

Densities and Viscosities of Binary Mixtures of Propanoic Acid with *N,N*-Dimethylaniline and *N,N*-Diethylaniline at $T = (303.15, 313.15, \text{ and } 323.15) \text{ K}$

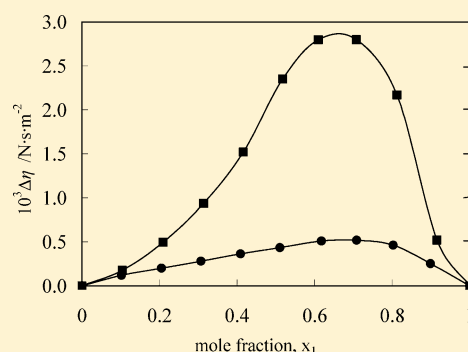
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Supporting Information

ABSTRACT: Densities and viscosities of binary mixtures of propanoic acid with *N,N*-dimethylaniline and *N,N*-diethylaniline including those of pure liquids have been measured at (303.15, 313.15, and 323.15) K over the entire composition range. From these experimental values, deviations in viscosity and excess molar volumes have been computed and fitted to Redlich–Kister type polynomial. The corresponding standard deviations have been calculated. The experimental data of viscosity have been used to test the applicability of the empirical relation of Grunberg–Nissan for all of the systems studied. Molecular interactions in the liquid mixtures have been investigated in light of the variation of deviation and excess values in the evaluated properties.



INTRODUCTION

The volumetric and viscometric study of liquid mixtures enables the determination of some useful thermodynamic and other properties that are highly sensitive to molecular interactions.^{1–6} Propanoic acid is a naturally occurring carboxylic acid, and it is used as a food preservative (calcium and sodium propionate). It is also useful as an intermediate in the production of other chemicals, especially polymers, plastics, and cosmetics. Aromatic anilines *N,N*-dimethylaniline and *N,N*-diethylaniline are used as intermediates to manufacture dyes and vanillin and as a stabilizer for calorimetric peroxidase determination.

Thermodynamic properties of liquid acid–base mixtures were studied by Rattan et al.,⁷ Solimo et al.,⁸ and Garcia et al.⁹ with propanoic acid as one component in binary mixtures. Katz et al.¹⁰ and Rama Murthy et al.^{11–13} studied the volumetric and viscometric properties in binary liquid mixtures of aniline and substituted anilines such as *N*-methylaniline, *N,N*-dimethylaniline, *N*-ethylaniline, and *N,N*-diethylaniline as one component and toluene, *n*-butanol, *m*-cresol, or *o*-cresol as the other component. Recently Ghasemi et al.¹⁴ reported the densities and viscosities for the binary and ternary mixtures of 1,4-dioxane + 1-hexanol + *N,N*-dimethylaniline. In view of the industrial importance of binary liquid acid–base mixtures are studied such as propanoic acid + *N,N*-dimethylaniline or *N,N*-diethylaniline in which propanoic acid is taken as one component and *N,N*-dimethylaniline or *N,N*-diethylaniline as the other component. In the present paper, experimentally measured densities, ρ , and viscosities, η , at different temperatures have been reported. From the experimental results various properties have been calculated and presented: deviation in viscosity, $\Delta\eta$, and excess molar volume, V_m^E .

EXPERIMENTAL SECTION

High purity and analytical reagent (AR) grade compounds of propanoic acid ($w = 0.99$), *N,N*-dimethylaniline ($w = 0.99$), and *N,N*-diethylaniline ($w = 0.99$) obtained from LOBA Chemicals, India are used in the present study. All of the chemicals are further purified by standard methods,^{15,16} like distillation and fractional distillation under reduced pressure, and only the middle fractions were collected. Finally, after the purification method the purity of the propanoic acid, *N,N*-dimethylaniline, and *N,N*-diethylaniline is found to be $w = 0.997$, $w = 0.993$, and $w = 0.993$, respectively. The purity analysis of chemicals is presented in Table 1. Before use, the chemicals were stored

Table 1. Purity Analysis

chemical name	source	initial mole fraction purity	purification method	final mole fraction purity
propanoic acid	Loba	0.99	distillation	0.997
<i>N,N</i> -dimethylaniline	Loba	0.99	fractional distillation under reduced pressure	0.993
<i>N,N</i> -diethylaniline	Loba	0.99	fractional distillation under reduced pressure	0.993

over 0.4 nm molecular sieves approximately for 72 h to remove water content and degassed. The densities and viscosities measured at (303.15 and 313.15) K for the pure liquids used in this

