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THERMODYNAMIC PARAMETERS FOR SOLVATION OF NANO ZNO AND ITS DOPES IN 50% DMSO - 50% DMF (V/V) SOLUTIONS AT DIFFERENT TEMPERATURES

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ABSTRACT

The molal solubility for nano zinc oxide (ZnO) , copper (2%) doped ZnO , Fe(2%) doped ZnO , Ti(2%) doped ZnO , Ag (25) doped ZnO in 50%DMF and 50% DMSO, (v/v), volume to volume were measured at 293.15, 303.15 , 313.15, 323.15K .

The thermodynamic parameters, Gibbs free energy of solvation, enthalpies and entropies of solvation were evaluated for nano zinc oxide and nano zinc oxide doped from the solubility measurements. These thermodynamic parameters explain the effect of doping elements on the thermodynamic behviours of nano zinc oxide in solvation process.

Keywords: Thermodynamic parameters, Solvation, Gibbs free energies, Enthalpies, Entropies, Zinc oxide dopes, Mixed DMF-DMSO solvents.

Contribution/ Originality

This paper uses the thermodynamic parameters for discussing the solubility behaviour because few studies is finding for nano particles solvation explanation.

1. INTRODUCTION

Nanoparticles have attracted much attention due to their individual features, which are unavailable in conventional macroscopic materials. Several studies have found biomedical applications for nano particles mainly as antibacterial material [1] cell imaging [2] drug delivery, and cancer therapy [2]. Also semiconductor Nanoparticles have much attention in recent years due to novel

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Optical, electrical and mechanical properties, which results with their quantum confinement effects compared with their bulk counter parts. Among various nano semiconductor, zinc oxide (ZnO) nano particles are the most frequently studied [3]. Normal ZnO has band gap of 3.37 ev and high binding energy [1, 2] and high exciting binding energy 60 eV [3] Application uses of nano ZnO include solar energy [3], varistors [4], luminescence [5]., photo-catalysis [6],electrostatic coating [7], transparent protection films [8] and chemical sensors [9-16]. In particular, zinc oxide has other applications such as gas sensors, blue lasers, short-wavelength light-emitting devices. ZnO belongs to a group of metal oxides that are characterized by their photo-catalytic and photo-oxidizing ability against chemical and Biological species [16-18]. Therefore, ZnO was recognized for its utility in biological applications as an antibacterial material [2, 7, 8, 18, 19] Metal doped ZnO has pharmathotical importance as its show antibacterial activities against microorganisms such staphylococcus and bacillus [3]. Copper doped zinc oxide has photo catalytic and photoluminescence properties [3]. Iron doped zinc oxide has photo catalytic oxidation of dye bearing waste water. We reported in this paper the solubility of nano zinc oxide and nano zinc oxide doped in mixed solvent which has a great importance in many industrial processes as well as laboratory uses. Thermodynamics of nano zinc oxide and nano zinc oxide doped were calculated which help in explanation of the preferential solvation of ions and single ion thermodynamic functions [20-31].

2. EXPERIMENTAL

2.1. Materials

All chemical reagents were analytical grade and were used without further purification. The materials which used in our preparations are; Zinc nitrate $Zn(NO_3).6H_2O$ (99%), zinc acetate $Zn(CH_3COO)_2.2H_2O$, ammonium carbonates $(NH_4)_2CO_3.H_2O$, ammonium hydroxide NH_4OH (35%), sodium hydroxide NaOH, oxalic acid $H_2C_2O_4.2H_2O$, silver nitrate $AgNO_3$ (99%), copper nitrate $Cu(NO_3)_2.3H_2O$, ferric nitrate $Fe(NO_3)_3.9H_2O$, ferric sulphate $FeSO_4 \cdot 7H_2O$ and absolute ethanol (99%) all provided from Adwic. Double distilled water was used throughout all the experiments.

