

Experimental Study of Ultrasonic and Volumetric Properties

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Abstract – The volume and energy calculated for liquids in this theory were found to be close with the experimental values. But this theory fails to predict the entropy values. Prigogine and Garikian (Ali and Tariq, 2008) used this approach and obtained a complicated expression for the potential function. Prigogine et al. (Bhatia et. al., 2009, Roy et. al., 2007) used smoothed potential model by substituting square well potential for Lennard-Jones and Devonshire potential. When the difference in size of component molecules is small, this theory predicts positive excess free energy and large excess volume for mixtures. Molecular models are used to specify both the nature of the process and to derive equations for physicochemical properties.

INTRODUCTION

Study of propagation of ultrasonic waves in liquid systems and solids is now well understood by means of examining certain physical properties of the materials. The data obtained from ultrasonic propagation parameters in liquid mixtures and solutions viz., ultrasonic velocity and their variation with concentration of one of the components, helps to understand the nature of molecular interactions in the mixtures further, it provides a means to verify theories that deal with the structure of liquids. The fact that low amplitude signals have the added advantage of low distortion of waveform studies in solutions leads to studies in pure liquids. The advantage is the desired property of the solution can be obtained by simply varying its concentration (Leyendekkers, 1986). Thus studies in solutions offer wide applications in the processes of industries and technology (Aminabhavi et. al., 1999) (Garcia et. al., 1997) (Jaykumar et. al., 1996). Several empirical and semi-empirical formulae have been developed correlating ultrasonic velocity with other parameters. Several researchers so far studied several acoustic and thermodynamic properties of non aqueous – non electrolyte, non aqueous – electrolyte, aqueous – electrolyte and aqueous – non electrolyte liquid mixtures/solutions with the following aims:

1. To study the molecular interactions in liquid mixtures/solutions.
2. To explain the propagation of ultra sound in liquids different theories were suggested and various expressions were derived and

experimental work is carried out to verify the theories suggested.

3. To study and determine the important characteristic parameters of liquids calculated from experimental results such as isentropic compressibility, intermolecular free length, specific acoustic impedance, free volume, internal pressure, molar radius and molar volume etc.
4. To study the change of phase occurring in liquids, to study the rates of chemical reactions and biochemical reactions such as fermentation etc.
5. To determine the important thermodynamic properties for characterizing the thermodynamic state of liquid systems.

In the present study, for the liquid mixtures/solutions, the following are discussed:

1. Study of molecular interactions in the light of variation of various acoustic and thermodynamic parameters.
2. Comparing the ultrasonic velocities computed using various theories with experimental values and to finding out the suitability of these theories to the systems investigated.
3. Comparing the viscosities calculated using various empirical relations with experimental data.

4. Study of molecular interactions in the light of deviation/excess properties of calculated parameters.
5. Fitting of various deviation/excess properties to Redlich-Kister type polynomial and calculate the standard deviation.

ULTRASONIC VELOCITY MEASUREMENT

Measurement of ultrasonic velocity is generally made either by continuous wave methods, or by pulse methods (Leyendekkers, 1986, Aminabhavi et. al., 1999, Garcia et. al., 1997) Continuous waves are used in optical diffraction, reverberation and interferometry methods. In pulse echo or pulse superposition methods, RF pulses of short duration are used.

CONTINUOUS WAVE METHODS

Striation method, interferometer method, optical diffraction method and Brillouin scattering are some of the important continuous wave methods involve the measurement of frequency and wavelength of sound wave in a given medium. The product of frequency and wavelength of the wave gives the ultrasonic velocity. In optical methods (Jaykumar et. al., 1996), (Radhamma et. al., 2004), (Ciocirlan and Iulian, 2009), (Kumar and Deepika, 2012). when a sound beam passes through a medium it alters the local density and hence the refractive index of the medium. In 1866, Striation or Schlieren method was developed by Toepler (Naidu et. al., 2002). for measuring wavelength of sound waves. In this method sharp image of a vertical slit is focused on a vertical wire through a cell containing the liquid. Stationary sound waves are setup in the cell by a crystal and plane parallel reflector arrangement. A series of bright and dark fringes are formed alternatively and can be seen by focusing a telescope on the wire. The distance between these fringes is proportional to the wavelength of sound. The acoustic wave remains virtually stationary because of slowness of speed of sound compared with speed of light

REVIEW OF LITERATURE

Solids possess long range order in their structure, while gases lack the order in their structure. Liquids do neither have the rigidity of solids nor the fluidity of gases. Hence, liquids exhibit properties intermediate to solids and gases. X-ray studies revealed that liquids possess short range order. The development of the theory of liquids is based upon their structure, molecular interactions in the liquid mixtures, and the dynamic processes of the molecular thermal motion. Based on the above, various theories of liquids are proposed to understand and predict the ultrasonic, thermodynamic and transport properties of liquids.

