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Thermodynamic Interaction Parameters for Bulk and Nano Copper Acetate, CuAc with (CPB)* in Mixed Acetone (Ac) - H_2O Solvents

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Abstract
Characterization of nano copper acetate, CuAc, $Cu(OAc)_2$ is done by transmission electron microscope (TEM), atomic force microscope(AFM) and x-ray diffraction. The thermodynamic parameters for association and complex formation for both bulk $Cu(OAc)_2$ (normal) and nano $Cu(OAc)_2$ were evaluated by using condutometric measurements. The association thermodynamic parameters of both bulk [CuAc], $Cu(OAc)_2$ and nano-$Cu(OAc)_2$ salts in acetone (Ac)+H_2O were calculated in presence of N - bezylidene-4-chlorobenzohydrazide(CPB), nonisothermally at different temperatures, 293.15, 298.15, 303.15 and 308.15K. The thermodynamic solvation parameters for both types of $Cu(OAc)_2$[CuAc] were compared.

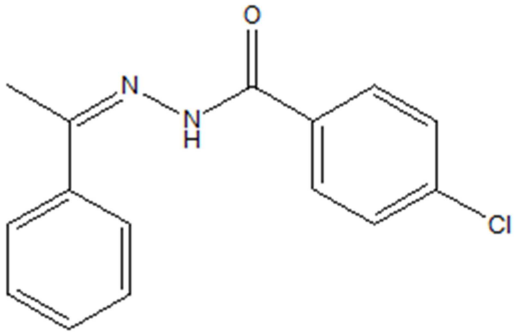
1. Introduction

Nano particles are very important for medicine and environment, because of their increased behaviours. The increase in the physical and chemical properties of nano materials are due to their high surface area and small volume, makes them very reactive, catalytic and able to pass through cell membranes [1]. Copper (II) acetate monohydrate is used in biochemical applications such as DNA extraction. Copper(II) complexes have been evaluated for anticancer, antibacterial and antifungal activities. Cu(II) complexes are known to cleave DNA; however ,increase deficiency is seen in the presence of an oxidizer (often H_2O_2). Copper acetate exists in a dimeric structure in the solid state as well as in non-aqueous solution. It has been shown to bind of both purine and pyrimidin enucleosides. Popular methods for DNA extraction may be hindered by endogenous levels of polysaccharides, phenolics and other organic molecules; Copper(II) acetatetre at menthes been shown to fix and remove tannins in samples from Pyrus (Pear) yielding high quality DNA[1].The difference in solvation parameters between nano and bulk $Cu(OAc)_2$can help us for the difference in solvation parameters between nano and bulk $Cu(OAc)_2$can help us for the uses of this salt in nano form. Some salts in nano form shows little solvation behavior in some solvents. Also the analytical determination of this salt and its nano form analytically by using our ligand as try to help in its quantitative determination.

