Phase Equilibria and Thermodynamic Description of Alloys of 8-Hydroxyquinoline – 4-Chlorobenzoic acid

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Abstract

In present article 8-hydroxyquinoline (HQ)-4-chlorobenzoic acid (CBA) system has been undertaken to study phase diagram and thermodynamic contribution in the system in terms of partial and integral mixing and excess functions. The phase equilibria of the system under taken shows the formation of 1:1 addition compound C and its eutectic E at 214º and199º c respectively. The negative value of molar free energy of mixing (G^M) of alloys at 0.810 and 0.895 mole fraction of CBA suggests that the mixing in these cases is spontaneous. The integral molar enthalpy of mixing value corresponds to the value of excess integral molar free energy of the system favors the regularity in the binary solutions. The positive value of excess free energy (g^E) for all the eutectic and noneutectic alloys suggests an association of weaker nature between unlike molecules and of stronger nature between like molecules. Gibbs-Duhem equation gives the graphical solution of partial molar heat of mixing(ΔH_i^{-M}), activity and activity coefficient of a particular constituent in the binary mix. Interfacial roughness of the binary alloys has also been explained.

Keywords : Organic alloys, phase diagram, activity, activity coefficient, mixing and excess function.

1. Introduction

The chemistry of organic materials, binary eutectics, noneutectics and molecular compounds has been the most active area of investigation due to their unusual anisotropic properties (Glazer, J. 1995,. Gachon, J.C. 1998) not normally shown by their parent components. The organic substances have recently been attracting the attention in connection with searches of new charge transfer materials in polymer matrix for various electronic devices and have been used as nonlinear materials for quantum electronics. These materials have considerably high nonlinearity, great possibility of varying the molecular structure, easier to engineer to fabricate instrument of high optical properties of the crystals, high resistance to high energy laser beams and are much cheaper than their inorganic counterparts. The eutectics, noneutectics and molecular compounds of metal origin have a remarkable commercial and technological importance in modern age. The idea behind single effort is to produce a material consists of very high strength but possibly brittle fibers embedded in a ductile matrix. In spite of great devotion to acquire the stronger and more reliable material to cater the ever growing need and diversified demand of modern civilization, the various studied on metallic eutectics, noneutectics and intermetallic compounds are inadequate and incomplete as high transformation temperature, opacity, difficulty involved in purification and experimentation, density driven convection which affects their solidification. In recent past extensive study (Sharma, B.L. Tandon, S. and Gupta, S. 2009, Tanvi Agarwal, Preeti Gupta, Shiva Sharma Das, Ashutosh Gupta and Nakshtra Bhadur Singh 2010) has been developed in the use of eutectic and noneutectic solidification to produce materials having two phase microstructure forming *in-situ* composites and thereby materials of desired properties. Due to low transformation temperature, transparency, ease in purification and experimentation, minimized convection effects and wider choice of materials are the special features which have prompted the authors to select a system having high enthalpy of fusions and solidify faceted or nonmetallic growth. 8-hydroxyquinoline (HQ)-4-cholorobenzoic acid (CBA) system has been taken for detailed physicochemical investigations, such as, phase diagram, activity, activity co-efficient, mixing and excess functions of binary organic crystals.

2. Experimental

2.1 Phase diagram study:

The phase diagram of HQ-CBA system was determined by thaw-melt method (Gupta, R.K. Singh, S.K. and Singh, R.A. 2007, Shekhar, H. Salim, S.S. and Kumar, N. 2009). Mixtures of different compositions were made in glass test tubes by repeated heating followed by chilling in ice. The melting and thaw temperatures were determined in a Toshniwal melting point apparatus using a precision thermometer.

2.2 Materials and their purification :

8-hydroxyquinoline (E.Merck, Bombay) was purified by fractional crystallization of ethanol and 4-chlorobenzoic acid (G.S. Chemical, New Delhi) was used directly as supplied. The purity of the compounds was confirmed by determining their melting points which were in good agreement with those quoted in reference literature.

2.3 Heat of fusion:

Heats of fusions of pure components and their eutectic and noneutectic alloys and addition compound were measured by DTA method (Sangstar, J. 1994) using a Stanton Redcroft STA-780 series unit.

