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Volumetric studies and thermodynamics of viscous flow of hydroxamic acids in acetone + water solvent at temperatures 303.15 and 313.15 K

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Abstract

Densities ρ and viscosities η of two hydroxamic acids, *N*-phenyl-2-chlorobenzo- and *N*-*o*-tolyl-4-chlorobenzo-, have been determined as a function of their concentration in aqueous acetone solution at temperatures 303.15 and 313.15 K. Apparent molar volumes, standard-state partial molar volumes and relative viscosities have been calculated. The viscosity data have been analyzed using Jones–Dole equation. The activation thermodynamic parameters of viscous flow have been evaluated using Feakins equation. These were obtained to throw light on the mechanism of viscous flow. Thermodynamic interactions in solutions have been studied in terms of a number of excess functions calculated from the experimental data. The effect of hydroxamic acid concentration and temperature on these parameters has been discussed. The results were interpreted in the light of solute–solvent interactions in aquo-organic media.

Keywords: Excess molar volume; Viscosity *B*-coefficient; Activation thermodynamic parameters; Hydroxamic acid

1. Introduction

The hydroxamic acid functional group, –C(=O)NROH, is a key structural constituent of many biomolecules, some of which are naturally occurring [1] and others, such as the peroxidase, matrix metalloproteinase and urease inhibitors [2,3], are of synthetic origin. These reagents show a wide spectrum of activities in analytical, agricultural, biological and medicinal fields. The versatile biological activity of these compounds is due to their strong metal ion chelating ability [1], their nitric oxide releasing ability [4] and to provide sites for acylation [5]. Hydroxamic acids can also act as anticancer [6], antiinfectives [7] and antitumor agents [8]. Experimental data of physical properties such as density and viscosity of hydroxamic acids in mixed solvent give information on the existence of specific molecular interactions. Investigations on thermodynamic properties can provide valuable information that ultimately leads to a better understanding of various interactions taking place in solutions of nonelectrolytes and the temperature dependence of these interactions.

2. Experimental

Both the hydroxamic acids were prepared in the laboratory following the procedure reported in literature [9]. The solutes were purified by recrystallization thrice with benzene and dried over phosphorus pentoxide in vacuum desiccator for several hours. The purity of these compounds was ascertained by determining the melting point, U.V. and I.R. spectrum, which tally with the literature data [10]. Stock solution of 0.05 M was prepared by dissolving hydroxamic acids in 100 ml 40% (v/v) acetone + water mixed solvent. Hydroxamic acids are insoluble in aqueous phase therefore a mixed solvent system was used. Solutions of varying concentrations from 0.01 to 0.05 M were then prepared from stock solution by mass dilution technique. Uncertainties in solution concentrations were estimated to be ±0.001 units.

Densities of mixed solvent (acetone + water, 40%, v/v) and hydroxamic acid solutions were determined using a 10 cm^3 double-armed pycnometer at temperatures 303.15 and 313.15 K. The pycnometer was calibrated at both the temperatures with water. The estimated accuracy of density measurements of solutions was $\pm 0.03\,kg\,m^{-3}$. The reproducibility of density measurements was $\pm 0.04\,kg\,m^{-3}$.

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The viscosity measurements were made using a commercial capillary viscometer of an Ostwald–Sprengel type. The viscometer was submerged in a thermostatic bath (Maharana, Instrument Manufacturing Company, Ajmer, India) at temperatures (298.15, 303.15 and 313.15 K) with a resolution of ±0.01 K and 10 cm^3 of the solution was added from a pipette. The viscosity values were determined using the relation

$$\eta = \rho\left(Kt - \frac{L}{t}\right) \tag{1}$$

where η is the viscosity, ρ the density of the liquid, t the flow time, and K and L are the constants for a given viscometer. The flow time was measured with an electronic stopwatch capable of measuring time within ±0.01 s. At least three repetitions of each data set reproducible to 0.05 s were obtained. The values of constants K and L were obtained by measuring the flow times of pure water at three temperatures (298.15, 303.15 and 313.15 K) using the density and viscosity values of water from literature [11]. Linear regression analysis of a plot of $\eta t/\rho$ against t^2 for distilled water at three temperatures provided estimates of $K = 2.45 \times 10^{-9}\ \mathrm{m^2\,s^{-2}}$ and $L = -0.87986 \times 10^{-4}\ \mathrm{m^2}$, as slope and intercept of the plot. The estimated accuracy of experimental viscosities was ±0.001 mPa s.