* 4-Chloro-N'-(1-Phenylethylidene) Benzohydrazide

2. Experimental

2.1. Materials



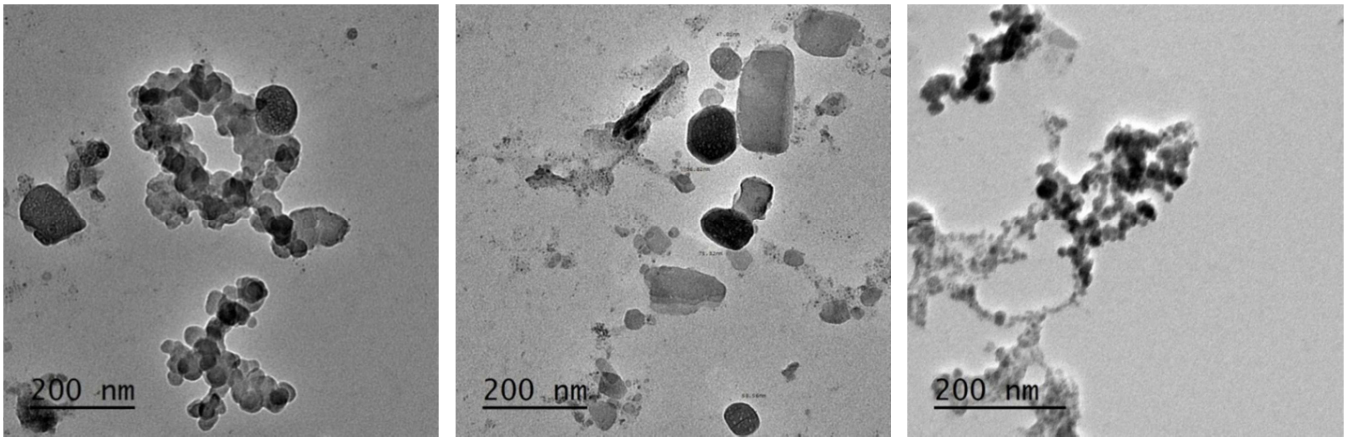
4-chloro-N'-(1-phenylethylidene) benzohydrazide (CPB) was prepared by mixing equimolar amounts of 4-chlorobenzohydrazide (0.01mole;1.70gm) and acetophenone (0.01mole;1.06gm), in 50 ml ethanol. The reaction mixture is maintaine flux temperature for 4 hrs. The product is filtered and recrystallized from absolute ethanol and finally dried in a vacuum desiccator over anhydrous $CaCl_2$.

Copper acetate monohydrate $Cu(OAc)_2$ was provided from Al Nasr chemicals Co .Nano- copper acetate $Cu(OAc)_2$ salt was obtained (prepared) by ball milling method using copper acetate salt.

The ball milling was a Retsch MM2000 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.

2.2. Transmission Electron Microscopy (TEM) Images

Transmission electron microscope is a special kind of electron microscope forming 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 images very complicated interference incident and diffracted beams. The images were measured by using JEOL HRTEM–JEM2100 (JAPAN) show that TEM of copper acetate $Cu(OAc)_2$ obtained in water have spherical shape (Fig.1).



(a) (b) (c)

Fig (1). *TEM images for nano CuAc.*

2.3. Condutometric Measurements

A solution of bulk and nano – copper acetate ($1x10^{-4}$M) were placed in a titration cell, thermostated at a given temperature and the conductance of the solution wasmeasured [2-21]. The ligand, 4-chloro-N'-(1-phenyl ethylidene) benzohydrazide (CPB), ($1x10^{-3}$M) was transferred step by step to the titration cell using a precaliberated 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 complex formation constant K_f, and the molar conductance of the complex ML, were evaluated by computer fitting for the molar conductance mole ratio data. The temperatures used are (293.15,298.15,303.15, and 308.15K). The specific conductivity K_s was achieved by using a conductivity bridge of the type (JENCO–3173COND).

3. Results and Discussion

3.1. Transmission Electron Microscopy (TEM) Images

The photographs from (TEM) are presented for nano-$Cu(OAc)_2$,CuAc salt. The images show that the nano copper acetate $Cu(OAc)_2$ in the form of regular spheres with little diffusion with water solvent, the boundaries clear as spheres, gathered spheres and attracted spheres in nets, the sizes ranging from 36.15 to 96.82 nm as shown in Fig.1.a,b,c .

3.2. Atomic Force Microscope (AFM)

The images of atomic force microscope for nano sample $Cu(OAc)_2$,CuAc measured in Mansoura University Nanotechnology center using Nanosurf FlexAFM, Switzerland apparatus is shown in Fig.(2).It is seen in Fig.(2) that all roughness data support the smooth surface of the nano CAc.