3. Results and Discussion

3.1 Phase diagram:

The phase diagram of HQ-CBA system determined by thaw-melt method is reported in the form of temperature-composition curve. The system shows the formation of an addition compound, C(214^{0}C) and one eutectic. The eutectic at the eutectic temperature a binary solution (L) is in equilibrium with two solid phases. The melting points of HQ is 73^{0}C and it increases with the addition of the second component CBA (235^{0}C) and attains maximum and then decreases to minimum. Eutectic alloy E 0.790 mole fraction of CBA is obtained at 199^{0}C. At the eutectic temperature phases namely a liquid phase L and two solid phases are in equilibrium and the system is invariant. In the region indicated by L a homogeneous binary liquid solution exists while the two solid phases exists below the horizontal line. In the case, in HQ+L region located on the extreme left side of the diagram a binary liquid and solid HQ exist while in a similar region located on the extreme right side of the diagram a binary liquid and the second component of the system co-exist. At maximum temperature at mole fraction of CBA an intermediate phase is a molecular complex is formed which on decomposition gives

$$(HQ)_1\ (CBA)_2\ (L) \quad \overset{\text{cooling}}{\rightleftharpoons} \quad 1\ HQ\ (S) + 2\ CBA(S)$$

The formation of molecular compound (1:1) with congruent melting point shows a characteristic maximum C. Near the C, the compound C with liquid CBA in the right region coexist. Below the horizon, at left hand side a mixture of two solids HQ and C and below horizon at right hand side mixture of CBA and C have been found. The system at eutectic temperature T_e has only one component and is consequently invariant. The better hump and sharp inclination in the intermediate region in the phase diagram infers the formation of a definite compound and possess the regarding stability of addition of CBA in HQ with eutectic alloy.

3.2 Thermochemistry:

The molecular complex is association of two or more molecules in definite stoichiometry and the association is some what stronger than vander Wall's associations. The chemical interaction between electron donor and electron acceptor molecules leads to an association of molecules in definite proportions to form molecular complex. In the formation of molecular complex, physical as well as chemical forces are involved. Thermochemical studies unfold the nature of interactions between components forming the addition compound.

3.3 Heat of fusion:

The values of heats of fusion of eutectic, noneutectic and molecular complex are calculated by the mixture law using equation

$$(\quad H)_e = x_1 \quad H_1 + x_2 \quad H_2 \qquad \textbf{...(1)}$$

where 1 belongs for HQ and 2 belongs for CBA. x and H are the mole fraction and heat of fusion of the component indicated by the subscript, respectively. The value of heat of fusion of binary alloys $A_1 - A_{11}$, E and compound C($A_{1:1}$) is reported in Table1. The difference in experimental and calculate value of enthalpy of fusion of alloys suggests the association characteristics between components.

3.4 Entropy of fusion:

The entropy of fusion (S) of pure components and the eutectic, noneutectic alloys and molecular complex can be calculated using the following equation

$$S = \frac{\Delta H}{T} \qquad \textbf{...(2)}$$

where H is the heat of fusion and T is the fusion temperature. The values for the entropy of fusion (Table1) are positive in all cases, this points to an increase in the randomness (Shukla, B.M. and Singh, N.P. 1985)of the system during melting and infers both factors namely energy and entropy favour the melting process of components, alloys and compound.

3.5 Activity and activity coefficient:

For ideal mixture, the composition and temperature of alloy can be predicted the activity coefficient by solving the following equation

$$- \ln (x_1)_e = \frac{\Delta H_1}{R} \quad \frac{1}{T_e} \quad \frac{1}{T_1} \qquad \text{...(3)}$$

$$- \ln (x_2)_e = \frac{\Delta H_2}{R} \quad \frac{1}{T_e} \quad \frac{1}{T_2} \qquad \text{...(4)}$$

where T_e is the temperature of alloys and T_1 and T_2 are the melting points of components HQ and CBA, respectively. The activity coefficient of components HQ and CBA, for the systems ubder investigation may be calculated from the equation given below:

$$-\ln x_i^l \quad {}_i^l = \frac{\Delta H_i}{R} \left(\frac{1}{T_e} - \frac{1}{T_i} \right) \qquad \text{...(5)}$$

where x_i^l and ${}_i^l$ are the mole fraction and activity coefficient of the component i in the liquid phase respectively, H_i is the heat of fusion of component i at melting point T_i and R is the gas constant. The values of activity and activity coefficient of the components given Table2.

