

## Research Article

# Conductance Studies on Complex Formation between Nano Zinc Sulphate and Calcon Carboxylic Acid in Methanol (MeOH) – Water at Different Temperatures

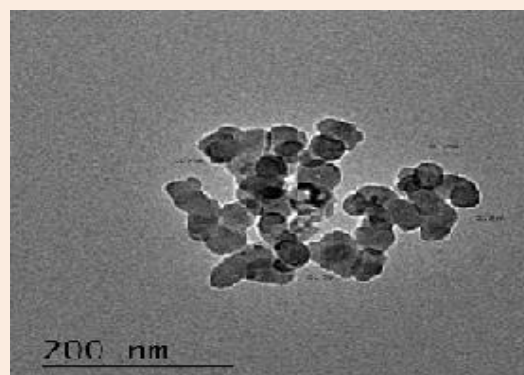
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## Abstract

This article summarizes different aspects of the complexes of the nano ZnSO<sub>4</sub> with ligand Calcon carboxylic acid (Calcon CA) as studied conductometrically in methanol-water solvent at 298.15, 303.15, 308.15 and 313.15K by applying the conductometric method. Consequently, we will study the effect of solvent properties, (MeOH/H<sub>2</sub>O) on stoichiometry, the selectivity between ligand and ions in various systems and thermodynamic parameters of complexation. On drawing the relation between molar conductance and the ratio of metal to ligand concentrations, different lines are obtained indicating the formation of 1:2, and 1:1 (M:L) stoichiometry complexes. The stability constant of the complexes were obtained from fitting the molar conductivity curves using a computer program. This research focused on the study of thermodynamic complexation reactions between the ligand, Calcon CA, with Zn<sup>2+</sup> metal cation in aqueous methanol solvent.



**Keywords:** Conductance, nano zinc sulphate, complex formation, calcon carboxylic acid

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## Introduction

Studies on electrolyte conductance of an electrolyte and the effect of ion – solvation on it in aqueous and partially aqueous media have received considerable attention in recent years as they are important both from fundamental and technological points of view. The use of non aqueous and partially aqueous solvents has been widely accepted, in place of water due to their wide applicability in various fields. It has become a practice to use solvent mixtures, water being one among the solvent mixtures. The solvent mixtures not only give an idea about ion- solvent and solvent – solvent – interactions but also the preferential solvation of ion [1-5]. Nano structured materials have been fascinating the world of science and technology during the last 15 years because of their tremendous possibilities in generating novel shapes, structures, and the unusual phenomena associated with these materials. The characteristic length of these materials (at least one length) lies between 1–100 nm. This makes the properties of matter within this length scale significantly different from individual atoms or molecules and from bulk materials. Nano material of zinc sulphate was produced by ball milling method. It can be characterized by SEM, TEM and XRD.

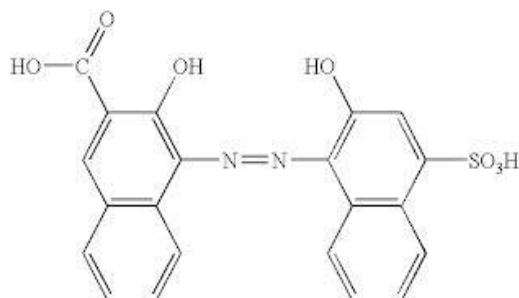
## Experimental

### Materials and Reagents

All chemicals used were of the highest purity available and were purchased from Merck.