2.2. Synthesis of Pure and Dopes ZnO Nanoparticles

0.15 M $H_2C_2O_4.2H_2O$ distilled water solution was slowly dropped into the mixture containing 0.1M $Zn(CH_3COO)_2.2H_2O$ and 2% $Cu(NO_3)_2 \cdot 3H_2O$ with constant stirring For 15 h. The precipitate obtained was filtered and washed with acetone for at least three times, dried at 120 °C. Finally, the copper doped ZnO sample was ignited at 450 °C for 2 h .The sample denoted as Z_{2Cu}. The last procedure was applied to prepare all the other samples:

Z_{pure},, Z_{2Ag}, Z_{2Cu}, Z_{2Fe}, Z_{2Ti} abbreviated to pure ZnO, Ag (2%)-doped ZnO, Cu (2%)-doped ZnO, Fe (2%)-doped ZnO, Ti (2%)-doped ZnO samples, respectively.

2.3. Characterization Methods

2.3.1. X-Ray Diffraction (XRD)

The crystalline phases for all investigated samples were identified by X-ray diffraction (XRD) using a Diano (made by Diano Corporation, U.S.A.). The patterns were run with Cu-filtered CuKα radiation (λ = 1.5418Å) energized at 45 kV, and 10 m A. The samples were measured at room temperature in the range from 2θ = 2 to 80°. The XRD phases present in the samples were identified with the help of ASTM Powder Data Files.

2.3.2. Scanning Electron Microscope (SEM)

Scanning electron microscopy (SEM), JEOL JEM-100CXII was taken to examine the morphology and dimension of the investigated samples using conductive carbon paint.

2.4. Preparation of Saturated Solution and Solubility Measurement

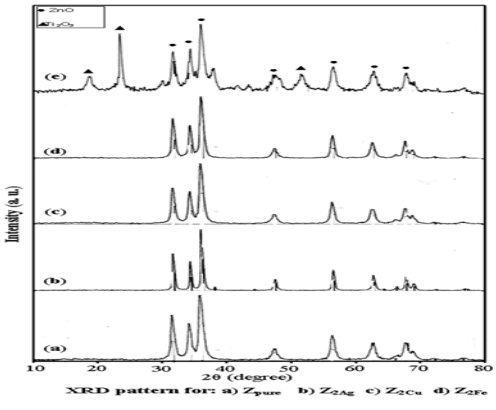
The saturated solution of nano ZnO and nano doped ZnO were prepared by dissolving solid amount in closed test tubes containing 50%DMF &50% DMSO. The tube were placed in water thermostat for three days till equilibrium reached. The solubility of nano zinc oxide and nano zinc oxide doped were measured by tacking 1 ml of each saturated solution putting in a small weighed beaker (10 ml) and evaporated under I.R. lamp till dryness [20, 30-44] and then weighed with average error in weight about 0.0001 g [20, 30-44].

The molal solubility for nano zinc oxide and nano zinc oxide doped were calculated by subtracting the evaporated weights of samples minus the weight of beaker empty. The same procedures repeated at different temperatures.