3.6 Gibbs- Duhem equation:

The partial molar quantity, activity and activity coefficient can also be determined by using Gibbs-Duhem equation

$$\Sigma x_i \, dz_i^- = 0$$

$$\text{Or,} \quad x_1 dH_1^{-M} + x_2 dH_2^{-M} = 0 \qquad \text{...(6)}$$

$$\text{Or,} \quad dH_2^{-M} = \frac{X_1}{X_2} dH_1^{-M}$$

$$\text{Or,} \quad [H_2^{-M}]_{x_2 = y} = \int_{x_2 = y}^{x_2 = 1} \frac{x_1}{x_2} dH_1^{-M} \qquad \text{...(7)}$$

where 1 belongs for HQ and 2 belongs for CBA. Using eqn. (7) a graph (Fig.2) between x_1/x_2 and H_1^{-M} gives the solution of the partial molar heat of mixing of a particular constituent in the binary alloys and plots between x_1/x_2 vs $\ln a_1$ and $\ln {}_1$ (Fig.3) determine the value of activity and activity coefficient of a specific component in binary alloys respectively.

3.7 Mixing functions:

Integral molar free energy of mixing (G^M), molar entropy of mixing (S^M) and molar enthalpy of mixing (H^M) and partial thermodynamic mixing functions of the binary alloys when two components are mixed together were determined by using the following equations

$$G^M = RT (x_1 \ln a_{11} + x_2 \ln a_{21}) \qquad \text{...(8)}$$

$$S^M = -R (x_1 \ln x_{11} + x_2 \ln x_{21}) \qquad \text{...(9)}$$

$$H^M = RT (x_1 \ln {}_{11} + x_2 \ln {}_{21}) \qquad \text{...(10)}$$

$$Gi^{-M} = i^{-M} = RT \ln ai_l \qquad \text{...(11)}$$

where Gi^{-M} (i^{-M}) is the partial molar free energy of mixing of component i (mixing chemical potential) in binary mix. and ${}_i$ and a_i is the activity coefficient and activity of the component, respectively. The negative value (Wisniak, J. and Tamir, A. 1978, Shekhar, H. Pandey, K.B. and Vishnu Kant 2010, Shekhar, H. and Salim, S.S. 2011) of molar free energy of mixing of alloys A_{10} and A_{11} (table3) suggests that the mixing in all cases is spontaneous. The integral molar enthalpy of mixing value corresponds to the value of excess integral molar free energy of the system favors the regularity in the binary solutions. The positive value of S^M of all alloys predicts the increase of randomness during mixing due to higher amplitude of phase distribution between the components forming the binary melt.

3.8 Excess thermodynamic functions:

In order to unfold the nature of the molecular interactions between the components forming the eutectic, noneutectic alloys and molecular complex, the excess thermodynamic functions, such as, excess integral free energy (g^E), excess integral entropy (s^E) and excess integral enthalpy (h^E) were calculated using the following equations

$$g^E = RT\,(x_1 \ln\gamma_1^1 + x_2 \ln\gamma_2^1) \qquad \text{...(12)}$$

$$s^E = -R\left(x_1 \ln\gamma_1^1 + x_2 \ln\gamma_2^1 + x_1 T \frac{\delta \ln\gamma_1^1}{\delta T} + x_2 T \frac{\delta \ln\gamma_2^1}{\delta T}\right) \qquad \text{...(13)}$$

$$h^E = -RT^2 \left(x_1 \frac{\delta \ln\gamma_1^1}{\delta T} + x_2 \frac{\delta \ln\gamma_2^1}{\delta T}\right) \qquad \text{...(14)}$$

and excess chemical potential or excess partial free energy of mixing

$$g_i^{-E} = \mu_i^{-E} = RT \ln\gamma_i^1 \qquad \text{...(15)}$$

The values of $\ln\gamma_i^1$/ T can be determined by the slope of the liquidus curve near the alloys from in the phase diagram. The values of excess thermodynamic functions are given in Table 4. The value of the excess free energy is a measure of the departure of the system from ideal behavior. The reported excess thermodynamic data substantiate the earlier conclusion of an appreciable interaction (Shekhar, H. and Pandeya, K.B. 2008, Rai, R.N. and Reddi, R.S.B. 2009) between the parent components during the formation of alloys. The positive value in the present system suggest an association of weaker nature between unlike molecules and of stronger nature between like molecules. However, its sign provides informati on regarding a change in density of the alloys during the phase transformation. The positive value of excess entropy is a measure of the change in configurational energy due to a change in potential energy and indicates an increase in randomness.