3. Results

The measured values of densities and viscosities of both the hydroxamic acids in 40% (v/v) acetone + water mixed solvent at temperatures 303.15 and 313.15 K are listed in Table 1.

3.1. Volumetric studies

Table 2
Standard-state partial molar volume V_ϕ° and experimental slope S_V in 40% (v/v) acetone + water

Hydroxamic acid	V_ϕ° ($\times 10^{-6}\ \mathrm{m^3\,mol^{-1}}$)	S_V ($\times 10^{-6}\ \mathrm{m^3\,dm^{3/2}\,mol^{-3/2}}$)
N-Phenyl-2-chlorobenzo		
303.15 K	158.7059	−224.3134
313.15 K	184.1438	−301.1734
N-*o*-Tolyl-4-chlorobenzo		
303.15 K	202.2387	−325.4570
313.15 K	245.0398	−508.6125

The experimental values of density were used to calculate the apparent molar volumes V_ϕ of hydroxamic acids in mixed solvent using the following relation:

$$V_\phi = \frac{M_2}{\rho} - 1000\frac{\rho - \rho_1}{c\rho_1\rho} \tag{2}$$

where c is the molarity of hydroxamic acid in solution, M_2 the molar mass of the nonelectrolyte, and ρ_1 and ρ are the densities of the mixed solvent and solution, respectively. The resulting values of V_ϕ are reported in Table 1. The standard-state (infinite dilution) apparent molar volume were obtained by least squares fitting to the equation [12]

$$V_\phi = V_\phi^\circ + S_\mathrm{V} c^{1/2} \tag{3}$$

in which S_V is the experimental slope. The calculated values of V_ϕ° and S_V are given in Table 2. From the measured values of density, molar volume (V) was calculated using the relation,

$$V_\mathrm{m} = \frac{x_1 M_1 + x_2 M_2}{\rho} \tag{4}$$

where x_1, M_1 and x_2, M_2 are the mole fraction and molecular weight of the mixed solvent and solute, respectively. The solvent mixture was treated as a pure solvent and its molar volume ($V_{\mathrm{m},1}$) was taken as a mean value, which is defined as [13]

$$V_{\mathrm{m},1} = \frac{x_1^0 M_1^0 + x_2^0 M_2^0}{\rho_1} \tag{5}$$

Table 1
Densities ρ, viscosities η and apparent molar volume V_ϕ in 40% (v/v) acetone + water

c (mol $\mathrm{dm^{-3}}$)	ρ ($\times 10^3\ \mathrm{kg\,m^{-3}}$)		η (mPa s)		V_ϕ ($\times 10^{-6}\ \mathrm{m^3\,mol^{-1}}$)	
	303.15 K	313.15 K	303.15 K	313.15 K	303.15 K	313.15 K
N-Phenyl-2-chlorobenzohydroxamic acid						
0.000	0.9406	0.9384	1.0602	0.8967	–	–
0.010	0.9416	0.9393	1.0636	0.9094	148.4332	164.8944
0.020	0.9433	0.9408	1.0759	0.9163	113.0391	129.5642
0.030	0.9446	0.9421	1.0883	0.9253	112.9328	125.3208
0.035	0.9452	0.9427	1.0928	0.9315	114.8626	125.7483
0.040	0.9458	0.9433	1.1069	0.9387	116.4268	126.1099
0.050	0.9471	0.9446	1.1226	0.9504	116.5579	124.4171
N-*o*-Tolyl-4-chlorobenzohydroxamic acid						
0.000	0.9406	0.9384	1.0602	0.8967	–	–
0.010	0.9414	0.9390	1.0690	0.9055	182.4369	215.1977
0.020	0.9430	0.9407	1.0789	0.9070	142.1199	149.9583
0.030	0.9443	0.9419	1.0897	0.9070	138.7633	145.8525
0.035	0.9449	0.9426	1.0972	0.9136	138.6565	144.2711
0.040	0.9456	0.9431	1.1058	0.9159	138.8175	146.2399
0.050	0.9468	0.9442	1.1201	0.9256	138.9412	146.2316