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Table 2. Comparison of Densities ($\rho/\text{kg}\cdot\text{m}^{-3}$) and Viscosities ($10^3\eta/\text{N}\cdot\text{s}\cdot\text{m}^{-2}$) of Pure Liquids with Literature Data at (303.15 and 313.15) K

component	T/K	$\rho/\text{kg}\cdot\text{m}^{-3}$		$10^3\eta/\text{N}\cdot\text{s}\cdot\text{m}^{-2}$	
		present work	lit.	present work	lit.
propanoic acid	303.15	982.1	982.0 ⁷	0.950	0.9498 ⁷
	313.15	973.4	971.0 ⁷	0.848	0.8453 ⁷
N,N-dimethylaniline	303.15	948.0	948.0 ¹²	1.174	1.173 ¹⁰
	313.15	938.5	939.75 ¹⁰	0.982	0.985 ¹⁰
N,N-diethylaniline	303.15	926.0	925.9 ¹²	1.703	1.709 ¹²
	313.15	917.7	918.0 ¹²	1.402	1.402 ¹²

investigation are compiled in Table 2 together with the literature data^{7,10,12} available. These results are in good agreement with the reported data.

Binary mixtures are prepared by mass in airtight bottles. The mass measurements are performed on a Mettler Toledo (Switzerland make) ABB5- S/FACT digital balance with an accuracy of ± 0.01 mg. The uncertainty in the mole fraction is 10^{-4} . Densities of pure liquids and their mixtures has been determined by using a 5 cm³ two stem double-walled Parker & Parker type pycnometer.¹⁷ This pycnometer is calibrated with triply distilled water. The pycnometer filled with air bubble free experimental liquids was kept in a transparent walled constant temperature bath for (20 to 30) min to attain thermal equilibrium. The positions of the liquid levels in the two arms are recorded with the help of traveling microscope. At least three to four measurements are performed, from which an average value of density of the experimental liquid is determined.

An Ostwald viscometer has been used to determine the viscosity of the liquid mixture, which is calibrated as described by Naidu and Prasad¹⁸ using triply distilled water. The viscometer was also calibrated using benzene, CCl₄ liquids, and so forth at working temperatures. The length of one of the capillary tubes of viscometers used in the present study is 8 cm, and its diameter is 0.56 mm. In this method water is taken into the viscometer without air bubbles and is immersed into the constant temperature bath for a period of 30 min so that the water inside the viscometer attains the temperature of the bath. The time of flow of given volume of water is measured using an electronic digital stopwatch with an accuracy of ± 0.01 s. This procedure is repeated three times, and the average flow of time (t_0) is noted. The same procedure is repeated for the experimental mixture, and the average time of flow (t) is determined.

The coefficient of viscosity of the experimental mixture has been calculated using

$$\eta/\eta_0 = (\rho t)/(\rho_0 t_0) \quad (1)$$

where ρ , ρ_0 ; t , t_0 ; and η , η_0 refer to the density, flow of time, and viscosity of the liquid mixture and water, respectively. In the present study, the constant temperature water bath (digital electronic) supplied by Concord Instruments Co. Ltd., Chennai (RAAGA type) has been used. The instrument can maintain an accuracy of temperature to ± 0.01 K as per its specifications. The reproducibility in the measured parameters of density and viscosity are 3 in 10^5 parts and ± 0.2 %, respectively. The uncertainties determined pertinent to density and viscosity measurements of the binary mixtures are given in Table 3.

RESULTS AND DISCUSSION

The experimental values of density and viscosity have been used to calculate the deviation/excess properties with the following equations.

$$V_m = \frac{x_1 M_1 + x_2 M_2}{\rho} \quad (2)$$

$$V_m^E = V_m - (x_1 V_1^* + x_2 V_2^*) \quad (3)$$

$$\Delta\eta = \eta - (x_1 \eta_1^* + x_2 \eta_2^*) \quad (4)$$

where ρ , η , and V_m are the density, viscosity, and molar volume of the mixture. M_1 , M_2 , V_1^* , V_2^* , η_1^* , and η_2^* are the molar masses, molar volumes, and viscosities of pure components 1 (propanoic acid) and 2 (N,N-dimethylaniline or N,N-diethylaniline), respectively, and x_i represents the mole fraction of the component i in the mixture. The experimental values of densities, viscosities, and calculated values of molar volumes, deviation in viscosity, and excess molar volumes are reported in Table 3. It has been observed that the density varies almost linearly with the concentration of propanoic acid as well as with temperature, but the viscosity changes nonlinearly showing maxima in all of the systems studied at all temperatures investigated. This type of behavior could be attributed to complex formation.