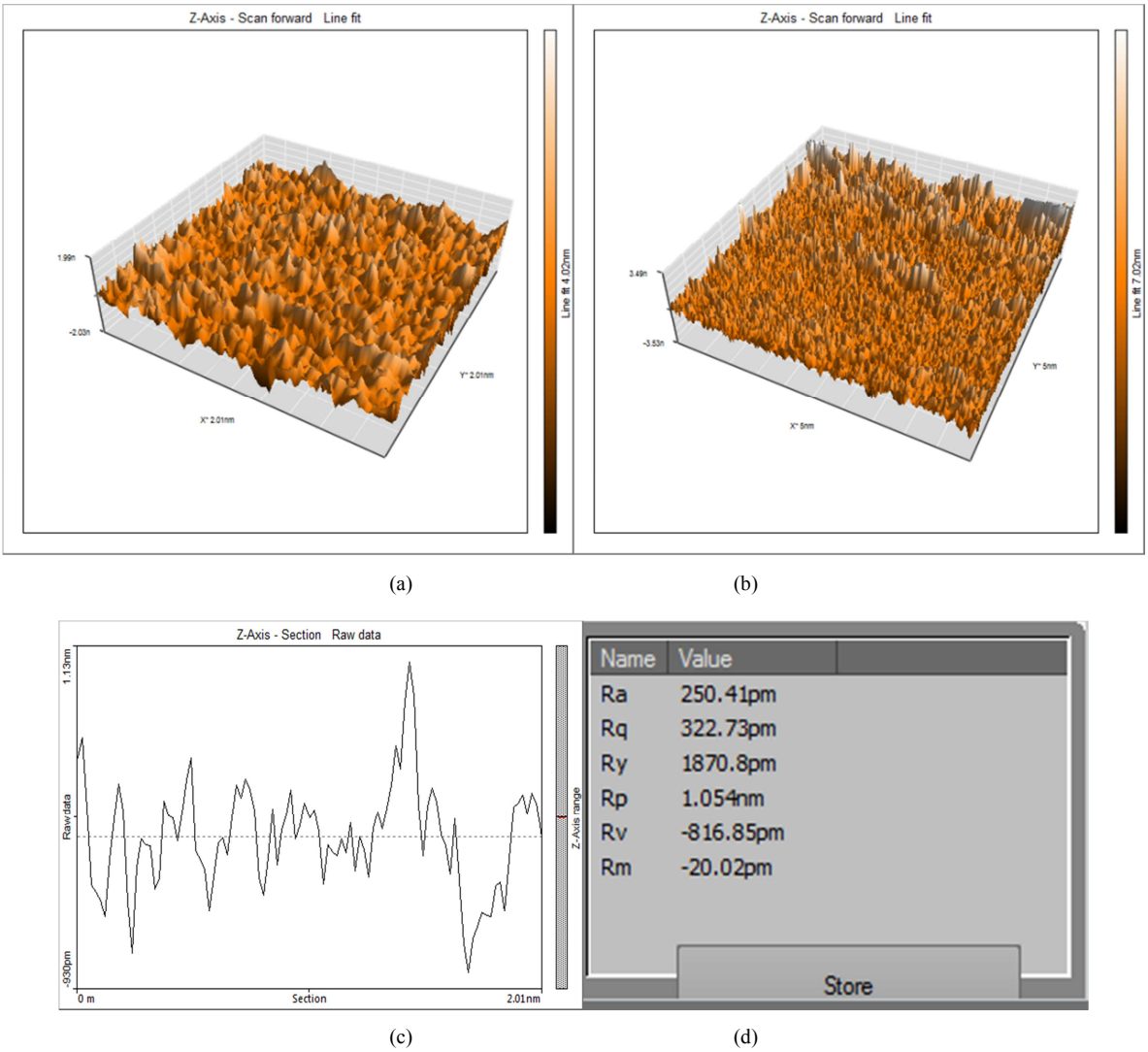


Fig (2). *AFM of nano CuAc.*

3.3. Conductometric Results

The stability of a transition metal complex with ligand 4-chloro-N'-(1-phenylethylidene) benzohydrazide (CPB) 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 complexes are strongly depend on donor ability and dielectric constant of the solvent [25-36],the shape and the size of the solvent molecules attracted[37-45].