4. Interfacial Roughness

The microstructure of a material has been found to be significant in deciding its mechanical, electrical, magnetic and optical properties. The growth morphology of organic binary alloys during solidification depends on the growth characteristics of individual constituent phases which solidify with either faceted (nonmetallic) or nonfaceted (metallic) interfaces. This behaviour is related to the nature of the solid- liquid interface and can be predicted from the value of the entropy of fusion. According to Hunt and Jackson (Hunt, J.D. and Jackson, K.A. 1966) the type of growth from eutectic melt depends upon a factor , defines as,

$$= \frac{\Delta H}{RT} = \frac{\Delta S}{R} \qquad \text{...(16)}$$

where is a crystallographic factor depending upon the geometry of the molecules and has a value less than or equal to one. S / R ( ) is known as Jackson's roughness parameter and R is the gas constant. When is greater than two, the solid-liquid interface is a atomically smooth and exhibits faceted growth (Rai, U.S. and Pinky Pandey 2003, Shekhar, H. and Pandey, K.B. 2010). When is less than two the solid-liquid interface is atomically rough and leads nonfaceted growth. The value of (Table1) of all the alloy under investigation are greater than two indicate that they exhibit faceted growth. Further a prediction of microstructure of eutectic can be made from Splingler's equation [10]

$$= \frac{T_1 - T_e}{T_2 - T_e} \qquad \text{...(17)}$$

where T_1 and T_2 are the melting temperature of low melting and high melting component, respectively and Te is the eutectic temperature. The normal eutectics are formed when lies between 0.1 and 1.0 but it lies between 0.01 to 0.1 anomalous structure is obtained. The microstructure of the simple eutectic of HQ-CBA system containing mole fraction of CBA at 0.790 appears with normal faceted growth.

5. Conlusion

The phase diagram of the HQ-CBA system shows the formation of 1:1 molecular complex with only one eutectic. The experimental observation of alloy growth indicates that a binary melt solidifies spontaneously only below its equilibrium temperature. The Gibbs-Duhem equation gives the solution of partial molar enthalpy of mixing, activity and activity coefficient of a component in binary mix. The value of surface roughness is greater than two gives indication about faceted growth in the alloys.

Acknowledgement

Authors thank the Head, Department of Chemistry, V.K.S. University, Ara-802301, India for providing research facilities.

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Table 1. Tempreture-composition data, values of heat of fusion(ΔH), entropy of fusion(ΔS) and interfacial roughness(α) of HQ-CBA system.

Alloys & Compound	Mole fraction of CBA	M.P (°C)	ΔH (kJ/mol)	ΔS (J/mol/K)	α
A_1	0.095	87	19.27	53.53	6.44
A_2	0.190	145	19.84	47.46	5.71
A_3	0.285	194	20.41	43.70	5.26
A_4	0.335	199	20.71	43.88	5.28
A_5	0.480	211	21.58	44.59	5.36
$(A_{1:1})$	0.500	214	21.70	44.56	5.36
A_6	0.520	213	21.82	44.90	5.40
A_7	0.580	210	22.18	45.92	5.52
A_8	0.685	204	22.81	47.82	5.75
A_9	0.765	201	23.29	49.13	5.91
E	0.790	199	23.44	49.66	5.97
A_{10}	0.810	201	23.56	49.70	5.98
A_{11}	0.895	208	24.07	50.04	6.02
HQ	0.000	73	18.70	54.05	6.50
CBA	1.000	235	24.70	48.62	5.85

Table 2. Values of activity and activity coefficient of components in binary melt of HQ-CBA system.