**The ligand**

Molecular Structure:

Molecular Formula:  $C_{21}H_{14}N_2O_7S$ **Preparation of nano zinc sulphate**

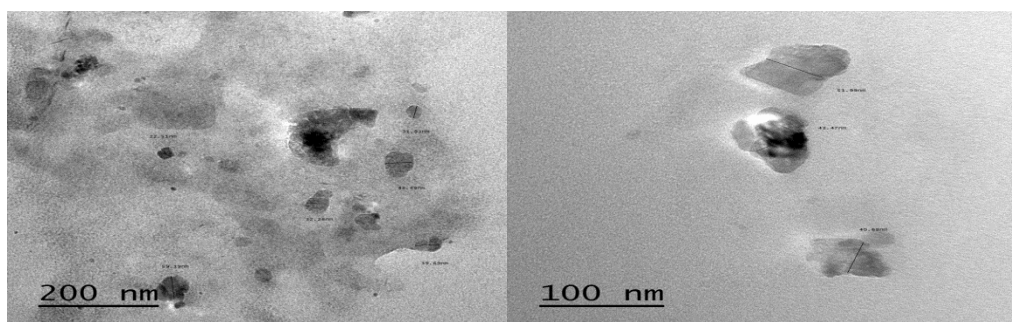
The nano nickel sulphate was prepared by shaking zinc sulphate in ball-mill apparatus of the type Retsch MM2000 swing mill for a period of one hour. The mill has a 10 cm<sup>3</sup> stainless steel tube. Two stainless steel balls of 12 mm diameter were used. Ball milling was performed at 20225 Hz at room temperature.

**Conductometric titration**

In a typical experiment, 5 ml of the Calcon CA solution ( $5.0 \times 10^{-3} M$ ) was placed in the titration cell, thermostated at the preset temperature and the conductance of the solution was measured after the solution reached thermal equilibrium. Then, a known amount of the  $ZnSO_4 \cdot 7H_2O$  solution ( $1.0 \times 10^{-3} M$ ) was added in a stepwise manner using a calibrated micropipette. The conductance of the solution was measured after each addition until the desired cations-to-ligand mole ratio was achieved. The specific conductance values were recorded using conductivity bridge HANNA, H1 8819N with a cell constant equal to 1. The conductometer was conducted with a thermostat of the type the Kottermann 4130 ultra thermostat. The temperature was adjusted at 298.15, 303.15, 308.15 and 313.15K.

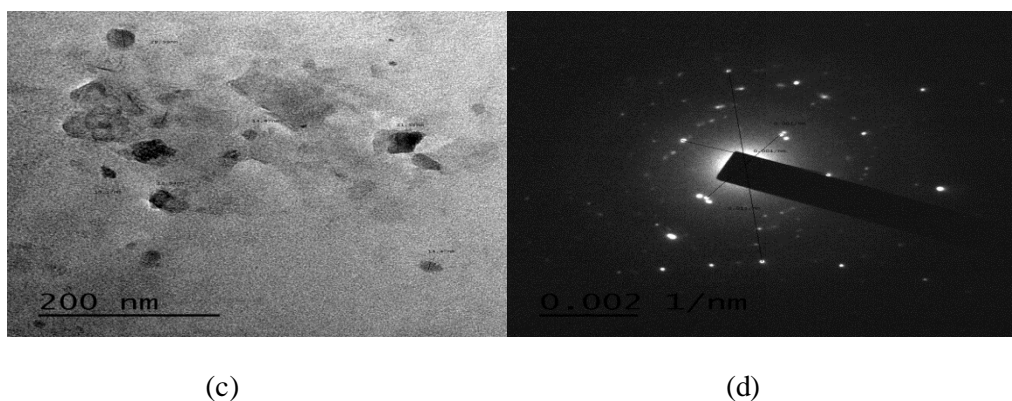
**Results and Discussion****TEM Images**

The TEM images of nanozinc sulphate sample in distilled water were measured using electron microscope model Joel 2010 and shown in **Figure 1** and graphic abstract. The images show almost deformed spherical particle shapes and broken dishes with an average size of 19 and 50 nm, respectively. It was clear from the electron diffraction image that the nano  $ZnSO_4$  is in crystalline form, with mean value of  $r_1$  340 nm (radius of the first ring) and  $r_2$  (mean radius of the second ring) equal 484 nm. The ratio of  $r_2$  to  $r_1$  equal 1.424.