3.3.1. Calculation of Thermodynamic Parameters of Association for Bulk and Nano-Copper Acetate $Cu(OAc)_2$ in Mixed Ac-H_2O Solvents

The specific conductance values (Ks) of different concentrations for bulk and nano-copper acetate $Cu(OAc)_2$ in MixedAc-H_2O mixtures were measured experimentally in absence of ligand, 4-chloro-N'-(1-phenylethylidene) benzohydrazide (CBP) at different temperatures (293.15, 298.15,303.15and308.15K). The molar conductance (Λ) values were calculated [46-59] using equation (1):

$$\wedge_m = \frac{(K_s - K_{solv}) \times K_{cell} \times 1000}{C} \Lambda m = \frac{(Ks - Ksolv) \times 1000}{C} \quad (1)$$

Where K_s and $K\ s_{olv}$ are the specific conductance of the solution and the solvent, respectively; Kcell (equal 1) is the cell constant and C is the molar concentration of the bulk and nano-$Cu(OAc)_2$ solutions. The limiting molar conductance (Λ_o) at infinite dilutions was estimated for bulk and nano-$Cu(OAc)_2$ in Ac-H_2O mixtures in absence of ligand

at different temperatures by extrapolating the relation between Λ_m and C_m½ to zero concentration. The association constants KASS for nano and bulk $Cu(OAc)_2$ were calculated from equation (2) in presence of (CPB) and tabulated in Tables (1) and (2).

$$KA = \frac{\Lambda\circ\left[(\Lambda\circ)-(\Lambda)\right]}{4Cm2.S(z)^2\,\Lambda 3\,\gamma_{\pm}^2} \quad (2)$$

Table (1). *Molar electrical conductance (Λ_m), limiting mola rconductance (Λ_0), association constants (KASS), K_A and free energies of association (GASS), enthalpy (ΔH) and entropy(ΔH) for8.41×10^{-6}M bulk [CuAc], Cu($CH_3COO)_2$ in mixed Ac-H_2O solvents at 293.15,298.15,303.15and 308.15K.*

C mol/l	(X_s) Acetone	$\Lambda_0\Omega^{-1}$, Cm^2mol	$\Lambda_m\Omega^{-1}$, Cm^2mol	$\gamma_{\pm}$	K_A	ΔG_A KJ/mol	ΔH_A KJ/mol	ΔS_A J/mol.K
		74.5247	59.6214		1.8587×10^7	-40.8019		93.0107
	0.0578	76.4857	61.1342		1.8227×10^7	-41.4493	-13.5358	93.6223
		79.3355	65.1602		1.4417×10^7	-41.5534		92.4214
		91.2475	73.2000		1.4891×10^7	-42.3217		93.4150
		49.1478	40.6626		2.1999×10^7	-41.2128		119.1718
8.4100×10^{-6}	0.1407	59.7485	48.617	0.9966	2.0528×10^7	-41.7441	-6.2776	118.9553
		62.3548	50.2762		2.1020×10^7	-42.5039		119.4995
		66.7458	54.0704		1.8982×10^7	-42.9436		118.9874
		32.4784	25.8810		4.3837×10^7	-42.8935		61.0757
	0.2692	36.8574	29.5728		3.6820×10^7	-43.1926	-24.98.92	61.0546
		42.7485	34.0014		3.3738×10^7	-43.6967		61.7102
		44.8457	36.7586		2.5898×10^7	-43.7397		60.8485

Table (2). *Molar electrical conductance e(Λ_m), limiting molar conductance (Λ_0), association constants (KASS), K_A and free energies of association (GASS), enthalpies (ΔH) and entropies (ΔH) for 8.41×10^{-6}M nano Cu($CH_3COO)_2$ in mixedAc-H_2O solvents at 293.15,298.15,303.15 and 308.15K.*

