuSO_4$ salts were compared.

1. Introduction

Nanoparticles are very important for medicine and environment, because of their increased applications. The increasing in the physical and chemical properties of nanomaterials are due to theirs 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 at low alkalinity [1].

2. Experimental

2.1. Chemicals and Ligand



Formula and Molecular weight

Formula	$C_{19}H_{10}Br_4O_5S$
Molecular weight	669.96 g mol^{-1}

Copper Sulfate ($CuSO_4.5H_2O$) was provided from Al Nasr chemicals Co. while nano-Cu SO_4 salt was prepared by ball milling of the same copper sulfate salt. The ball milling was a Retsch MM 2000 Swing mill with 10 cm^3 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 (303.15K).

Bromophenol blue (BPB) was used as purchased from Merck co.

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. In contrast to other microscopes the electrons in TEM pass through and 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 $CuSO_4$ obtained in water are diffused ones.

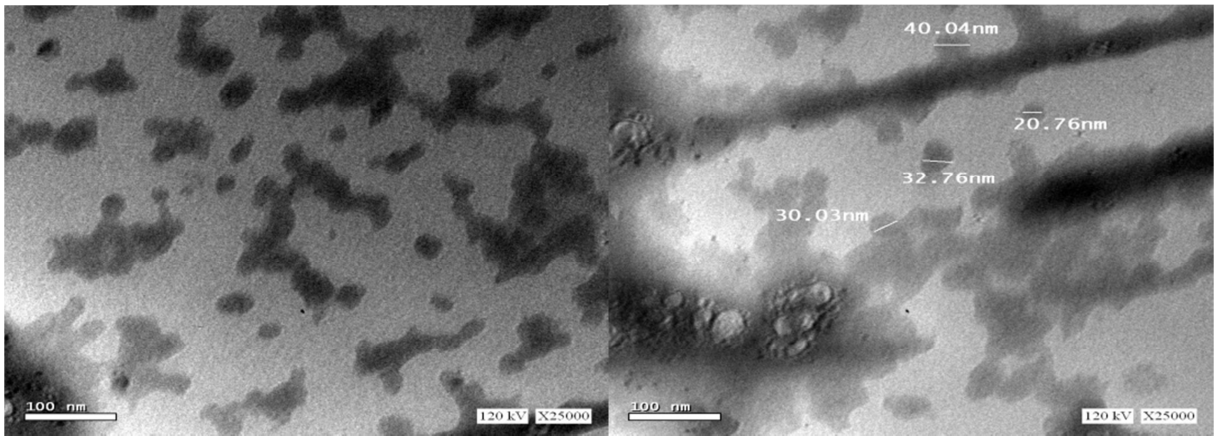
2.2.2. Condutometric Measurements

A solution of bulk and nano-$CuSO_4$ ($1x10^{-4}$ N) was placed in a titration cell, thermostated at a given temperature and the conductance of the solution was measured [2-21] by titration with ligand ,the cell constant is one. The ligand BPB ($1X10^{-3}$ N) (bromophenol blue) was transferred step by step to the titration cell using a precaliberated micropipette with 0.2 ml additions of ligand 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 (BPB) with metal salt. 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)The conductivity method is cheap one method.

3. Data Results and Discussion

3.1. Transmission Electron Microscopy (TEM) Images

The photographs from (TEM) are presented for nano-$CuSO_4$ salt.The images show that the nano $CuSO_4$ in the form irregular spheres, little diffusion with water solvent and the sizes ranging from 20-40 nm.



(a) (b)

Fig. (1). *TEM images of nano $CuSO_4$*

3.2. Conductometric Results

The stability of a transition metal complex with ligand depends on a range of factors including 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-$CuSO_4$ in DMF

The specific conductance values (K_s) of different concentrations for bulk and nano-$CuSO_4$ in DMF were measured experimentally in absence of ligand (BPB) at different temperatures (292.15, 303.15, 308.15 and 313.15K). The molar conductance (Λ_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; Kcell is the cell constant and C is the molar concentration of the bulk and nano-$CuSO_4$ solutions.

The limiting molar conductance (Λ_o) at infinite dilutions was estimated for bulk and nano-$CuSO_4$ in DMF in absence of ligand at different temperatures by extrapolating the relation between Λ_m and $C_m^{1/2}$ to zero concentration. The relation between Λ_m and $C_m^{1/2}$ at 313.15K is shown in Fig.(2) and Fig. (3).