(a)

(b)



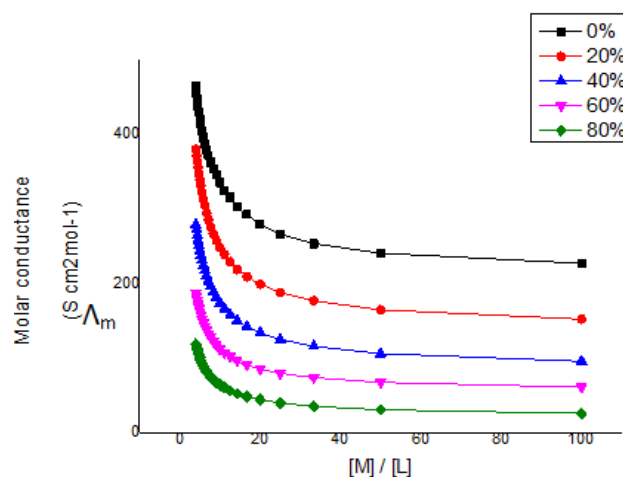
**Figure 1** TEM images for nano zinc sulphate, (d) is electron diffraction image.

The molar conductance ( $\Lambda_m$ ) values were calculated using equation (1):

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

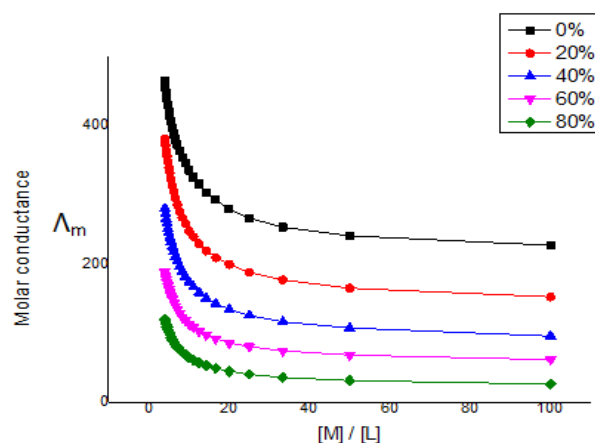
Where  $K_s$  and  $K_{solv}$  are the specific conductance of the solution and the solvent, respectively;  $K_{cell}$  is the cell constant and  $C$  is the molar concentration of the  $ZnSO_4 \cdot 7H_2O$  solution.

By drawing the relation between molar conductance ( $\Lambda_m$ ) and the molar ratio of metal to ligand  $[M]/[L]$  concentrations different lines are obtained with sharp breaks indicating the formation of 2:1, 1:1 and 1:2  $[M : L]$  stoichiometry complexes, Figures. (2-5).

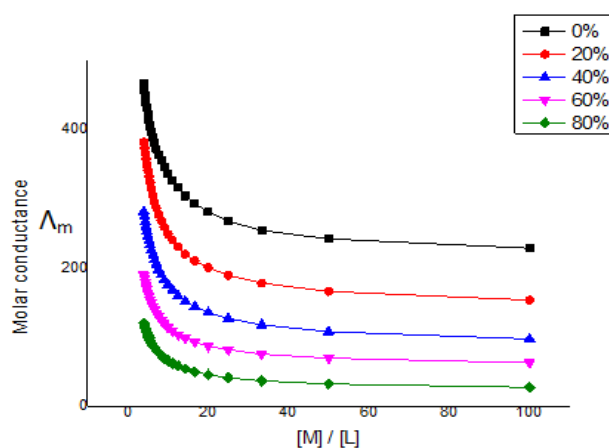


**Figure 2** The relation between molar conductance ( $\Lambda_m$ ) and the  $[M]/[L]$  molar ratio of nano  $ZnSO_4 \cdot 7H_2O$  to Calcon CA concentrations in different aqueous methanol solvents at 298.15 K.