Table 3
Molar volume V_m, relative viscosities η_r and Bc (2.5υ) in 40% (v/v) acetone + water

c (mol dm^{-3})	V_m ($\times 10^{-6}$ m^3 mol^{-1})		η_r		Bc (2.5υ)	
	303.15 K	313.15 K	303.15 K	313.15 K	303.15 K	313.15 K
N-Phenyl-2-chlorobenzohydroxamic acid						
0.010	25.1206	25.1696	1.0032	1.0144	0.0032	0.0144
0.020	25.1365	25.1896	1.0148	1.0221	0.0148	0.0221
0.030	25.1606	25.2148	1.0265	1.0321	0.0265	0.0321
0.035	25.1742	25.2288	1.0307	1.0390	0.0307	0.0390
0.040	25.1880	25.2428	1.0440	1.0470	0.0440	0.0470
0.050	25.2131	25.2681	1.0588	1.0600	0.0588	0.0600
N-*o*-Tolyl-4-chlorobenzohydroxamic acid						
0.010	25.1289	25.1818	1.0083	1.0100	0.0083	0.0100
0.020	25.1507	25.1997	1.0173	1.0117	0.0173	0.0117
0.030	25.1797	25.2302	1.0278	1.0190	0.0278	0.0190
0.035	25.1949	25.2450	1.0349	1.0210	0.0349	0.0210
0.040	25.2103	25.2629	1.0430	1.0325	0.0430	0.0325
0.050	25.2410	25.2967	1.0565	1.0405	0.0565	0.0405

where x_1^0, M_1^0 and x_2^0, M_2^0 are the mole fractions and molecular weights of water and acetone, respectively and ρ_1 is the density of the mixed solvent. The values of molar volume are listed in Table 3.

3.2. Studies on viscous flow

The solute–solvent interaction can be discussed through the change of dynamic property such as viscosity. For dilute solutions, the viscosities were analyzed by the empirical Jones–Dole equation [14]

$$\eta_r = \frac{\eta}{\eta_1} = 1 + Ac^{1/2} + Bc \qquad (6)$$

where η_1 and η are the viscosities of mixed solvent and solution, respectively, η_r the relative viscosity, c the concentration and A and B are the adjustable parameters. The A coefficient arises from ion–ion interaction and the B-coefficient from ion–solvent interaction. In case of nonelectrolytes, $A = 0$. This reduces the Jones–Dole equation to

$$\eta_r = \frac{\eta}{\eta_1} = 1 + Bc \qquad (7)$$

The plots of η_r versus c were found to be linear for both the hydroxamic acid solutions. The B-coefficient obtained as slope of the straight lines has been recorded in Table 4. Einstein [15] proposed an equation,

Table 4
Viscosity B-coefficients and dB/dT in 40% acetone + water

Hydroxamic acids	B-coefficient (m^3 mol^{-1})	dB/dT
N-Phenyl-2-chlorobenzo		
303.15 K	1.3818 (±0.1009)	
313.15 K	1.1563 (±0.0759)	−0.0226
N-*o*-Tolyl-4-chlorobenzo		
303.15 K	1.2131 (±0.0664)	−0.0418
313.15 K	0.7948 (±0.1229)	

$$\eta = \eta_1(1 + 2.5\upsilon) \qquad (8)$$

which describes the concentration dependence of the relative viscosity of solution of nonelectrolytes. υ is the aggregate volume of the particles in a unit volume of the solution. The term 2.5υ is taken to be valid for nonelectrolytes, and it is equivalent to the product Bc in the Jones–Dole equation.