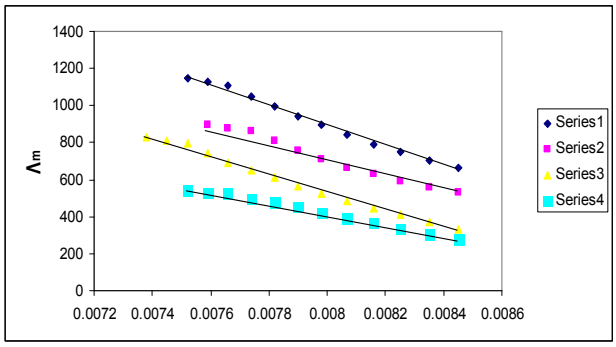


Fig. (2). *The relation between Λ_m and $Cm^{1/2}$ for nano copper sulfate in presence of bromophenol blue (BPB) at 313.15K*

Series 1: 20%DMF + 80% H_2O
Series 2: 40%DMF + 60% H_2O
Series 3: 60%DMF + 40% H_2O
Series 4: 80%DMF + 20% H_2O

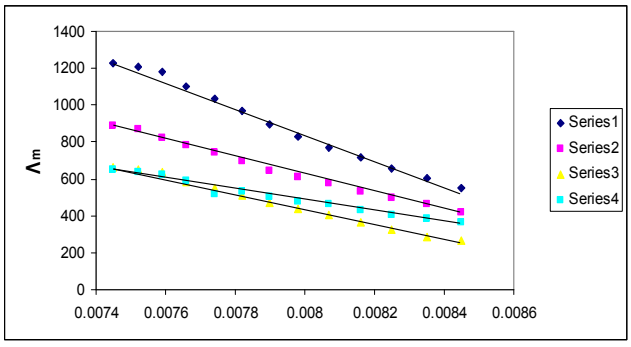


Fig. (3). *The relation between Λ_m and $Cm^{1/2}$ for bulk copper sulfate in presence of bromophenol blue (BPB) at 313.15K*

Series 1: 20%DMF + 80% H_2O
Series 2: 40%DMF + 60% H_2O
Series 3: 60%DMF + 40% H_2O
Series 4: 80%DMF + 20% H_2O

The values of association constants for nano and bulk $CuSO_4$ were calculated from equation (2) in presence of bromophenol blue(BPB)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 (ΔGA) for bulk and nano-$CuSO_4$ in DMF at 313.15K were calculated [60-78] from the association constant (KA) 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-$CuSO_4$ in DMF – H_2O at 313.15K were calculated and reported in Table (1) and (2).

The dissociation degree (α) for bulk and nano-$CuSO_4$ in DMF in presence of bromophenol blue (BPB) 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 , freeenergies and degree of dissociation for bulk $CuSO_4$ at different concentration of DMF- H_2O in presence of bromophenol blue (BPB) at 313.15K .*

X_s(DMF mole fraction)	C	$C_M^{1/2}$	Λ_m	Λ_0	α	log $\gamma_\pm$	$\gamma_\pm$	K_A	$\Delta G_{A(k.J/mole)}$
0.0621	6.122×10^{-5}	0.0078	971.904	6200	0.157	-0.0039	0.9911	5.706×10^5	-34.491
0.1346	6.122×10^{-5}	0.0078	782.424	4230	0.185	-0.0039	0.9911	3.961×10^5	-33.542
0.2595	6.122×10^{-5}	0.0078	638.680	3325	0.192	-0.0039	0.9911	3.641×10^5	-33.322
0.4831	6.122×10^{-5}	0.0078	532.506	2850	0.187	-0.0039	0.9911	3.873×10^5	-33.483

Table (2).*The association constant, free energies and degree of dissociation for nano $CuSO_4$ at different concentration of DMF- H_2O in presence of bromophenol blue (BPB) at 313.15K .*

X_sDMF mole fraction	C	$C_M^{1/2}$	Λ_m	Λ_0	α	log $\gamma_\pm$	$\gamma_\pm$	K_A	$\Delta G_{A(k.J/mole)}$
0.0621	6.122×10^{-5}	0.0078	998.039	4700	0.212	-0.0039	0.9911	2.904×10^5	-32.734
0.1346	6.122×10^{-5}	0.0078	806.926	4400	0.183	-0.0039	0.9911	4.037×10^5	-33.589
0.2595	6.122×10^{-5}	0.0078	607.644	3920	0.155	-0.0039	0.9911	5.847×10^5	-34.567
0.4831	6.122×10^{-5}	0.0078	478.601	2880	0.166	-0.0039	0.9911	5.021×10^5	-34.158

3.2.2. Calculation of Thermodynamic Parameters of Complex Formation for Bulk and Nano-$CuSO_4$ with BromophenolBlue (BPB) in DMF

The specific conductance values (K_s) of different concentrations of bulk and nano-$CuSO_4$ 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_m) values were calculated [21-70] using equation (1).