3. RERSULTS AND DISCUSSION

3.1. X-Ray Diffraction (XRD)

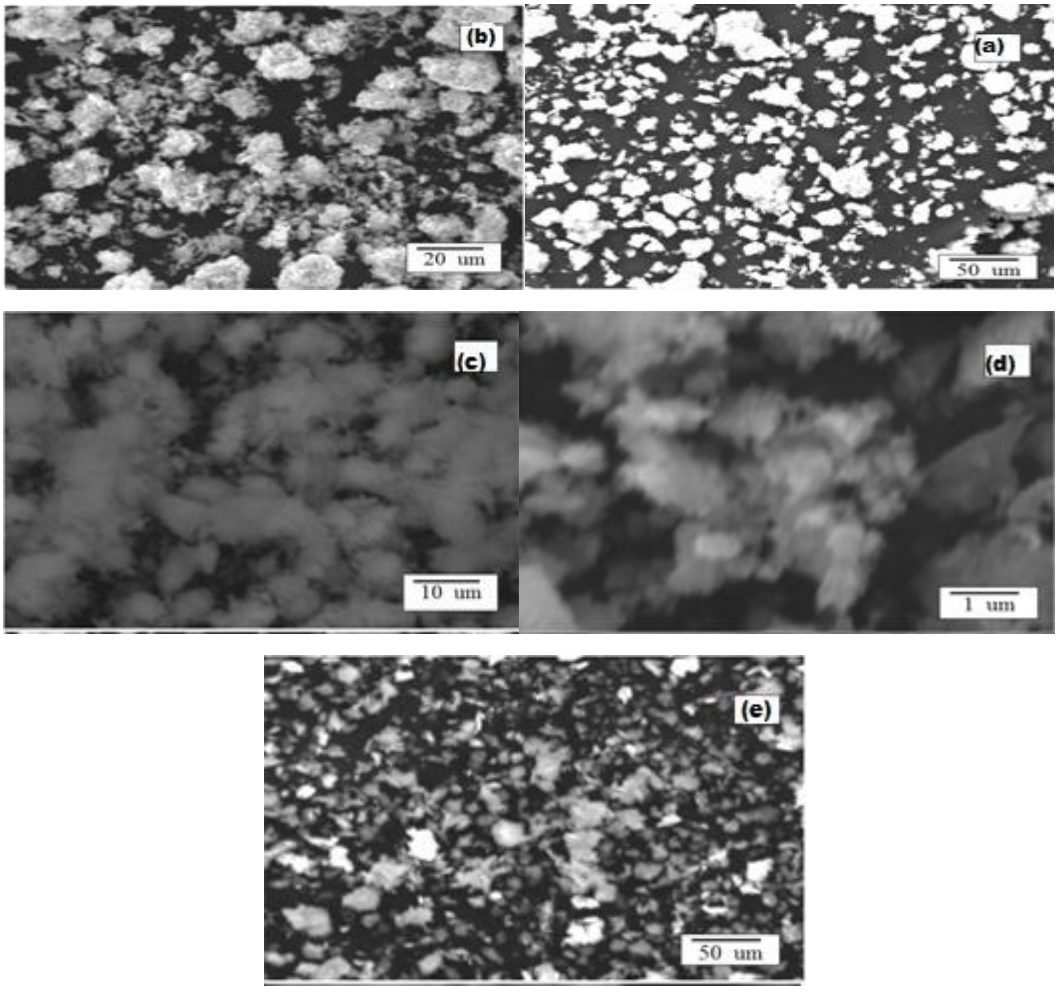
X-ray diffraction for ZnO and doped ZnO represented in the following Figure. The beaks are similar with slight difference in case of pure ZnO.



XRD pattern for: a) Z_{pure} b) Z_{2Ag} c) Z_{2Cu} d) Z_{2Fe} e) Z_{2Ti}

3.2. Scanning Electron Microscope (SEM)

The SEM image of ZnO and doped ZnO by using scan electron microscope are shown as ZnO in figure (a) , Z_{2Ag} (b) , Z_{2Ti} (c) , Z_{2Fe} (d) , Z_{2Cu} (e). Broken dishes shapes were observed for all.



3.3. Gibbs Free Energies of Solvation

The Gibbs free energy of solvation of nano zinc oxide and nano zinc oxide doped were measured in presence of 50% DMSO+50%DMF because the solubility is very small in both pure solvents. The molal solubility (S_m) of zinc oxide and zinc oxide doped in 50%DMSO +50%DMF at 293.15 , 303.15 , 313.15 , 323.15K were measured gravimetrically by taking the mean values for at least three readings for each solution . The S_m values are reported in Tables 1, 2, 3 and 4 at different temperatures. The solubilities and also the thermodynamic parameters for ZnO and its doping metals are greater in the mixture of DMF and DMSO solvents than the individual ones .The free energies for the doping samples decrease in positivity, favor more application can be used with best result in case of ferric sample [44-51].