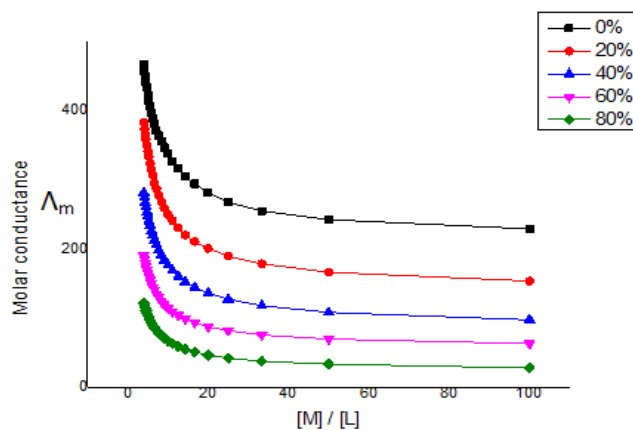
As is obvious from **Figures (2-5)**, in all cases studied, addition of the  $ZnSO_4 \cdot 7H_2O$  solution to the ligand (Calcon CA) solutions caused a continuous decrease in the molar conductance of the solutions, indicating the lower mobility of the complexes cations compared to the solvated ones. In all cases, the slope of the molar conductance-mole ratio plots change sharply at the point where the ligand to cation mole ratio is one, indicating the formation of a relatively stable 2:1, 1:1 and 1:2  $[M : L]$  complex between the ligand and the cation solutions used.



**Figure 3** The relation between molar conductance ( $\Lambda_m$ ) and the  $[M]/[L]$  molar ratio of nano  $ZnSO_4 \cdot 7H_2O$  to Calcon CA concentrations in different aqueous methanol solvents at 303.15 K.

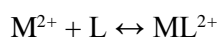


**Figure 4** The relation between molar conductance ( $\Lambda_m$ ) and the  $[M]/[L]$  molar ratio of nano  $ZnSO_4 \cdot 7H_2O$  to Calcon CA concentrations in different aqueous methanol solvents at 308.15 K.



**Figure 5** The relation between molar conductance ( $\Lambda_m$ ) and the  $[M]/[L]$  molar ratio of nano  $ZnSO_4 \cdot 7H_2O$  to Calcon CA concentrations in different aqueous methanol solvents at 313.15 K.

The 1:1 binding of the  $Zn^{2+}$  cations with Calcon CA ligand can be expressed by equilibrium:



And the corresponding formation constants ( $K_f$ ) for  $Zn^{2+}$ -Calcon CA complexes were calculated by using equation:

$$K_f = \frac{[ML^{2+}]f_{ML^{2+}}}{[M^{2+}][L]f_{M^{2+}}f_L} \quad (2)$$

Where,  $[ML^{2+}]$ ,  $[M^{2+}]$ ,  $[L]$  and  $f$  represents the equilibrium molar concentrations of complex, free cation, free ligand and the activity coefficients of the species indicated, respectively.

Under the dilute conditions used, the activity coefficient of the uncharged ligand,  $f_L$  can be reasonably assumed to as unity [6, 7]. The use of Debye - Hückel limiting law of electrolytes [8] leads to the conclusion that  $f_{M^{2+}} \approx f_{ML^{2+}}$ , so that the activity coefficients in Equation. 2 cancel out.

Thus, the complex formation constant in terms of the molar conductance can be expressed as [9- 11]:

$$K_f = \frac{[ML]}{[M][L]} = \frac{\Lambda_M - \Lambda_{obs}}{(\Lambda_{obs} - \Lambda_{ML})[L]} \quad (3)$$

$$[L] = C_L - \left\{ C_M + \frac{\Lambda_M - \Lambda_{obs}}{(\Lambda_M - \Lambda_{ML})} \right\} \quad (4)$$

Here,  $\Lambda_M$  is the molar conductance of the  $ZnSO_4 \cdot 7H_2O$  solution before addition of the ligand,  $\Lambda_{ML}$  the molar conductance of the complex,  $\Lambda_{obs}$  the molar conductance of the solution during titration,  $C_L$  the analytical concentration of the ligand, and  $C_M$ , the analytical concentration of the  $ZnSO_4 \cdot 7H_2O$  solution. The complex formation constant,  $K_f$ , and the molar conductance of the complex,  $\Lambda_{ML}$ , were obtained by computer fitting of Equations. (4) and (5) to the molar conductance- mole ratio data using a nonlinear least-squares program KINFIT [12].