Alloys	ln ${}_1$	ln ${}_2$	${}_1$	${}_2$	$\ln a_1$	$\ln a_2$	a_1	a_2	x_1/x_2
A_1	0.35	0.05	1.42	1.05	0.25	2.30	1.28	0.10	9.53
A_2	1.33	0.40	3.78	1.49	1.12	-1.27	3.06	0.28	4.26
A_3	2.02	0.74	7.54	2.10	1.68	-0.51	5.39	0.60	2.51
A_4	2.14	0.65	8.52	1.91	1.73	-0.45	5.66	0.64	1.98
A_5	2.51	0.45	12.27	1.56	1.85	-0.29	6.38	0.75	1.08
$C(A_{1:1})$	2.57	0.44	13.06	1.55	1.87	-0.26	6.53	0.77	1.00
A_6	2.61	0.39	13.60	1.48	1.88	-0.26	6.53	0.77	0.92
A_7	2.71	0.79	15.03	2.20	1.84	0.25	6.31	1.28	0.72
A_8	2.94	-0.00	18.91	1.00	1.78	-0.39	5.96	0.68	0.46
A_9	3.20	-0.15	24.53	0.86	1.75	-0.41	5.76	0.66	0.31
E	3.29	-0.21	26.84	0.81	1.73	-0.45	5.64	0.64	0.27
A_{10}	3.41	-0.21	30.26	0.81	1.75	-0.43	5.75	0.65	0.23
A_{11}	4.08	-0.19	59.14	0.83	1.83	-0.30	6.21	0.74	0.12

Table 3. Values of partial and integral thermodynamic mixing functions of HQ-CBA system.

Alloys & Compound	G_1^{-M} (J/mol)	G_2^{-M} (J/mol)	ΔG^M (J/mol)	S_1^{-M} (J/mol/K)	S_2^{-M} (J/mol/K)	ΔS^M (J/mol/K)	H_1^{-M} (J/mol)	H_2^{-M} (J/mol)	ΔH^M (J/mol)
A_1	739.28	-6889.98	14.50	0.83	19.57	2.61	1038.08	155.22	962.26
A_2	3885.33	-4423.99	2306.56	1.75	13.81	4.04	4616.83	1348.59	4008.01
A_3	6538.36	-1984.03	4109.48	2.79	10.44	4.97	7841.29	2891.45	6426.54
A_4	6800.65	-1750.20	3936.12	3.39	9.09	5.30	8400.73	2540.28	6439.04
A_5	7456.43	-1158.90	3321.07	5.44	6.10	5.76	10089.39	1793.50	6121.27
$C (A_{1:1})$	7595.77	-1056.77	3269.50	5.76	5.76	5.76	10400.89	1748.35	6074.62
A_6	7596.33	-1050.56	3099.95	6.10	5.44	5.76	10560.93	1593.28	5881.50
A_7	7396.85	991.87	3681.96	7.21	4.53	5.66	10879.28	3179.86	6410.60
A_8	7078.91	-1530.79	1181.27	9.60	3.14	5.17	11658.11	-33.01	3667.27
A_9	6896.46	-1615.74	384.63	12.04	2.23	4.53	12603.42	-558.72	2511.30
E	6788.88	-1750.20	43.01	12.97	1.96	4.27	12910.72	-825.08	2060.21
A_{10}	6896.46	-1694.56	-62.26	13.81	1.75	4.04	13442.40	-865.06	1882.93
A_{11}	7302.24	-1203.71	-310.58	18.74	0.92	2.79	16316.18	-761.19	1033.15

Table 4. Values of excess partial and integral thermodynamic functions of HQ-CBA system.