Table-1. Solvation parameters (S_M, $\log\gamma_\pm$, pK_{sp}), Gibbs free energy of solvation (ΔG) and enthalpies and entropies of solvation parameters and Gibbs free energies for nano zinc oxide and nano zinc oxide doped in 50%DMF and 50% DMSO at 293.15K

Sample	S_M molality	$\log\gamma_\pm$	pK_{sp}	ΔG (kJ/mol)	ΔH (kJ/mol)	$T\Delta S$ (kJ/mol)	ΔS (kJ/mol)
ZnO	0.1130	-1.354	4.7163	26.473	30.901	4.429	0.0151
ZnO+TiO_2	0.1244	-1.343	4.6113	25.883	3.930	-21.953	-0.0749
ZnO+Cu^{+2}	0.1333	-1.332	4.5266	25.408	18.763	-6.645	-0.0227
ZnO+Fe^{+3}	0.1686	-1.263	4.1796	23.460	23.713	0.253	0.0009
ZnO+Ag	0.1348	-1.330	4.5130	25.332	47.480	22.148	0.0756

Molality, g.mole/1000g solvent

Positive enthalpies in all Tables denote that the solvation is endothermic and high heat needed. Adding doping metal decrease the positivity of enthalpies favoring less heat required. In case of TiO_2 giving the least energy needed followed by Cu^{+2} and Fe^{+3}.

Table-2. Solvation parameters(S_M, $\log\gamma_\pm$, pK_{sp}), Gibbs free energy of solvation (ΔG) and entropies of solvation parameters and Gibbs free energies for nano zinc oxide and nano zinc oxide doped in 50%DMF and 50% DMSO at 303.15K

Sample	S_M molality	$\log\gamma_\pm$	pK_{sp}	ΔG (kJ/mol)	$T\Delta S$ (kJ/mol)	ΔS (kJ/mol)
ZnO	0.1381	-1.337	4.4823	26.017	4.884	0.0161
ZnO+TiO2	0.1266	-1.353	4.5914	26.651	-22.721	-0.0749
ZnO+Cu^{+2}	0.1358	-1.341	4.5048	26.148	-7.385	-0.0244
ZnO+Fe^{+3}	0.1181	-1.363	4.6715	27.115	-3.403	-0.0112
ZnO+Ag	0.1372	-1.338	4.4909	26.067	21.413	0.0706

Table-3. Solvation parameters(S_M, $\log\gamma_\pm$, pK_{sp}), Gibbs free energy of solvation (ΔG) and entropies of solvation parameters and Gibbs free energies for nano zinc oxide and nano zinc oxide doped in 50%DMF and 50% DMSO at 313.15K

Sample	S_M molality	$\log\gamma_\pm$	pK_{sp}	ΔG (kJ/mol)	$T\Delta S$ (kJ/mol)	ΔS (kJ/mol)
ZnO	0.1523	-1.322	4.3431	26.041	4.860	0.0155
ZnO+TiO2	0.1289	-1.362	4.5689	27.395	-23.465	-0.0749
ZnO+Cu^{+2}	0.1497	-1.327	4.3684	26.193	-7.430	-0.0237
ZnO+Fe^{+3}	0.1311	-1.359	4.5482	27.271	-3.558	-0.0114
ZnO+Ag	0.1978	-1.207	3.8801	23.265	24.215	0.0773

Table -4. Solvation parameters (S_M, $\log\gamma_{\pm}$, pK_{sp}), Gibbs free energy of solvation (ΔG) and entropies of solvation parameters and Gibbs free energies for nano zinc oxide and nano zinc oxide doped in 50%DMF and 50% DMSO at 323.15K

Sample	S molality	$\log\gamma_{\pm}$	pK_{sp}	ΔG (kJ/mol)	TΔS (kJ/mol)	ΔS (kJ/mol)
ZnO	0.1669	-1.301	4.1965	25.965	4.936	0.0153
ZnO+TiO_2	0.1312	-1.370	4.5461	28.129	-24.199	-0.0749
ZnO+Cu^{+2}	0.1640	-1.308	4.2248	26.141	-7.378	-0.0228
ZnO+Fe^{+3}	0.1445	-1.348	4.4179	27.335	-3.623	-0.0112
ZnO+Ag	0.2013	-1.207	3.8426	23.775	23.704	0.0734

Also, the activity coefficients were calculated by the use of Debye – Hückel equation (1) [34, 44-48] and their values are given in Tables 1, 2, 3 and 4.

$$\log \gamma_{\pm} = -0.5062 \times (S_m)^{1/2} \quad (1)$$

Where,S_m is the molal solubility.