The stability constants of the resulting 2:1, 1:1 and 1:2 [M : L] complexes were determined from the computer fitting of Equations 3 and 4 to the molar conductance-mole ratio data. A sample computer fit of the mole ratio data is shown in **Figure 1** and all  $K_f$  values are summarized in **Table 1** [13-30].

**Table 1** The formation constants ( $\log K_f$ ) of formation of of nano  $ZnSO_4 \cdot 7H_2O$  and Calcon CA complexes in mixed methanol-water at different temperatures

MeOH% by volu	log $K_f$											
	1:2 M:L				1:1 M:L				2:1 M:L			
	298.15	303.15	308.15	313.15	298.15	303.15	308.15	313.15	298.15	303.15	308.15	313.15
0	2.8545	2.8544	2.8769	2.8767	3.7054	3.7087	3.7038	3.6929	3.9481	3.9410	3.9364	3.9449
20	2.9286	2.9368	2.942	2.939	4.0161	4.0164	4.012	4.013	4.1315	4.1212	4.109	4.117

40	3.0289	3.0297	3.036	2.995	4.1069	4.1061	4.088	4.104	4.3505	4.341	4.343	4.354
60	3.0798	3.0744	3.091	3.082	4.2166	4.2013	4.183	4.179	4.4320	4.430	4.424	4.404
80	3.1680	3.1609	3.151	3.143	4.5432	4.5217	4.493	4.468	5.2519	5.217	5.185	5.166

The Gibbs free energies of formation for each stoichiometry complex were calculated by:

$$\Delta G_f = -RT \ln K_f \quad (5)$$

The calculated  $\Delta G_f$  values are presented in **Table 2**.

**Table 2** The Gibbs free energies ( $\Delta G_f$ ) of nano  $ZnSO_4 \cdot 7H_2O$  and Calcon CA complexes in methanol-water at different temperatures

MeOH % by vol.	$(\Delta G_f) J mol^{-1} K^{-1}$											
	1:2 M:L				1:1 M:L				2:1 M:L			
	298.15	303.15	308.15	313.15	298.15	303.15	308.15	313.15	298.15	303.15	308.15	313.15
0	-16.296	-16.5687	-16.97	-17.24	-21.1531	-21.527	-21.85	-22.14	-22.5389	-22.87	-23.22	-23.653
20	-16.718	-17.0468	-17.35	-17.62	-22.927	-23.313	-23.67	-24.06	-23.5856	-23.92	-24.24	-24.691
40	-17.291	-17.5863	-17.91	-17.96	-23.4454	-23.833	-24.12	-24.60	-24.8359	-25.20	-25.62	-26.108
60	-17.582	-17.845	-18.24	-18.48	-24.071	-24.386	-24.68	-25.05	-25.301	-25.71	-26.10	-26.408
80	-18.085	-18.347	-18.59	-18.84	-25.93	-26.246	-26.51	-26.79	-29.98	-30.28	-30.59	-30.97

The enthalpy changes of complexation ( $\Delta H_f$ ) were calculated from the plots of  $\log K_f$  against  $1/T$ , (slope =  $-\Delta H/2.303R$ ) (Figure. 6) using Van't Hoff Equation [20-35] :

$$\log K = -\frac{\Delta H}{2.303R} \left(\frac{1}{T}\right) + constant \quad (6)$$

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

Entropy change  $\Delta S$  were for complexes calculated [8] by using Gibbs-Helmholtz Equation (6) [31-45].

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

The calculated values of ( $\Delta H_f$ ) and ( $\Delta S_f$ ) for  $ZnSO_4 \cdot 7H_2O$ -Calcon CA stoichiometric complexes are presented in **Table 3**.