By drawing the relation between molar conductance (Λ_m) for bulk and nano-$CuSO_4$ 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_f) for bulk and nano-$CuSO_4$complexes were calculated for each type of complex (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_m is the limiting molar conductance of the bulk and nano-Cu SO_4 alone, Λ_{obs} is the molar conductance of solution during titration,Λ_{ML} is the molar conductance of the complex and [L] is the ligand concentration.

The Gibbs free energies of formation for each stoichiometry complex (ΔG_f) were calculated by using equation (6) [10-25]:

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

The obtained values (K_f) for bulk and nano-$CuSO_4$ stoichiometry complexes and their calculated ΔG_f 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 $CuSO_4$in presence of bromophenol blue (BPB) at different concentration of DMF- H_2O at 313.15K .*

X_s(Vol % DMF)	M/L	[L]	$\Lambda_{obs.}$	Λ_{ML}	Λ_M	K_f	$\Delta G_{f(k.J/mole)}$
0.0621	1:2	4.000×10^{-4}	1033.333	400	2150	4.407×10^3	-21.835
		4.117×10^{-4}	1101.666	400	2150	3.473×10^3	-21.215
		4.231×10^{-4}	1183.914	400	2150	2.912×10^3	-20.757
	1:1	1.176×10^{-4}	168.877	55	2150	1.47934×10^5	-30.978
		1.428×10^{-4}	215.844	55	2150	8.4208×10^4	-29.512
		1.667×10^{-4}	255.610	55	2150	5.6647×10^4	-28.480
0.1346	1:2	4.000×10^{-4}	821.667	340	1460	3.313×10^3	-21.093
		4.117×10^{-4}	870.452	340	1460	2.699×10^3	-20.559
		4.231×10^{-4}	887.502	340	1460	2.471×10^3	-20.329
	1:1	1.176×10^{-4}	193.812	100	1460	1.14771×10^5	-30.318
		1.428×10^{-4}	248.512	100	1460	5.7125×10^4	-28.502
		1.667×10^{-4}	278.411	100	1460	3.9729×10^4	-27.557
0.2595	1:2	3.478×10^{-4}	512.113	180	1100	5.089×10^3	-22.210
		3.617×10^{-4}	551.465	180	1100	4.082×10^3	-21.636
		3.750×10^{-4}	585.600	180	1100	3.381×10^3	-21.145
	1:1	1.176×10^{-4}	128.074	68	1100	1.37574×10^5	-30.789
		1.428×10^{-4}	155.174	68	1100	7.5899×10^4	-29.242
		1.667×10^{-4}	180.007	68	1100	4.9272×10^4	-28.117
0.4831	1:2	4.000×10^{-4}	516.667	260	1100	5.681×10^3	-22.496
		4.117×10^{-4}	593.335	260	1100	3.691×10^3	-21.374
		4.231×10^{-4}	621.118	260	1100	3.134×10^3	-20.948
	1:1	1.176×10^{-4}	174.544	123	1100	1.52675×10^5	-31.060
		1.428×10^{-4}	205.343	123	1100	7.6085×10^4	-29.248
		1.667×10^{-4}	222.009	123	1100	5.3196×10^4	-28.317

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

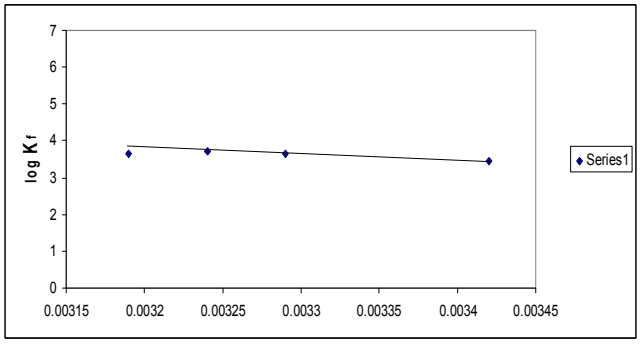


Fig. (4). *The relation between log Kf and 1/T for 1:2 complex ratio of bulk $CuSO_4$ in presence of bromophenol blue (BPB).*