The solubility product which is the main parameter for evaluating the free energies was calculated by using equation (2) [48-50, 52-58].

$$pK_{sp} = -2 (\log S_m + \log \gamma_{\pm}) \quad (2)$$

This study uses simple equations for the evaluation of the thermodynamic parameters. Moreover. The transfer Gibbs free energies as another parameter for comparing the thermodynamic parameters were calculated from the values of solubility products and Gibbs free energy in mixed solvents by using equations 4 & 5 giving little significant data.

$$\Delta G = 2.303\ RT\ pK_{sp} \quad (3)$$

$$\Delta G_t = \Delta G_S - \Delta G_w \quad (4)$$

3.4. Enthalpies and Entropies of Solvation

This study in one of very few studies which investigated the thermodynamic s for nano particles in solutions. The enthalpies and entropies complete the behaviour of solvent in nano fluid formation. The entropy of solvation of nano ZnO and nano ZnO doped was determined from the slope for each solution percentage of the linear relation between ΔG *versus* T (Table 1). Also, we can determine the enthalpy change (ΔH) at 303.15 K as example, by using equation (5).Positive entropy values indicate that the solvation processes took place [58-79].

$$\Delta G_s = \Delta H_s - T\ \Delta S_s \quad (5)$$

3.5. Zeta Potential Measurement

The Zeta potential for nano ZnO in pure DMF was measured and found to be – 11.3 mv, this low value whether it is positive or negative indicate low solubility of the nano ZnO and moderate association of the particles. The data are given in followed Table. Also low conductivity value 0.50 m s / cm indicate also low ability for ionization in the solvent. Three peaks reported at -120, -10.5 and -75.3 m v as given below.

Zeta Potential Report
v2.3

Malvern

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Sample Details

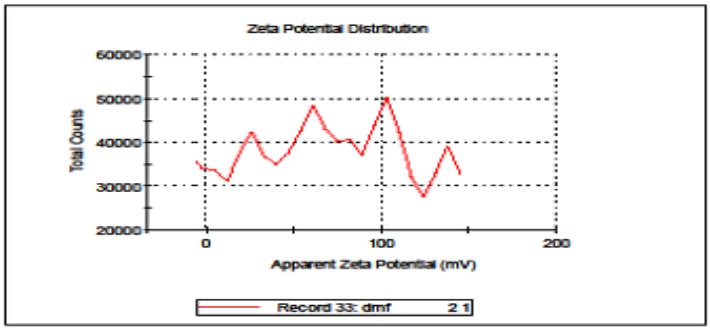
Sample Name:	dmf 2 1		
SOP Name:	mansettings.nano		
General Notes:			
File Name:	Dr. Esam A. Gomaa	**Dispersant Name:**	dmf
Record Number:	33	**Dispersant RI:**	1.320
Date and Time:	04	**Viscosity (cP):**	0.7690
		Dispersant Dielectric Constant:	36.7

System

Temperature (°C):	25.0	**Zeta Runs:**	20
Count Rate (kcps):	234.1	**Measurement Position (mm):**	2.00
Cell Description:	Clear disposable zeta cell	**Attenuator:**	9

Results

			Mean (mV)	Area (%)	St Dev (mV)
Zeta Potential (mV):	-11.3	**Peak 1:**	-120	19.4	17.3
Zeta Deviation (mV):	269	**Peak 2:**	-10.5	13.0	13.6
Conductivity (mS/cm):	0.507	**Peak 3:**	-75.3	12.6	11.6
Result quality	See result quality report				



4. CONCLUSION

The paper's primary contribution is finding the solubility decrease with rise of temperature. It was concluded that that both the solubility and free energies decrease with rise of temperature indicating less solubility and more aggregation of the solid materials. The enthalpy and entropy of solvation decrease by doping ZnO with 2% doped substances other than using silver in 50% DMSO-50% DMF solutions. We aim to illustrate the effect of doping metals to nano ZnO to facilitate their applications.

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