The values of deviation in viscosity and excess molar volume have been fitted to Redlich–Kister type polynomial¹⁹ equation,

$$Y^E = x_1 x_2 \sum_{i=0}^n A_i (x_2 - x_1)^i \quad (5)$$

where $Y^E = \Delta\eta$, V_m^E ; the subscription i in the eq 5 takes values from 0 to 4. A_i are the adjustable parameters of the function and are determined using the least-squares method. The corresponding standard deviations $\sigma(Y^E)$ have been computed using the relation.

$$\sigma(Y^E) = \left[\frac{\sum (Y_{\text{exp}}^E - Y_{\text{cal}}^E)^2}{(m - n)} \right]^{1/2} \quad (6)$$

where m is the total number of experimental points and n is the number of coefficients in eq 5. The coefficients A_i in eq 5 and the standard deviations of all the binary mixtures have been presented in Table 4.

The variation of deviation in viscosity with the mole fraction of propanoic acid is shown in Figure 1. Generally, negative values of $\Delta\eta$ indicate the presence of dispersion forces or mutual loss of specific interactions in like molecules operating in the systems arising due to weak intermolecular interactions, and positive values of deviation in viscosity indicate strong specific interactions.^{20,21} The sign and magnitude of $\Delta\eta$ depend on the combined effect of factors such as molecular size, shape, and intermolecular forces. In general ethyl groups have more +I effect than methyl groups. In DEA the ethyl groups are more electron-releasing than methyl groups of DMA on the nitrogen atom. So, the nitrogen in DEA possesses more electron density than in DMA; therefore, DEA is more favored to accept the proton of the PA. Hence it is an appropriate reason to support the observed more positive $\Delta\eta$ values in the propanoic acid + N,N-diethylaniline system.

Table 3. Experimental Values of Densities, ρ , Viscosities, η , Molar Volumes, V_m , Deviation in Viscosities, $\Delta\eta$, and Excess Molar Volumes, V_m^E , for All of the Systems at (303.15, 313.15, and 323.15) K