**Table 3** The enthalpies ( $\Delta H_f$ ) and entropies ( $\Delta S_f$ ) of formation of nan  $ZnSO_4 \cdot 7H_2O$  and Calcon CA complexes in methanol- water at different temperatures

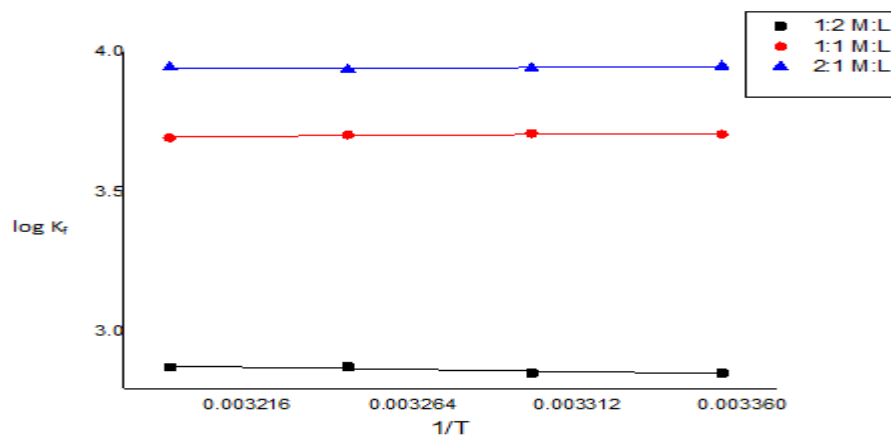
MeOH % by vol.	1:2 M:L				1:1 M:L				2:1 M:L			
	298.15	303.15	308.15	313.15	298.15	303.15	308.15	313.15	298.15	303.15	308.15	313.15
<b>0</b> $\Delta S_f$	65.316	65.1388	65.398	65.229	65.9231	66.0705	66.057	65.924	73.8227	73.716	73.655	73.8461
$\Delta H_f$	3.1781				-1.4981				-0.528			
<b>20</b> $\Delta S_f$	60.773	60.8523	60.879	60.761	75.3042	75.3360	75.278	75.323	72.7231	72.631	72.504	72.7694
$\Delta H_f$	1.4005				-0.4750				-1.903			
<b>40</b> $\Delta S_f$	46.835	47.0350	47.332	46.727	75.4862	75.5221	75.241	75.583	84.812	84.623	84.629	84.814
$\Delta H_f$	-3.3275				-0.9391				0.4509			
<b>60</b> $\Delta S_f$	62.037	61.8831	62.168	61.942	65.04614	65.0115	64.912	65.081	74.330	74.466	74.529	74.305
$\Delta H_f$	0.9146				-4.6784				-3.139			
<b>80</b> $\Delta S_f$	50.842	50.867	50.853	50.847	56.690	56.779	56.716	56.710	65.980	65.885	65.831	66.001
$\Delta H_f$	-2.92679				-9.0338				-10.31			

Enthalpies and entropies of complexation reactions show that, in most cases, the reaction is entropic controlled although, in all cases, a negative value of enthalpy change is also obtained. Comparison of  $\Delta H_f$  values of complexation (Table 1) clearly revealed that the steric hindrance in organic ligand is an important factor in enthalpy changes in the process of complexation reactions. On the other hand, in most cases, the entropy changes during complexation reactions are quite favorable. The entropic change during the complex formation is in fact affected by several factors including the change in flexibility of the reactants in the course of complexation reaction and the differences between the extent of solvation– desolvation of the uncompleted and complexes species. The values of  $K_A$  decrease with increasing temperature, this may be attributed to the short range interaction and the hydrogen bonding formed at low temperature, also the value of  $K_A$  increase as water percentage increase due to H-bonding formation and increasing viscosity.