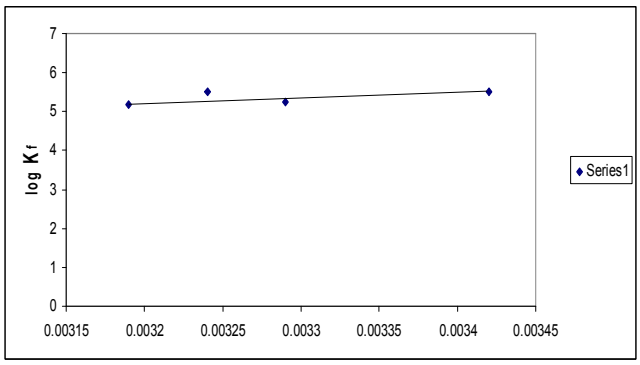


Fig. (5). *The relation between log Kf and 1/T for 1:1 complex ratio of bulk $CuSO_4$ in presence of bromophenol blue (BPB)*

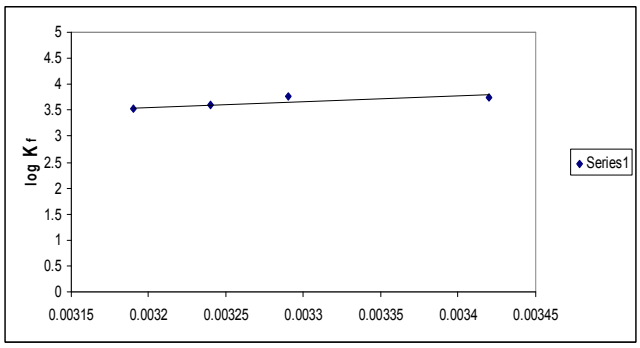


Fig. (6). *The relation between log Kf and 1/T for 1: 2 complex ratio of nano $CuSO_4$ in presence of bromophenol blue (BPB)*

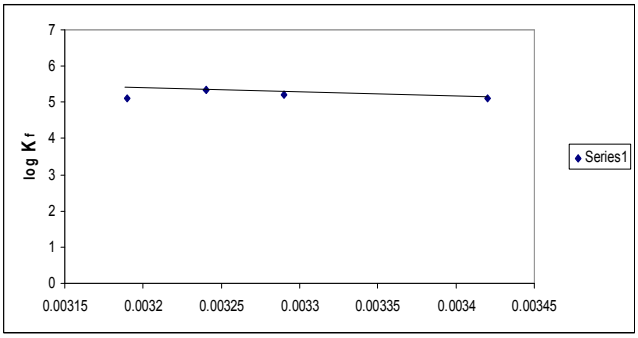


Fig. (7). *The relation between log K_f and 1/T for 1: 1 complex ratio of nano $CuSO_4$in presence of bromophenol blue (BPB)*

From the relation between log $K_f$and 1/T, ΔH_fcan be calculated for each type of complexes, from the slope of each line which equal ($-\Delta H_f/2.303R$). The entropy (ΔS_f) for bulk and nano-$CuSO_4$ stoichiometry 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 (ΔH_f), ($T\Delta S_f$) and (ΔS_f) for bulk and nano-$CuSO_4$ at 3131.15K stoichiometric complexes are presented in Table (4) and (5) respectively.

Table (4). *The values of (ΔH_f) , ($T\Delta S_f$) and (ΔS_f) in (k.J/mole)for bulk $CuSO_4$ in presence of bromophenol blue (BPB) at 3131.15K*

Complex ratio	ΔH	TΔS	ΔS
1:2	28.919	50.754	0.162
1:1	-27.459	3.519	0.011

Table (5). *The values of (ΔH_f) , ($T\Delta S_f$) and (ΔS_f) in (k.J/mole) for nano $CuSO_4$ at 313.15K*

Complex ratio	ΔH	TΔS	ΔS
1:2	-16.054	5.046	0.016
1:1	45.165	75.806	0.242

It was found that irregular spheres are seen in TEM images for nano $CuSO_4$which explain that the association of nano $CuSO_4$ is small in solution phase. If the association is big the boundaries of the particles can not be found. Therefore the association thermodynamic parameters for bulk $CuSO_4$ is bigger than nano $CuSO_4$ in presence of bromophenol blue (BPB).

The association constants and free energies of association for both bulk and nano $CuSO_4$are bigger for complex formation in presence of bromophenol blue (BPB) because complexation and association are proceed which seen in association parameters.

The entropies of complex formation and degree of ionization are bigger in case of bulk $CuSO_4$ than that of nano $CuSO_4$ which support the above explanation.

4. Conclusion

The association and complex formation parameters for bulk$CuSO_4$ are bigger than that of nano $CuSO_4$ because of the bigger in both entropies and dissociation degrees for the former. Comparison between the nano and bulk electrolyte thermodynamics needed. The aim of work is to evaluate copper sulphate in different media by using easy and accurate method.

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