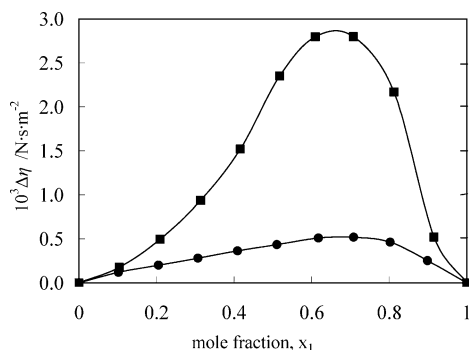
mole fraction of propanoic acid (x_1)	ρ	$10^3 \eta$	$10^6 V_m$	$10^3 \Delta\eta$	$10^6 V_m^E$	mole fraction of propanoic acid (x_1)	ρ	$10^3 \eta$	$10^6 V_m$	$10^3 \Delta\eta$	$10^6 V_m^E$
	kg·m ⁻³	N·s·m ⁻²	m ³ ·mol ⁻¹	N·s·m ⁻²	m ³ ·mol ⁻¹		kg·m ⁻³	N·s·m ⁻²	m ³ ·mol ⁻¹	N·s·m ⁻²	m ³ ·mol ⁻¹
N,N-Dimethylaniline + Propanoic Acid						N,N-Diethylaniline + Propanoic Acid					
T/K = 303.15						T/K = 303.15					
0.0000	948.0	1.174	127.84	0.000	0.00	0.0000	926.0	1.703	161.17	0.000	0.00
0.1020	954.4	1.270	121.94	0.119	-0.55	0.1047	933.4	1.801	151.46	0.177	-0.73
0.2054	959.8	1.329	116.18	0.201	-0.89	0.2096	942.1	2.040	141.68	0.495	-1.51
0.3071	964.5	1.386	110.65	0.281	-1.10	0.3142	952.1	2.404	131.94	0.938	-2.29
0.4092	969.3	1.446	105.14	0.364	-1.25	0.4164	963.8	2.910	122.37	1.521	-3.10
0.5107	974.0	1.494	99.72	0.434	-1.35	0.5177	977.9	3.663	112.82	2.350	-3.96
0.6178	978.6	1.545	94.10	0.510	-1.36	0.6097	989.1	4.040	104.55	2.796	-4.34
0.7083	982.2	1.534	89.41	0.519	-1.31	0.7077	996.2	3.968	96.42	2.798	-4.07
0.8023	985.3	1.457	84.63	0.463	-1.16	0.8123	995.0	3.260	88.63	2.169	-2.89
0.8987	986.4	1.225	79.94	0.253	-0.80	0.9156	990.9	1.532	81.16	0.519	-1.51
1.0000	982.1	0.950	75.43	0.000	0.00	1.0000	982.1	0.950	75.43	0.000	0.00
uncertainty	1.0	0.038				uncertainty	1.3	0.155			
Expanded Uncertainty (Level of Confidence = 0.95)						Expanded Uncertainty (Level of Confidence = 0.95)					
		2.0	0.076					2.6	0.310		
T/K = 313.15						T/K = 313.15					
0.0000	938.5	0.982	129.13	0.000	0.00	0.0000	917.7	1.402	162.62	0.000	0.00
0.1020	943.7	1.019	123.32	0.050	-0.40	0.1047	924.6	1.450	152.90	0.106	-0.67
0.2054	949.1	1.058	117.49	0.103	-0.75	0.2096	932.8	1.590	143.10	0.304	-1.39
0.3071	954.2	1.093	111.84	0.152	-1.00	0.3142	942.2	1.765	133.33	0.537	-2.11
0.4092	959.2	1.124	106.24	0.197	-1.19	0.4164	953.7	2.118	123.67	0.947	-2.93
0.5107	964.0	1.149	100.76	0.235	-1.29	0.5177	967.3	2.527	114.06	1.412	-3.77
0.6178	968.6	1.151	95.08	0.252	-1.30	0.6097	977.5	2.880	105.79	1.816	-4.08
0.7083	972.2	1.119	90.33	0.232	-1.24	0.7077	984.0	2.738	97.61	1.728	-3.78
0.8023	975.4	1.060	85.50	0.186	-1.09	0.8123	982.7	2.086	89.74	1.134	-2.60
0.8987	975.7	0.965	80.81	0.104	-0.66	0.9156	980.1	1.247	82.05	0.353	-1.35
1.0000	973.4	0.848	76.10	0.000	0.00	1.0000	973.4	0.848	76.10	0.000	0.00
uncertainty	1.2	0.020				uncertainty	1.5	0.098			
Expanded Uncertainty (Level of Confidence = 0.95)						Expanded Uncertainty (Level of Confidence = 0.95)					
		2.4	0.040					3.0	0.196		
T/K = 323.15						T/K = 323.15					
0.0000	930.7	0.826	130.21	0.000	0.00	0.0000	912.8	1.049	163.50	0.000	0.00
0.1020	934.8	0.843	124.50	0.029	-0.29	0.1047	919.5	1.069	153.75	0.055	-0.70
0.2054	939.7	0.869	118.67	0.068	-0.63	0.2096	926.4	1.173	144.08	0.196	-1.29
0.3071	944.5	0.895	112.99	0.106	-0.90	0.3142	934.9	1.315	134.37	0.374	-1.97
0.4092	949.1	0.920	107.38	0.143	-1.09	0.4164	944.5	1.555	124.90	0.649	-2.61
0.5107	953.5	0.940	101.87	0.175	-1.20	0.5177	955.3	1.865	115.49	0.994	-3.25
0.6178	958.0	0.944	96.13	0.192	-1.24	0.6097	965.3	2.100	107.13	1.260	-3.67
0.7083	961.1	0.912	91.37	0.171	-1.19	0.7077	971.3	1.949	98.89	1.143	-3.43
0.8023	963.5	0.864	86.55	0.134	-1.01	0.8123	974.7	1.510	90.48	0.740	-2.80
0.8987	963.5	0.778	81.84	0.060	-0.60	0.9156	974.2	0.972	82.55	0.237	-1.80
1.0000	961.4	0.706	77.05	0.000	0.00	1.0000	961.4	0.706	77.05	0.000	0.00
uncertainty	1.0	0.015				uncertainty	1.2	0.065			
Expanded Uncertainty (Level of Confidence = 0.95)						Expanded Uncertainty (Level of Confidence = 0.95)					
		2.0	0.030					2.4	0.130		