Alloys & Compound	g_1^{-E} (J/mol)	g_2^{-E} (J/mol)	g^E (J/mol)	s_1^{-E} (J/mol/K)	s_2^{-E} (J/mol/K)	s^E (J/mol/K)	h_1^{-E} (kJ/mol)	h_2^{-E} (kJ/mol)	h^E (kJ/mol)
A_1	1047.56	149.65	962.26	-38.33	-6.40	-35.75	-12.93	-2.15	-11.91
A_2	4622.08	1390.10	4008.01	-47.20	-25.91	-43.15	-15.11	-9.44	-14.03
A_3	7842.93	2873.15	6426.54	-43.97	75.38	-9.96	-12.69	38.08	1.78
A_4	8397.80	2550.73	6439.04	1.83	57.38	20.24	9.26	29.63	15.99
A_5	10100.18	1810.79	6121.27	19.37	28.45	23.73	19.48	15.58	17.61
C ($A_{1:1}$)	10405.72	1781.52	6093.62	21.15	-13.78	3.68	20.70	-4.93	7.89
A_6	10545.98	1575.83	5881.50	107.60	108.81	104.59	62.84	51.06	56.71
A_7	10882.44	3172.37	6410.60	33.69	11.50	20.82	27.15	8.73	16.47
A_8	11659.39	-7.93	3667.27	62.80	5.96	23.87	41.62	2.84	15.05
A_9	12610.67	-591.12	2511.30	268.96	24.89	81.36	140.10	11.21	41.08
E	12910.64	-824.08	2060.21	116.60	-2.18	23.02	68.53	-1.85	12.93
A_{10}	13438.25	-827.56	1882.93	141.10	21.45	44.18	80.32	9.34	22.83
A_{11}	16316.06	-759.82	1033.15	304.67	-26.30	8.45	163.50	-13.46	5.12

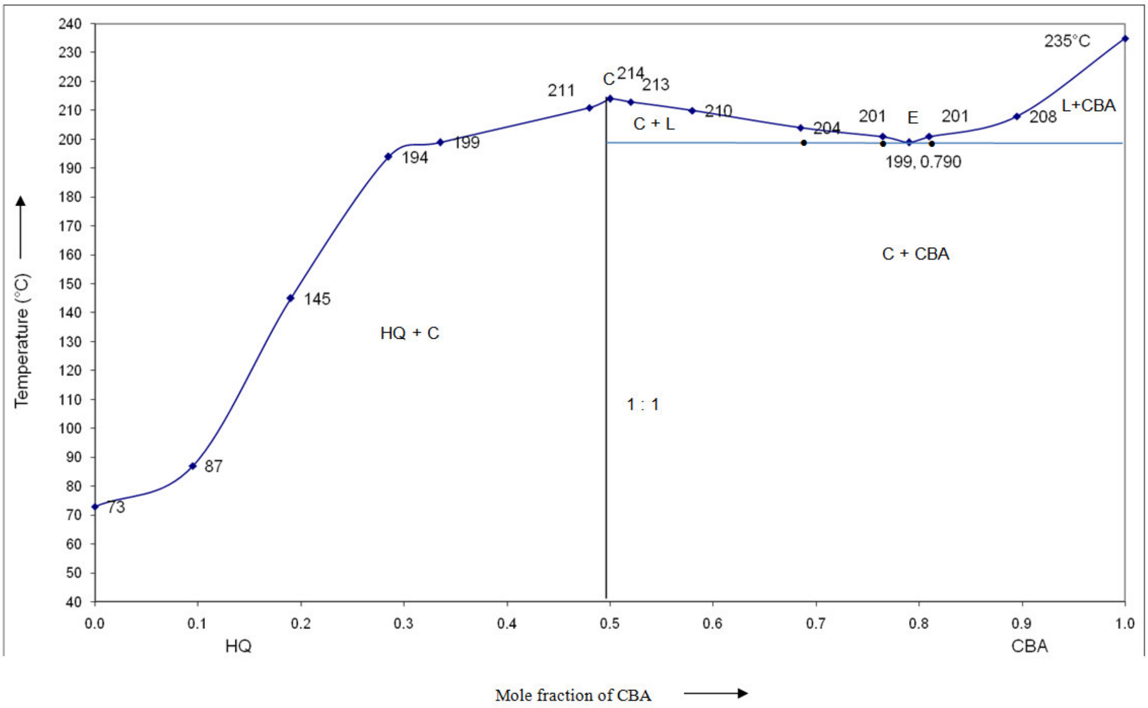

Figure 1. Phase diagram of 8-hydroxyquinoline (HQ) – 4-chlorobenzoic acid (CBA) system