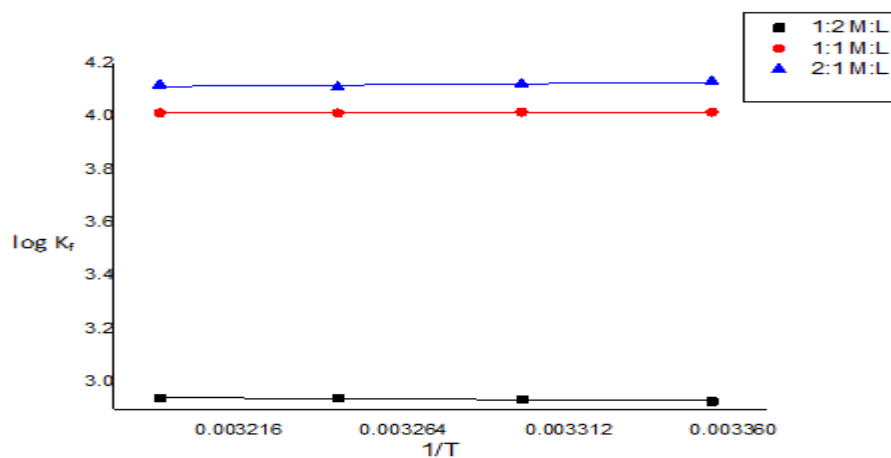
Interestingly to note, a comparison between the  $\Delta S_f$ ,  $\Delta H_f$  and  $\Delta G_f$  reported in **Table 1** reveals that generally a decrease in  $\Delta H_f$  value is accompanied with an increase in  $\Delta S_f$  in such a way that the free energy change  $\Delta G_f$  will remain more or less constant. Such enthalpy-entropy compensation effect was observed earlier in the case of complexation reactions of ligands with cation and inorganic guest species. Since the conductance of an ion depends mainly on its mobility, it is quite reasonable to treat the rate process taking place with the change of temperature on the basis of Equation (8) [46-74]:

$$\Delta \sigma = A e^{-\frac{E_a}{RT}} \quad (8)$$

Where, A is the frequency factor, R is the gas constant and  $E_a$  is the Arrhenius activation energy of the transfer process. Consequently, from the plot of  $\log(K_f)$  vs.  $1/T$ , the  $E_a$  values can be evaluated, giving high activation energy value due to solvation behavior.

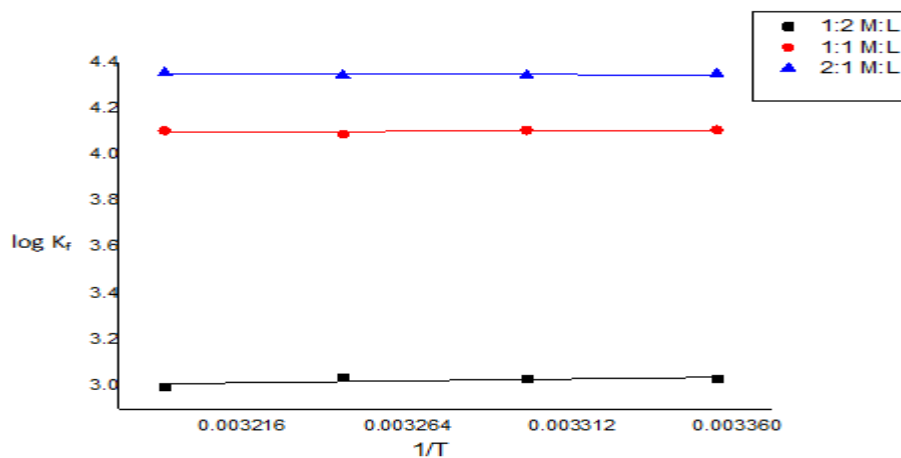


**Figure 6** The relation between  $\log K_f$  for (2:1), (1:1) and (1:2) stoichiometric complexes between nano  $ZnSO_4 \cdot 7H_2O$  and Calcon CA against  $1/T$  in pure water.