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In the cell model, each molecule spends much of its time confined by its neighbours in a comparatively restricted region. According to this model any one molecule moves in a cell formed by its neighbours and these molecules have significantly more freedom than those of solids. This model fails to account for features that distinguish a liquid from a solid. Hildebrand and Scott (Kumar and Deepika, 2012), (Naidu et. al., 2002) discussed shortcomings of this model. Further developments of this theory are due to Lennard-Jones and Devonshire (Comelli et. al., 2006), to express thermodynamic functions of pure liquids in terms of intermolecular forces) Kihara (Sah and Roy, 2011) and others (Sathyanarayana et. al., 2007).

32 Barker's Tunnel theory (Ali and Tariq, 2008) is essentially one dimensional disordered cell theory. According to this model liquid consists of subsystems of lines of molecules moving almost one dimensionally in a tunnel whose walls are formed by neighbouring lines. The volume and energy calculated for liquids in this theory were found to be close with the experimental values. But this theory fails to predict the entropy values. Prigogine and Garikian (Bhatia et. al., 2009) used this approach and obtained a complicated expression for the potential function. Prigogine et al. (Roy et. al., 2007), (Syal et. al., 2005) used smoothed potential model by substituting square well potential for Lennard-Jones and Devonshire potential. When the difference in size of component molecules is small, this theory predicts positive excess free energy and large excess volume for mixtures. Molecular models are used to specify both the nature of the process and to derive equations for physicochemical properties. Quasichemical models provide the language for studying both equilibrium and kinetic properties of liquids (Reichardt, 2003, Riddick et. al., 1986, Parker and Parker, 1925, Aminabhavi and Banerjee, 1998, Baluja et. al., 2008). Quasichemical models of association processes have been applied in several instances to thermodynamic and 34 spectroscopic properties of liquid systems (Reichardt, 2003, Riddick et. al., 1986, Parker and Parker, 1925, Aminabhavi and Banerjee, 1998, Baluja et. al., 2008, Marcheselli et. al., 1992, (Nikam and Kharat, 2005, Chauhan et.

al., 1995) Recently, along with the elaboration of association models and the thermodynamics of associated solutions, this approach has been also extended to dielectric, optic, and kinetic properties of mixtures. In this model thermodynamic property, Gibbs free energy G , enthalpy H , entropy S , and the corresponding excess properties are considered for liquid mixtures/solutions.

SCOPE OF STUDY

Acrylic esters are important industrial chemicals and are widely used as precursors in the production of technically important special type polymers. Amides, relevant liquid systems for the study of molecular interactions, are among the most common solvents used in chemical reactions and in many industrial processes and important model systems for the investigation of peptide and protein interactions in biological systems. Binary liquid mixtures containing glycols are used in the pharmaceutical, cosmetic and food industries. Propionic acid is a naturally occurring carboxylic acid and it is used as food preservative (calcium and sodium propionate). It is also useful as an intermediate in the production of other chemicals, especially polymers, plastics, cosmetics. Aromatic anilines N , N -dimethyl aniline, N,N -diethyl aniline are used as intermediates to manufacture dyes, vanillin, and as a stabilizer for calorimetric peroxidase determination. Alkanols are of interesting simple examples of biological and industrial important amphiphilic materials. Dimethyl sulfoxide is a highly polar ($\mu = 4.06$ D) self-associated solvent, is an important solvent in chemistry, biotechnology, and medicine and it is able to participate in hydrogen bonding. Dichloromethane used as a cleaning agent, paint remover and in extraction technology; paraffin extraction, recovery of specialty pharmaceuticals. 1,2-Dichloroethane mostly used in the production of vinyl chloride which is used to make a variety of plastic and vinyl products including polyvinyl chloride. Tri potassium citrate is used to control uric acid kidney stones. When orally administered it is rapidly absorbed and excreted in urine as carbonate. Tri-sodium citrate is used as food additive and as a preservative. In blood transfusions it is used as anticoagulant. In view of growing interest and importance of the chemicals in the industrial purpose the result of an ultrasonic velocity, density and viscosity to study the related acoustical parameters of above chemicals are presented in this work. However, no effort has been made to collect the ultrasonic velocity, density and viscosity data on prepared binary and ternary (equimolar) mixtures in the previous literature. Therefore studies of acoustic, volumetric and transport properties of following systems are made in the present study. Moreover, the study intends to provide the information on the molecular interactions between the constituent liquid molecules for the

prepared binary and ternary (equimolar) liquid systems.