3.3. Thermodynamics of viscous flow

In Eyring's simple model [16], the molecules move one-by-one from their equilibrium positions through their transition states, in which intermolecular bonds are stretched, to other equilibrium positions. For a solvent he found the equation

$$\Delta\mu_1^{0\neq} = RT\,\ln\left(\frac{\eta_1 \bar{V}_1^0}{hN}\right) \qquad (9)$$

where T is the absolute temperature, h the Planck's constant, N the Avogadro's number, and $\Delta\mu_1^{0\neq}$ is the contribution per mole of solvent to the free energy of activation for viscous flow of the solution. The activation energy per mole of solution can be written as,

$$\Delta G^{0\neq} = x_1\,\Delta\mu_1^{0\neq} + x_2\,\Delta\mu_2^{0\neq} \qquad (10)$$

where $\Delta\mu_2^{0\neq}$ is the contribution per mole of solute to the Gibbs energy change of activation of viscous flow of the solution.

The viscosity data have also been analyzed on the basis of a transition state theory of the relative viscosity as suggested by Feakins et al. [17], the B-coefficient is related to $\Delta\mu_2^{0\neq}$ by equation,

$$B = \frac{\bar{V}_1^0 - \bar{V}_2^0}{1000} + \frac{\bar{V}_1^0}{1000}\,\frac{\Delta\mu_2^{0\neq} - \Delta\mu_1^{0\neq}}{RT} \qquad (11)$$

Table 5
Activation parameter, $\Delta\mu_1^{0\neq}$ of 40% (v/v) acetone + water and $\Delta\mu_2^{0\neq}$, $\Delta H_2^{0\neq}$, $\Delta S_2^{0\neq}$ of hydroxamic acids

c (mol dm^{-3})	$\Delta\mu_1^{0\neq}$ (kJ mol^{-1})		$\Delta\mu_2^{0\neq}$ (kJ mol^{-1})		$\Delta H_2^{0\neq}$ (kJ mol^{-1})		$\Delta S_2^{0\neq}$ (kJ mol^{-1} K^{-1})
	303.15 K	313.15 K	303.15 K	313.15 K	303.15 K	313.15 K	
N-Phenyl-2-chlorobenzohydroxamic acid							
0.010	62.8181	64.4593	214.8658	200.5152	42.0690	43.5040	1.4350
0.020	62.8197	64.4614	214.7701	200.4075	42.1038	43.5401	1.4362
0.030	62.8221	64.4640	214.6246	200.2715	42.0763	43.5116	1.4357
0.035	62.8235	64.4654	214.5422	200.1962	42.0552	43.4898	1.4346
0.040	62.8249	64.4669	214.4592	200.1209	42.0327	43.4665	1.4338
0.050	62.8274	64.4695	214.3086	199.9848	42.9901	43.4225	1.4324
N-*o*-Tolyl-4-chlorobenzohydroxamic acid							
0.010	62.8190	64.4606	202.2577	169.3548	96.4548	99.7451	3.2903
0.020	62.8211	64.4624	202.1367	169.2802	96.3189	99.6046	3.2856
0.030	62.8240	64.4656	202.9765	169.1537	96.2200	99.5023	3.2823
0.035	62.8256	64.4671	201.8926	169.0919	96.1550	99.4351	3.2801
0.040	62.8271	64.4689	201.8076	169.0178	96.1232	99.4021	3.2789
0.050	62.8302	64.4724	201.6385	168.8785	96.0361	99.3121	3.2760

It can be rearranged as follows:

$$\Delta\mu_2^{0\neq} = \Delta\mu_1^{0\neq} + \frac{RT}{\bar{V}_1^0}\{1000B - (\bar{V}_1^0 - \bar{V}_2^0)\} \qquad (12)$$

where $\bar{V}_1^0$ is the partial molar volume of the solvent and $\bar{V}_2^0$ is the partial molar volume of solute at infinite dilution.