C mol/l	(X_s) Acetone	$\Lambda_0\ \Omega^{-1}$,Cm^2mol	$\Lambda_m\ \Omega^{-1}$,Cm^2mol	$\gamma_{\pm}$	K_A	ΔG_A KJ/mol	ΔH_A KJ/mol	ΔS_A J/mol.K
		66.2147	48.8000		3.5192×10^7	-42.3580		72.0939
	0.0578	69.3548	53.6800		2.4927×10^7	-42.2255	-21.2237	70.4404
		72.81345	56.1200		2.4391×10^7	-42.8789		71.4338
		75.2585	58.5600		2.2195×10^7	-43.3443		71.7852
		48.2314	39.0400		2.6363×10^7	-41.6539		92.1355
8.4100×10^{-6}	0.1407	53.8794	43.9200	0.9966	2.2464×10^7	-41.9676	-14.6444	91.6425
		57.2244	46.3600		2.2130×10^7	-42.6336		92.3280
		59.3685	48.8000		1.9149×10^7	-42.9660		91.9085
		33.8741	26.8400		4.3707×10^7	-42.8863		68.9347
	0.2692	36.8475	29.2800		3.9398×10^7	-43.3604	-22.6781	69.3688
		38.2222	31.7200		2.7619×10^7	-43.1921		67.6695
		42.1144	34.1600		2.9807×10^7	-44.0999		69.5173

3.3.2. Calculation of Thermodynamic Parameters of Complex Formation for Bulk and Nano – Copper Acetate with 4-Chloro-N'-(1-Phenyl Ethylidene) Benzohydrazide (CPB) in Ac-H_2O Mixures

The specific conductance values (Ks) of different concentrations of bulk and nano –Cu $(OAc)_2$ in Ac–H_2O mixtures were measured experimentally in the presence of ligand (CPB) at different temperatures (293.15,298.15, 303.15,and308.15K).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-$Cu(OAc)_2$ in presence of ligand (CPB) 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) stoichiometry complexes, as done in previous works [71-75].The formation constants (K_f) for bulk and nano-$Cu(OAc)_2$, CuAc complexes were calculated for each type of complexes (1:2) and (1:1)(M:L) by using equation (4)[75-78]:

$$K_f = \frac{\wedge_m - \wedge_{obs}}{\left(\wedge_{obs} - \wedge_{ML}\right)[L]}\,Kf = \frac{\Lambda m - \Lambda obs}{(\Lambda obs - \Lambda ML)[L]} \quad (3)$$

Where Λ_o is the limiting molar conductance of the bulk and nano-$Cu(OAc)_2$ alone, Λ_m is the molar conductance ofsolution during titration, Λ_{ML} is the molar conductance of the complex at the inflection and[L] is the ligand (CPB) concentration .The Gibbs free energies of formation for each

stoichiometry complex(ΔG)f were calculated by using equation (5)[10-25]:

$$\Delta G_f = -2.303RT \log K_f \quad (4)$$

The obtained values (K_f) for nano and bulk-CuAc stoichiometry complexes and their calculated ΔG_f values at 308.15K as example are evaluated and described in Tables.3 and 4.

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) stoichiometry complexes) as explained in previous works [26-60].

From the relation between log K_f and 1/T, ΔH_f can 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-$Cu(OAc)_2$ stoichiometric complexes were calculated [32,33] for each type of complexes (1:2) and (1:1)(M:L) by using Gibbs-Helmholtz equation(5)[60-78].

Table (3). *Complex formation parameters for the interaction of nano CuAc with CPB forming 1:1 complexes.*