EXPERIMENTAL

N,N -dimethyl formamide (DMF) (mass fraction purity 0.99) and butyl acrylate (BA) (mass fraction purity 0.99) are the products of LOBA Chemicals, and methyl acrylate (MA) (mass fraction purity 0.99), ethyl acrylate (EA) (mass fraction purity 0.99) and 2-ethyl hexyl acrylate (EHA) (mass fraction purity 0.99), obtained from KEMPHASOL Company, Bombay. All are Analytical Reagent grade (AR grade) used in the present investigation and are further purified by standard methods (Gheorghe et. al., 2013). The solutions of binary mixtures of DMF with MA, EA, BA and EHA are prepared in the specially designed glass bottles with air tight stoppers and adequate precautions are taken to minimize evaporation losses. The weighing of solutions is taken using METTLER TOLEDO (Switzerland make) ABB5-S/FACT digital balance with an accuracy of ± 0.01 mg. The ultrasonic velocity of pure liquids and their binary mixtures are measured by using a multi-frequency ultrasonic interferometer (M-82 Model) at a fixed frequency of 2MHz. The density measurements are carried out using a two stem pycnometer of Parker & Parker type [37] of capacity of bulb volume of 5 cm.

CALCULATED PROPERTIES

1. Molar volume (V_m)

The Molar volume is the volume occupied by one mole of a substance at a given temperature and pressure. Mathematically, it can be written as:

$$V_m = \frac{M}{\rho}$$

Where M = Molar mass of the compound

ρ = Density of the compound

If the sample is a mixture containing N components, the molar volume is defined as:

$$V_m = \frac{\sum_i x_i M_i}{\rho_{\text{mixture}}}$$

ρ_{mixture} = Density of the mixture

2. Compressibility (β)

It is a measure of the relative volume change of a fluid and can be defined as $\beta = -\frac{1}{V} \left(\frac{\partial V}{\partial p} \right)_T$ Where ' V ' is volume and ' p ' is pressure. Isentropic compressibility is a bulk parameter and is the ease with which the medium can be compressed (Leyendekkers, 1986). Non linear variation of isentropic compressibility as a function of composition of liquid mixture is sufficient evidence for

existence of molecular interactions in solution (Aminabhavi et. al., 1999), (Garcia et. al., 1997), (Jaykumar et. al., 1996). When an electrolyte is dissolved in water, generally, isentropic compressibility decreases. This lowering of isentropic compressibility is attributed to the influence of electrostatic field of ions on the surrounding solvent molecules. Depending on the nature of electrolyte, sometimes isentropic compressibility increases even non-linearly.

RESULTS AND DISCUSSION

The experimental values of u and ρ of pure liquids at 308.15 K along with their literature (Ali et. al., 2002), (Rai et. al., 1989), (Thirumaran and Sudha, 2010) (Oswal et. al., 2005) (Palani and Meenakshi, 2007) values are presented in Table 4.A.1. The data of ultrasonic velocity (u), and density (ρ) at 308.15 K in the mixtures of DMF with MA/EA/BA/EHA are presented in Table 4.A.2 and shown in figures 4.A.1, 4.A.2, 4.A.3 and 4.A.4 respectively. From the table and figures it is observed that, the ultrasonic velocity increased non-linearly and density first decreases up to 0.3 mole fraction of DMF and then increases with increase of mole fraction of DMF in all binary mixtures except in EHA+DMF binary system. In this binary system density increases non-linearly over the entire composition range. This non-linear variation suggests molecular interactions exist in the components of liquid mixtures. The experimental values of ρ and η are reported in Tables 5. B. 2, 5.B.3 and 5. B. 4 respectively for (DMF+MOH)+PA, (DMF+EOH)+PA and (DMF+POH)+PA mixtures at different temperatures over the entire mole fraction, x of PA. It has been observed that the density varies monotonically with the concentration of propanoic acid at all temperatures but the viscosity changes non-linearly showing maxima at about $x \approx 0.6$ in the PA rich region as all investigated temperatures in all the systems. The observed maxima (peak) have found to increase with increasing chain length of alcohols. Such deviations may be attributed to specific interactions arising from the formation of complexes among the mixing molecules. This type of behavior could be attributed to complex formation. The variation of $E_m V$ for all the systems at different temperatures is shown in Figures 5.B.1, 5.B.2 and 5.B.3. The variation of excess molar volume in the present investigation is density increases non-linearly over the entire composition range. This non-linear variation suggests molecular interactions exist in the components of liquid mixtures. The variation of deviation in ultrasonic velocity (Δu) with mole fraction of DMF over the entire composition range is shown in figure 4.A.5. The sign and magnitude of Δu play an important role in describing molecular rearrangement as a result molecular interactions exist between the component molecules in the mixtures. Generally, positive values of Δu indicating strong interactions where as negative values of Δu indicate dispersion forces due to weak interactions (Palani and

Meenakshi, 2007) (Mehra and Pancholi, 2005) From the figure 4.A.5 it is observed that, Δu values are negative for all the systems studied, such negative values suggest that dispersion forces are operating in the systems.