The variations of excess molar volume with the mole fraction of propanoic acid at all temperatures for the binary systems are shown in Figures 2 and 3. The deviation of a physical and chemical property of the liquid mixture from the ideal behavior is a measure of the interaction between molecules of the components of liquid mixtures, and such a type of deviation is generally attributed to dipole–dipole interactions and hydrogen bond between unlike molecules,²² respectively. The factors that are mainly responsible for the expansion of volume, that is, positive values of V_m^E are: (i) breaking one or both of the

components in a solution, that is, loss of dipolar association between the molecules (dispersion forces); (ii) the geometry of molecular structures which does not favor the fitting of molecules of one component into the voids created by the molecules of other component; (iii) steric hindrance of the molecules, where the negative values of V_m^E are due to strong specific interactions such as (iv) association of molecules through the formation of hydrogen bonds, association due to dipole–dipole interactions, or association due to induced dipole–dipole interactions; (v) accommodation of molecules due to larger differences in

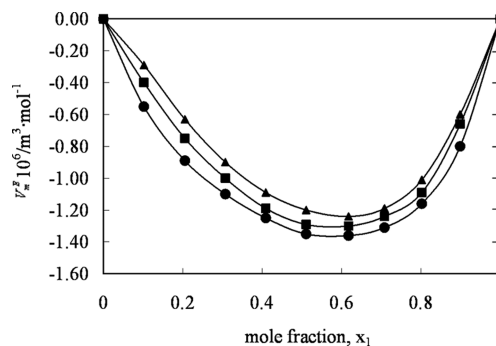
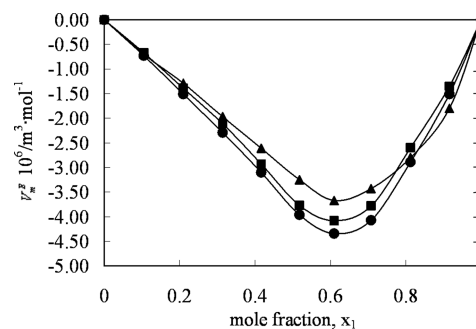
Table 4. Coefficients A_i of Redlich-Kister Type Polynomial Equation 5 and the Corresponding Standard Deviations (σ) of All of the Systems

	A_0	A_1	A_2	A_3	A_4	σ
<i>N,N</i> -Dimethylaniline + Propanoic Acid						
$T/K = 303.15$						
$10^3 \Delta\eta/N \cdot s \cdot m^{-2}$	1.715	-1.650	1.467	0.987	-1.474	0.009
$10^6 V_m^E/m^3 \cdot mol^{-1}$	0.01	-6.91	16.25	-21.59	12.24	0.02
$T/K = 313.15$						
$10^3 \Delta\eta/N \cdot s \cdot m^{-2}$	0.928	-0.584	-0.036	0.352	-0.149	0.002
$10^6 V_m^E/m^3 \cdot mol^{-1}$	0.01	-4.82	6.89	-7.52	5.45	0.02
$T/K = 323.15$						
$10^3 \Delta\eta/N \cdot s \cdot m^{-2}$	0.689	-0.524	-0.061	0.485	-0.376	0.003
$10^6 V_m^E/m^3 \cdot mol^{-1}$	0.01	-3.41	1.53	-0.21	2.08	0.02
<i>N,N</i> -Diethylaniline + Propanoic Acid						
$T/K = 303.15$						
$10^3 \Delta\eta/N \cdot s \cdot m^{-2}$	8.601	-14.681	5.040	15.866	-15.955	0.074
$10^6 V_m^E/m^3 \cdot mol^{-1}$	-0.09	-1.75	-31.27	45.85	-12.65	0.19
$T/K = 313.15$						
$10^3 \Delta\eta/N \cdot s \cdot m^{-2}$	5.460	-9.244	0.247	10.985	-6.042	0.041
$10^6 V_m^E/m^3 \cdot mol^{-1}$	-0.09	-0.056	-35.45	53.48	-17.29	0.19
$T/K = 323.15$						
$10^3 \Delta\eta/N \cdot s \cdot m^{-2}$	3.830	-6.168	-1.079	7.310	-2.734	0.037
$10^6 V_m^E/m^3 \cdot mol^{-1}$	-0.03	-4.97	-6.10	3.33	7.74	0.12