X_s Ac	T K	Λ_M Ω^{-1}, Cm^2mol	Λ_{ML} Ω^{-1}, Cm^2mol	Λ_{obs} Ω^{-1}, Cm^2mol	[L]×10^{-5}	K_f	ΔG_f KJ/mol	ΔH KJ/mol	ΔS J/mol.K
				50.1600	1.5300	2.8446×10^5	-30.6133		205.1
	293.15	56.1425	48.7854	51.0400	1.6700	1.3552×10^5	-29.2971		209.5
				51.9200	1.8000	7.4837×10^4	-28.2916		213.0
				52.4400	1.5300	4.5440×10^5	-33.3800		192.3
	298.15	60.0014	51.3524	53.3600	1.6700	1.9809×10^5	-29.7312		204.6
				54.2800	1.8000	1.0857×10^5	-28.7475		207.9
0.0578				54.7200	1.5300	3.3023×10^5	-32.0337	-90.7239	193.6
	303.15	62.3265	53.2145	55.6800	1.6700	1.6143×10^5	-30.7280		197.9
				56.6400	1.8000	9.2225×10^4	-27.8676		207.3
				57.0000	1.5300	2.9591×10^5	-31.2333		193.1
	308.15	64.4258	55.3598	58.0000	1.6700	1.4574×10^5	-29.9717		197.2
				59.0000	1.8000	8.2807×10^4	-29.0175		200.2
				36.9200	1.5300	1.6822×10^5	-29.3328		224.0
	293.15	41.2147	35.2514	37.4800	1.6700	1.0035×10^5	-28.5522		226.7
				38.0400	1.8000	6.3248×10^4	-27.8674		229.0
				41.0400	1.5300	1.7281×10^5	-30.9026		215.0
	298.15	45.2587	39.4444	41.7600	1.6700	9.0475×10^4	-27.8209		225.3
				42.4800	1.8000	5.0854×10^4	-26.8671		228.5
0.1407				43.0400	1.5300	1.3693×10^6	-35.6190	-94.9947	195.9
	303.15	49.1470	42.7485	43.7600	1.6700	3.1891×10^5	-32.4727		206.2
				44.4800	1.8000	1.4974×10^5	-29.0491		217.5
				44.8800	1.5300	8.6468×10^5	-33.8919		1983
	308.15	52.2145	44.3256	45.0800	1.6700	5.6630×10^5	-33.3933		1999
				45.8400	1.8000	2.3385×10^5	-31.6777		2055
				25.0800	1.5300	3.9271×10^5	-31.3994		1521
	293.15	28.8527	24.4521	25.5200	1.6700	1.8687×10^5	-30.0938		1566
				25.9600	1.8000	1.0658×10^5	-29.1828		1597
				27.3600	1.5300	1.8207×10^5	-31.0364		1508
	298.15	30.24152	26.3256	27.8400	1.6700	9.4957×10^4	-27.9388		1612
				28.3200	1.8000	5.3525×10^4	-26.9940		1643
0.2692				29.6400	1.5300	1.7442×10^5	-30.4246	-75.9895	150.3
	303.15	33.3256	28.2589	30.1600	1.6700	9.9709×10^4	-29.4935		153.4
				30.6800	1.8000	6.0707×10^4	-26.8482		162.1
				32.9200	1.5300	1.2112×10^6	-34.7275		133.9
	308.15	36.2224	32.7418	33.4800	1.6700	2.2245×10^5	-31.0378		145.9
				34.0400	1.8000	9.3394×10^4	-29.3258		151.4

$$\Delta G_f = \Delta H_f - T\Delta S_f \quad (5)$$

The calculated values of (ΔH_f), ($T\Delta S_f$) and(ΔS_f) for bulk and nano-$Cu(OAc)_2$at different temperatures stoichiometry complexes support the solvation free energy data.I t was found that regular spheres are seen in TEM images for nano $Cu(OAc)_2$which explain that the association of nano $Cu(OAc)_2$ is big in solution phase. Therefore the association thermodynamic parameters and the complex formation parameters for nano $Cu(OAc)_2$ is bigger than bulk $Cu(OAc)_2$in presence of 4-chloro-N'-(1-phenylethylidene) benzohydrazide (CPB). (CPB).The association constants and free energies of association for both bulk and nano CAc are bigger than complex formation in presence of 4-chloro-N'-(1-phenylethylidene) benzohydrazide (CPB) because both complexation and association are proceed which seen in association parameters. The paper compares the thermodynamic behaviour for nano and bulk $Cu(OAc)_2$. Valuable results were obtained indicating the greater activity for nano salt in absence and presence of ligand (CPB).This help for uses of nano $Cu(OAc)_2$better than bulk $Cu(OAc)_2$ as food additive, more stabilization of human pressure . Analytically we determine both nano and bulk $Cu(OAc)_2$ conductometrically from the association and complex formation thermodynamic parameters given in this work.