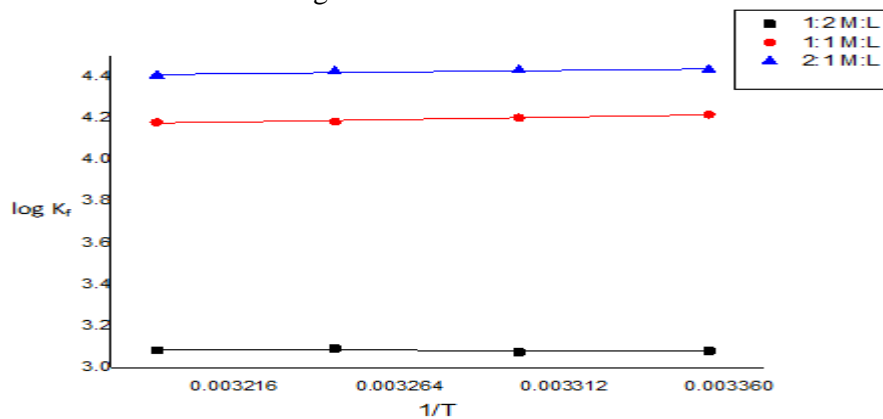


**Figure 7** The relation between  $\log K_f$  for (2:1), (1:1) and (1:2) stoichiometric complexes between nano  $ZnSO_4 \cdot 7H_2O$  and Calcon CA against  $1/T$  in 20% methanol- water

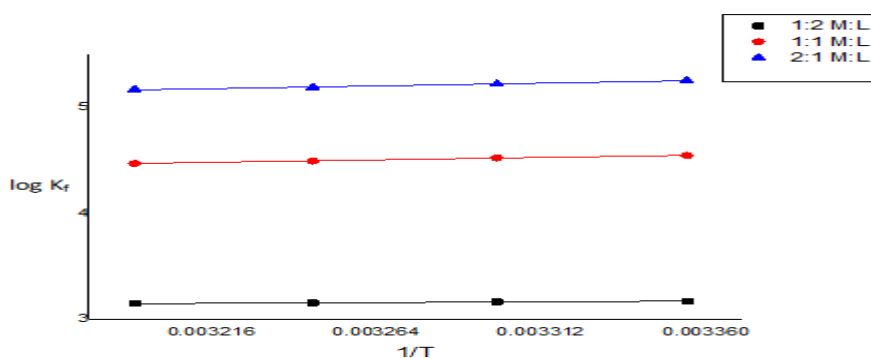




**Figure 8** The relation between  $\log K_f$  for (2:1), (1:1) and (1:2) stoichiometric complexes between nano  $ZnSO_4 \cdot 7H_2O$  and Calcon CA against  $1/T$  in 40% methanol- water.



**Figure 9** The relation between  $\log K_f$  for (2:1), (1:1) and (1:2) stoichiometric complexes between nano  $ZnSO_4 \cdot 7H_2O$  and Calcon CA against  $1/T$  in 60% methanol- water.



**Figure 10** The relation between  $\log K_f$  for (2:1), (1:1) and (1:2) stoichiometric complexes between nano  $ZnSO_4 \cdot 7H_2O$  and Calcon CA against  $1/T$  in 80 % methanol- water.

## Conclusion

This work focused on the study of thermodynamic complexation reactions between the ligand, Calcon CA, with nano  $Zn^{+2}$  metal cation. The stability constants of the complex formation between ligand and metal cations were investigated by applying the conductometric method at different temperatures. Based on the results, the stability constant for the complexation reaction of  $Zn^{+2}$ -Calcon CA shows a decrease with increasing temperatures.

In this thermodynamic study, the negative sign of the  $\Delta G_f$  shows that the ligand is capable of forming stable complexes and that the process will proceed spontaneously, while the positive sign of the entropy shows that  $\Delta S_f$  is the driving force of the complexation reaction in this complex formation. These facts mean that  $\Delta G_f$  is always negative and  $\Delta S_f$  is always positive.

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## Publication History

Received 11<sup>th</sup> Sep 2014  
Revised 24<sup>th</sup> Sep 2014  
Accepted 15<sup>th</sup> Oct 2014  
Online 30<sup>th</sup> Oct 2014