The activation entropy and enthalpy contributions due to solute were evaluated following the equation [18]:

$$\frac{-\mathrm{d}(\Delta\mu_2^{0\neq})}{\mathrm{d}T} = \Delta S_2^{0\neq} \qquad (13)$$

and

$$\Delta H_2^{0\neq} = \Delta\mu_2^{0\neq} + T\,\Delta S_2^{0\neq} \qquad (14)$$

The values of solvent and solute activation parameters are given in Table 5.

The excess volumes (V^{E}) of these solutions have been computed using the relation,

$$V^{\mathrm{E}} = V - (x_1V_1 + x_2V_2) \qquad (15)$$

where V, V_1 and V_2 are the molar volume of solution, mixed solvent and solute, respectively. The excess viscosities have been derived from the viscosities using the relation,

$$\eta^{\mathrm{E}} = \eta - (x_1\eta_1 + x_2\eta_2) \qquad (16)$$

where η, η_1 and η_2 are the viscosities of solution, mixed solvent and solute, respectively. The excess volume and excess viscosity data are given in Table 6. According to Grunberg and Nissan [19], the adjustable parameter, d, mentioned in Eq. (17) is regarded as a measure of strength of interactions between the components of the solution.

$$\ln\eta = x_1\,\ln\eta_1 + x_2\,\ln\eta_2 + x_1x_2d \qquad (17)$$

The values of d are reported in Table 6. The excess Gibbs free energy, $\Delta G^{*\mathrm{E}}$, of activation for viscous flow has been determined

Table 6
Excess molar volume V^{E}, excess viscosity η^{E} Grunberg and Nissan interaction parameter d and excess Gibbs free energy $\Delta G^{*\mathrm{E}}$ in 40% acetone + water

c (mol dm^{-3})	V^{E} ($\times10^{-6}$ m^3 mol^{-1})		η^{E}		d ($\times10^{-6}$ m^3 mol^{-1})		$\Delta G^{*\mathrm{E}}$ (J mol^{-1})	
	303.15 K	313.15 K	303.15 K	313.15 K	303.15 K	313.15 K	303.15 K	313.15 K
N-Phenyl-2-chlorobenzohydroxamic acid								
0.010	−0.0271	−0.0234	0.0034	0.0127	12.7619	55.8896	9.8705	38.8724
0.020	−0.0712	−0.0636	0.0157	0.0196	29.2679	42.9411	38.9561	59.0692
0.030	−0.1071	−0.0987	0.0281	0.0286	34.6955	41.5370	68.7666	85.5772
0.035	−0.1235	−0.1149	0.0326	0.0348	34.4158	43.1576	79.7624	103.6329
0.040	−0.1398	−0.1311	0.0467	0.0420	42.8400	45.3801	112.7381	124.3485
0.050	−0.1750	−0.1663	0.0624	0.0537	45.4354	46.0937	149.2494	157.6590
N-*o*-Tolyl-4-chlorobenzohydroxamic acid								
0.010	−0.0226	−0.1504	0.0087	0.0088	32.8969	38.8366	23.4174	28.9320
0.020	−0.0645	−0.0610	0.0184	0.0103	34.2192	22.7559	46.6097	33.5935
0.030	−0.0993	−0.0948	0.0295	0.0168	36.3556	24.6374	73.7836	53.8232
0.035	−0.1162	−0.1120	0.0370	0.0192	38.9836	23.9735	91.9384	61.1503
0.040	−0.1328	−0.1263	0.0456	0.0289	41.7988	31.4156	112.2749	89.6691
0.050	−0.1663	−0.1572	0.0598	0.0361	43.6003	31.2630	146.1872	111.7992

by using the Eyring equation [16],

$$\Delta G^{*\mathrm{E}} = RT[\ln \eta V - (x_1 \ln \eta_1 V_1 + x_2 \ln \eta_2 V_2)] \tag{18}$$

The significance of the letters has been described earlier. The values of $\Delta G^{*\mathrm{E}}$ are reported in Table 6.