**Figure 1.** Variation of deviation in viscosity against the mole fraction of propanoic acid for ●, *N,N*-dimethylaniline + propanoic acid; ■, *N,N*-diethylaniline + propanoic acid; mixtures at $T = 303.15$ K.

molar volumes. The variation of excess molar volume in the present investigation is negative over the entire mole fraction range. Both of the components of the liquid mixtures studied are polar in nature. The observed negative V_m^E values are due to the interaction between the proton of acid and lone pair of electrons on the nitrogen atom of aniline/resonant electron density on benzene ring of substituted aniline. Due to the large difference in molar volumes/masses of propanoic acid and substituted anilines, the accommodation of smaller molecules of one component into the voids created by the other component of molecules is also responsible for observed negative V_m^E values. The above factors are responsible for the negative values of V_m^E .²³ From Figures 2 and 3, it has been observed that as the temperature of the systems increases the negative excess molar volumes decrease indicating the decrease of interaction between the unlike molecules. The negative excess molar volumes follows the order *N,N*-diethylaniline > *N,N*-dimethylaniline; hence the strength of interactions as follows, *N,N*-diethylaniline > *N,N*-dimethylaniline.

The dynamic viscosities of the binary liquid mixtures have been calculated using various empirical relations like Grunberg and Nissan,²⁴ Ubbelohde et al.,²⁵ Katti and Chaudari,²⁶ and

**Figure 2.** Variation of excess molar volumes against the mole fraction of propanoic acid for *N,N*-dimethylaniline + propanoic acid mixtures at the temperatures, ●, $T = 303.15$ K; ■, $T = 313.15$ K; ▲, $T = 323.15$ K.**Figure 3.** Variation of excess molar volumes against the mole fraction of propanoic acid for *N,N*-diethylaniline + propanoic acid mixtures at the temperatures, ●, $T = 303.15$ K; ■, $T = 313.15$ K; ▲, $T = 323.15$ K.

Heric and Brewer,²⁷ and the corresponding interaction parameters are also evaluated. The detailed description of the above empirical relations is already reported in our previous paper.³ Theoretical values of viscosity of the liquid mixtures calculated using the above equations are given in Table S1 (Supporting Information). Table S2 presents the values of interaction

parameters along with the standard deviations, σ . The variation of these interaction parameters with temperature or composition follows the order *N,N*-diethylaniline > *N,N*-dimethylaniline at constant composition and temperature, respectively. Further the interaction parameter values are found to decrease with an increase of temperature for all of the systems studied. These results are in good agreement with the results derived from the excess properties. Prolongo et al.²⁸ reported positive values of interaction parameters corresponding to systems with negative excess molar volumes. This is in good agreement with our results. The estimated values of σ are smaller indicating that experimental values of viscosities are well-correlated by all of the viscosity models.

CONCLUSIONS

Densities and viscosities of binary mixtures of propanoic acid with *N,N*-dimethylaniline and *N,N*-diethylaniline have been measured over the entire composition range at $T = (303.15, 313.15, \text{ and } 323.15) \text{ K}$. The deviations in viscosity and excess molar volumes have been computed from the experimental results. These deviation/excess properties have been fitted to a Redlich–Kister type polynomial. These results indicate the presence of strong specific interactions such as interactions between protons of propanoic acid and a lone pair of electrons on the nitrogen atom of aniline which due to the observed difference in molar volumes of propanoic acid and substituted aniline are responsible for the observed negative V_m^E and positive $\Delta\eta$ values in the present investigated systems. The strength of interactions follows the order *N,N*-diethylaniline + propanoic acid > *N,N*-dimethylaniline + propanoic acid. The strength of interactions is also studied with the variation of temperature. The experimental viscosity values are compared with the viscosity values obtained from different empirical relations, and these are in good agreement with the experimental values.

ASSOCIATED CONTENT

Supporting Information

Theoretical values of viscosity of the liquid mixtures (Table S1) and values of interaction parameters along with the standard deviations, σ (Table S2). This material is available free of charge via the Internet at <http://pubs.acs.org>.

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