Table (4). *Complex formation parameters for the interaction of nano CuAc with CPB forming 1:2 complexes.*

X_s Ac	T K	$\Lambda_M\ \Omega^{-1}$, Cm^2mol	$\Lambda_{ML}\ \Omega^{-1}$, Cm^2mol	$\Lambda_{obs}\ \Omega^{-1}$, Cm^2mol	[L] $\times10^{-5}$	K_f	ΔG_f KJ/mol	ΔH_f KJ/mol	ΔS_f J/mol.K
				55.4400	5.6600	5.7186×10^4	-26.7026		65.5000
	293.15	66.2147	52.1111	56.3200	7.4100	3.1726×10^4	-25.6973		68.9000
				57.2000	8.3300	2.1266×10^4	-25.1198		70.9000
				57.9600	5.6600	5.5396×10^4	-27.9875		60.1000
	298.15	69.3548	54.3258	58.8800	7.4100	3.1040×10^4	-25.2131		69.4000
				59.8000	8.3300	2.0953×10^4	-24.6688		71.2000
0.0578								-45.9032	
				60.4800	5.6600	6.7645×10^4	-28.0369		58.9000
	303.15	72.81345	57.2587	61.4400	7.4100	3.6708×10^4	-26.9330		62.6000
				62.4000	8.3300	2.4315×10^4	-24.6179		70.2000
				63.0000	5.6600	5.8958×10^4	-27.2337		60.6000
	308.15	75.2585	59.3265	64.0000	7.4100	3.2510×10^4	-26.1898		64.0000
				65.0000	8.3300	2.1706×10^4	-25.5867		65.9000
				40.3200	5.6600	6.6735×10^4	-27.0790		361.3000
	293.15	48.2314	38.2255	40.9600	7.4100	3.5886×10^4	-26.0028		365.0000
				41.6000	8.3300	2.3591×10^4	-25.3814		367.1000
				45.3600	5.6600	6.8041×10^4	-28.5143		350.5000
	298.15	53.8794	43.1478	46.0800	7.4100	3.5896×10^4	-25.5674		360.4000
				46.8000	8.3300	2.3270×10^4	-24.9288		362.5000
0.1407								-133.0059	
				47.8800	5.6600	1.4650×10^4	-24.1805		359.0000
	303.15	57.2244	54.2587	48.6400	7.4100	4.8529×10^3	-21.7481		367.0000
				49.4000	8.3300	3.2016×10^3	-19.6755		373.8000
				50.4000	5.6600	4.9763×10^4	-26.8133		344.6000
	308.15	59.3685	47.2158	51.2000	7.4100	2.7668×10^4	-25.7833		348.0000
				52.0000	8.3300	1.8489×10^4	-25.1757		349.9000
				27.7200	5.6600	4.3724×10^4	-26.0483		122.5000
	293.15	33.8741	25.2333	28.1600	7.4100	2.6348×10^4	-25.2368		125.3000
				28.6000	8.3300	1.8806×10^4	-24.8100		126.7000
				30.2400	5.6600	3.1029×10^4	-26.5023		118.9000
	298.15	36.8475	26.4777	30.7200	7.4100	1.9492×10^4	-24.0789		127.1000
				31.2000	8.3300	1.4357×10^4	-23.7315		128.2000
0.2692								-61.9646	
				32.7600	5.6600	4.1145×10^4	-26.7836		116.1000
	303.15	38.2222	30.4145	33.2800	7.4100	2.3276×10^4	-25.7656		119.4000
				33.8000	8.3300	1.5681×10^4	-23.5486		126.7000
				35.2800	5.6600	2.9759×10^4	-25.5387		118.2000
	308.15	42.1144	31.2225	35.8400	7.4100	1.8338×10^4	-24.7464		120.8000
				36.4000	8.3300	1.3250×10^4	-24.3218		122.2000

4. Conclusion

The association and complex formation parameters for nano copper acetate are bigger than that of bulk $Cu(OAc)_2$ in mixed Ac-H_2O solvents because of the bigger in both entropies and dissociation degrees for the former. Comparison between the nano and bulk electrolyte thermodynamics is very important to discuss their behaviors in solutions. Uses of nano $Cu(OAc)_2$, CuAc is preferred than bulk $Cu(OAc)_2$ in the used Ac-H_2O solvents for any application. In this work lot of thermodynamic data was given to illustrate the behaviour in used mixed solvents.

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