- • Thaw temperature
- ♦ Melting temperature

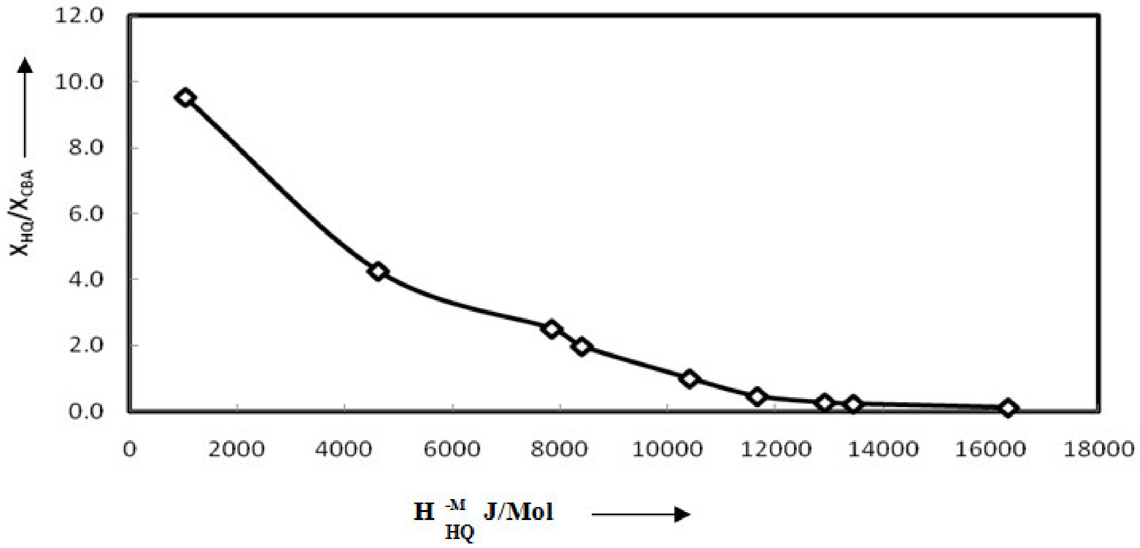

Figure 2. Graphical solution of partial molar heat of mixing of CBA in binary mixture

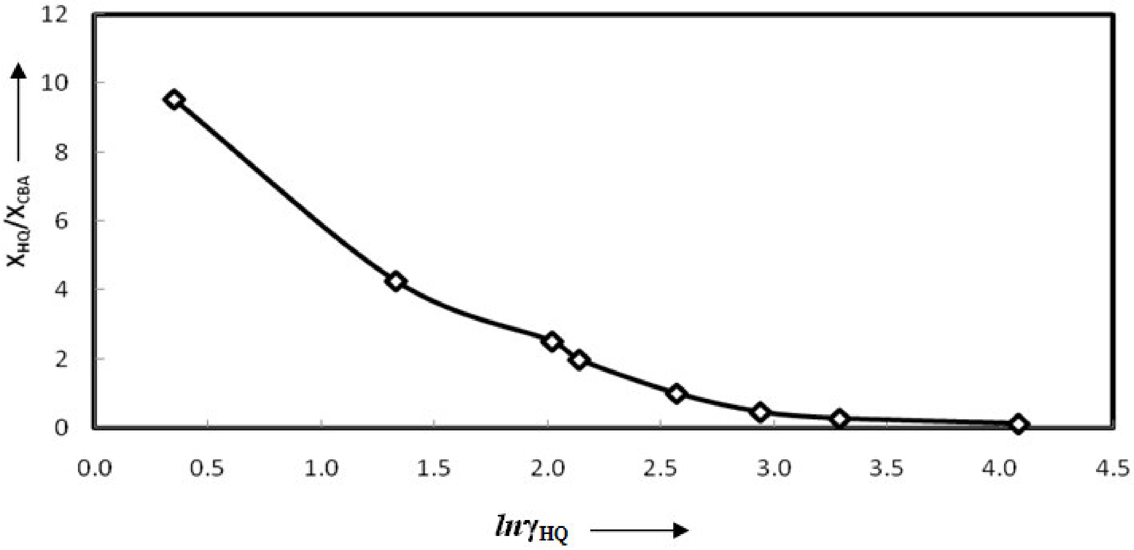

Figure 3. Graphical solution of activity coefficient of CBA in binary mixture

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