4. Discussion

It is observed from Table 1 that apparent molar volume (V_ϕ) of both the solutions decreases with increase in concentration of the solute. The magnitude of positive values of V_ϕ for both the hydroxamic acids indicates greater solute–solvent interactions. V_ϕ° is also regarded as a measure of solute–solvent interactions. The values of V_ϕ° are positive as evident from Table 2, for both the hydroxamic acids at all concentrations and at both the temperatures thereby showing the presence of strong solute–solvent interactions. Further there is an increase in the value of V_ϕ° with temperature suggesting the solvation of solute. S_V is a constant, which depends on the nature of solvent and is related to solute–solute interaction. For both the hydroxamic acids the value of S_V is large and negative indicating the presence of weak solute–solute interactions.

For the present system the values of Bc and 2.5υ were found to be of the same magnitude. The values of B-coefficients for both the hydroxamic acids in 40% (v/v) acetone + water mixed solvent are positive indicating that the solute–solvent interactions are strong. The sign of $\mathrm{d}B/\mathrm{d}T$ values give important information regarding the structure-making and structure-breaking roles of solutes in the solvent media. As evident from Table 4 a negative $\mathrm{d}B/\mathrm{d}T$ trend was observed. This is an indication of structure-making action of hydroxamic acids in mixed solvent by H-bonding.

According to Feakins model, the greater the value of $\Delta\mu_2^{0\neq}$, the greater is the structure-making ability of the solute. The values of $\Delta\mu_2^{0\neq}$ are larger than those of $\Delta\mu_1^{0\neq}$. This suggests that the formation of the transition state is accompanied by the breaking and distortion of the intermolecular bonds. For both the compounds $(\Delta\mu_2^{0\neq} - \Delta\mu_1^{0\neq}) > 0$, which suggests that the solute is a structure maker in the mixed solvent. It is interesting to note that for both the hydroxamic acids, $\Delta H_2^{0\neq}$ and $\Delta S_2^{0\neq}$ are positive indicating that the attainment of transition state for viscous flow is accompanied by bond breaking and decrease in order. These results also support the conclusions drawn from the absolute values of η and V_ϕ.

The molar volumes of mixed solvent are 25.0880×10^{-6} and $25.1329 \times 10^{-6}\,\mathrm{m^3\,mol^{-1}}$ at temperature 303.15 and 313.15 K, respectively. The molar volume of *N*-phenyl-2-chlorobenzohydroxamic acid are 263.1821×10^{-6} and $263.8425 \times 10^{-6}\,\mathrm{m^3\,mol^{-1}}$ and of *N*-*o*-tolyl-4-chlorobenzohydroxamic acid is 278.1162×10^{-6} and $278.8156 \times 10^{-6}\,\mathrm{m^3\,mol^{-1}}$ at temperatures 303.15 and 313.15 K, respectively. It is observed that the excess molar volumes of both the hydroxamic acids are negative over entire concentration range. This suggests the presence of dispersive-type intermolecular interaction. The source of negative contribution to V^E is the contraction in volume on mixing and the different sizes and shapes of component molecules of the solution. The V^E values become more negative as the temperature is increased. This indicates that the dipole–dipole interactions between the solute and solvent get increased leading to greater contraction in volume. Moreover, negative V^E also arise when a solute in aquo-organic mixture shows an amphiphilic character. As evident from Table 6, the excess viscosity values η^E are positive in the entire concentration range at both the temperatures. This indicates the presence of strong specific solute–solvent interactions such as hydrogen bond formation and charge transfer complexes. The values of Grunberg and Nissan adjustable parameter, *d*, as reported in Table 6, are positive for all the investigated systems suggesting complex formation involving mixed solvent. The values of $\Delta G^{*\mathrm{E}}$ are found to be positive for the entire range of concentration and are significantly large indicating strong interactions between the solute and the solvent.

5. Conclusions

Hydroxamic acid functional group shows a wide range of biological activity and viscosity is an important parameter that affects the permeation through biological membranes. Using viscosity data, the B-coefficient and thermodynamics of viscous flow have been computed. The behavior of these parameters suggests strong solute–solvent interactions in the system and hydroxamic acids act as structure makers in acetone–water mixed solvent.

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