CONCLUSION

- (i) The ultrasonic velocities and densities for (MA or EA or BA or EHA + DMF) binary mixtures are measured and the values of Δu , $E_m V$, Δk_s , $E_f L$ and $Z E$ are calculated.
 - (ii) The $E_m V$, Δk_s and $E_f L$ values are positive where as Δu and $Z E$ values are negative in all the binary mixtures, indicating dispersion forces between the acrylate and DMF molecules and it follows the order MA+DMF>EA+DMF>BA+DMF>EHA>DMF.
 - (iii) The partial molar volumes $V_{m,1}$, $V_{m,2}$, and excess partial molar volumes ${}^1 E V_m$, ${}^2 E V_m$ are also calculated. The observed higher partial molar volumes in the liquid mixture when compared to the respective molar volumes of pure components indicating weak interactions present in the systems.
 - (iv) The ultrasonic velocities computed from different velocity theories are correlated with the experimentally measured ultrasonic velocities. Jacobson's equation gives the good agreement between the theoretical and experimental ultrasonic velocity values.
 - (v) Besides, the ultrasonic velocity is fitted to two types of polynomials which describe the ultrasonic velocity data quantitatively as well as qualitatively even in the specific interaction predominant region where non-ideal behaviour of the system is noticed.
- (i) The densities and viscosities of liquid mixtures of propanoic acid with equimolar mixture of N,N-dimethyl formamide and methanol/ethanol/1-propanol have been measured over the entire composition range at $T = 303.15, 313.15$ and 323.15 K and the properties like, $E_m V$, $G E$, $V_{m,1}$, $V_{m,2}$, ${}^1 E V_m$, and ${}^2 E V_m$ have been computed from the experimental results.
 - (ii) The values of $V_m E$ are negative and $\Delta \eta$, $\Delta G^* E$ are positive at all temperatures studied, indicating the presence of strong interactions (hydrogen bonding O.....H-O-) such as interactions between carbonyl group (-C=O) of amide molecules and hydroxyl group(-OH) of alcohol groups and also the intermolecular interactions between carbonyl group(-C=O) of acid molecules and hydroxyl group(-OH) of

alcohols, dipole-dipole interactions and geometrical fitting of smaller molecules into the voids created by the larger molecules in the investigated systems.

- (iii) The strength of interactions follows the order (DMF+MOH) +PA < (DMF+EOH) +PA < (DMF+POH) +PA.
- (iv) The strength of interactions is also studied with the variation of temperature.
- (v) 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. The dynamic viscosities of the investigated liquid mixtures have been calculated using the Grunberg and Nissan [30], Hind and Ubbelohde (Parker and Parker, 1925), Katti and Chaudari (Aminabhavi and Banerjee, 1998), Heric and Brewer (Baluja et. al., 2008) relations. The theoretical values of viscosity of the (DMF+MOH)+PA, (DMF+EOH)+PA and (DMF+POH)+PA liquid mixtures calculated using the above theories are compiled in Tables 5.B.8, 5.B.9 and 5.B.10 respectively. Table 5.B.11 presents the values of the interaction parameters along with the standard deviations, σ . The terms G12, H12, Wvis and Δ_{12} are adjustable parameters representing the binary interactions. The variation of these parameters with composition follows the order (DMF+POH)+PA > (DMF+EOH)+PA > (DMF+MOH)+PA at constant temperature. Further the interaction parameter values are found to decrease with an increase in temperature of all the systems studied.

These results are in good agreement with the results derived from the excess properties. Prolongo et al (Marcheselli et. al., 1992) reported positive values of interaction parameter 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 the four viscosity models.

- (i) Ultrasonic velocities, u and densities, ρ of mixtures of 1-butanol or iso-butanol or tbutanol with equimolar mixture of (N,N-dimethyl acetamide + ethyl acrylate) over the entire composition range have been measured at $T = 308.15$ K.

- (ii) Using the experimental results, Δu , Δk_s , E_{Vm} , LE_f and ZE have been calculated and these properties with composition of the mixtures has been discussed in terms of molecular interactions in these mixtures.
- (iii) The deviation/excess properties are fitted to Redlich-Kister type polynomial and the corresponding standard deviations are calculated.
- (iv) Negative values of E_{mV} , Δk_s , LE_f and positive values of Δu , ZE are observed over the entire composition range. The observed negative and positive values of deviation/excess properties are attributed to the strong interactions between the unlike molecules of the mixtures.
- (v) The strength of interaction in the mixtures follow the order (DMA+EA) +, t-butanol > iso-butanol > 1-butanol.
- (vi) Further theoretical values of sound velocity in the mixtures are evaluated using various theories and are compared with experimental sound velocities to verify the applicability of such theories to the systems studied. Theoretical ultrasonic velocity data has been used to study molecular interactions in the systems investigated.

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