Journal of Applied Science & Process Engineering
Vol. 13, No. 1, 2026

Thermophysical Characterisation of the Diacetone Alcohol-Water Binary System

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Abstract

Understanding of thermodynamic variables in any binary liquid system is crucial for the development of industrial products. Among the various thermodynamic parameters, density (ρ), viscosity (η), surface tension (γ), apparent molar volume (ϕ_v) and partial molar volume ($\bar{V}_m$) have been extensively used to characterize the binary liquid systems. In this article, we have investigated the density (ρ), viscosity (η), surface tension (γ), apparent molar volume (ϕ_v) and partial molar volume ($\bar{V}_m$) of binary mixtures of diacetone alcohol (DAA)-water system across all mole fractions at 301 K. Viscosity increases as the mole fraction of diacetone alcohol increases up to 0.6 and then decreases drastically. Initially, positive deviations have been observed for the viscosity of the liquid binary mixtures, while it changes to negative at higher DAA concentrations. The increase in surface tension at higher DAA concentrations in the binary liquid mixture indicates stronger intermolecular forces, leading to an increase in the resistance to surface area expansion at the interface. Volume excess properties were analyzed to explore molecular interactions between the components. The excess volume (V^E) and volume changes (V_m) indicate the volume contraction at higher mole fractions of DAA due to stronger intermolecular interactions between diacetone alcohol and water. The structural and thermodynamic data of the DAA-water system can contribute to the advancement of process and industrial product development techniques.

Keywords: *Binary mixture, Diacetone alcohol, Water, Apparent molar volume, Partial molar volume*

1. Introduction

State variables such as composition, volume, pressure and temperature are important parameters used to understand the thermodynamics of the system. Among the factors mentioned above, composition is considered to be an important parameter which is not affected by temperature or pressure [1]. The transport properties such as surface tension and viscosity play an important the behavior of liquids, especially the mixture of two different substances. Viscosity measures the fluid resistance to flow arising from the internal friction and molecular interactions. In contrast, surface tension arises due to the energy associated with the liquid interface from cohesive interactions between molecules at the surface [2]. In addition, composition, temperature and the type of intermolecular forces also affect the viscosity of a solution made up of two liquids/components.

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Manuscript History:
Received 4 September, 2025, Revised 25 November, 2025, Accepted 28 November, 2025, Published 30 April, 2026

https://doi.org/10.33736/jaspe.10743.2026

Pure substances exhibit constant viscosity under specified conditions, whereas the liquid mixtures containing two different molecules, i.e. A and B, could exhibit non-linear characteristics. Viscosity is determined by the energy needed to break cohesive bonds between similar molecules (A-A; B-B) and dissimilar molecules (A- B), leading to deviation from ideal behavior [3]. Two types of deviations from ideal behavior have been observed: i) positive deviation in the liquid mixture arises due to the hydrogen bonds linking the molecules thereby increasing the viscosity and ii) the cohesive forces weakened by disruptive interactions, reducing the internal friction leading to negative deviation [4,5]. Real systems usually show volume deviations due to intermolecular interactions thus affecting the packing density.

Studies often employ macroscopic intensive properties to characterize the internal structures and molecular interactions within these binary liquid mixtures at different temperatures and pressures. The anomalous properties in the density, viscosity and volume excess molar volume, apparent molar volume and partial molar volume are used to determine the interactions of different molecules in the binary liquid mixtures [6]. The partial molar volume ($\bar{V}_m$) indicates the change in the volume of one component when a small amount of the other component is added at constant temperature and pressure [7]. With the assumption that the volume of the other component remains constant, the apparent molar volume has also been used to understand the volumetric behaviour in a binary liquid mixture [8].

Apparent molar volume, viscosity measurements and thermodynamic properties for different types of alcohols (methanol, ethanol and butanol) in aqueous solution at different temperatures have been studied [9-12]. Volumetric, viscometric and ultrasonic studies of dimethyl sulfoxide with normal alcohols/binary mixtures of diethyl oxalate, 1,4-dioxane, carbon tetrachloride and butanol/chloroform-2-butanone system, correlating the properties have been reported [13-18]. Electrical conductivity and viscosity measurements reveal insights into molecular interaction and the presence of nano domains in binary organic liquid mixtures [19].

The surface tension of a liquid mixture is determined by its composition and the type of intermolecular forces operating at the interface [20]. Surface changes occur when the molecules with lower surface tensions or weaker cohesive forces migrate nearer to the surface [21]. Thus, the total surface tension of the liquid mixture differs from the average values of the pure components [22-24]. Related investigations encompass the dynamic surface tension of water, physicochemical analysis of electrolytes in mixed solvents and the relationship between solute molecular volumes and solvation phenomena [25]. Further, molecular interactions and the formation of nano domains in binary organic liquid mixtures, including studies on systems like triethyl phosphate with various formamides and ionic liquid mixtures using the pendant drop method and surface light scattering, are employed to measure interfacial tension and viscosity [26].

Corresponding states method/Jouyban–Acree model/UNIFIC VISCO group contribution methods are used to predict and correlate the binary liquid mixture behavior [27]. Reported computational and experimental studies have covered the properties of diethyl carbonate with alcohols [28]. Computational studies on ethanol–water mixtures based on density and temperature on excess molar volumes and viscosities of ethylenediamine and water have been reported [29].

Diacetone alcohol (DAA) is a colourless and slightly viscous liquid. Due to its exceptional water and oil solubility, non-corrosiveness, and low volatility, it is used in a wide range of industrial products such as a solvent in coatings, paints, linkers, insecticides, and textile processing [30-38]. Also, it is used in the production of synthetic leather and silk [30]. Tetramethyl oxo piperidine and pigments are synthesized using a mixture of diacetone alcohol and benzene, whereas polymeric photonic crystals for optical switches are made using diacetone alcohol with chlorobenzene [39,40]. The surface-active interactions and surface composition play a crucial role during the preparation of stable emulsions, foams and surfactant systems [41].

Thus, physical parameters such as viscosity and surface tension can influence its wetting, flow, levelling, overall film formation, and appearance [42,43]. The properties such as (i) density(ρ) evaluates molecular packing and arrangement, ii) viscosity (η) to assess flow resistance, iii) surface tension (γ) to explore interfacial phenomena, (iv) apparent molar volume (ϕ_v) to understand solute-solvent interactions, and (v) partial molar volume ($\bar{V}_m$) investigates the compositional effects. The combination of volumetric, viscometric and surface tension measurements is a very good methodology to study the intermolecular interactions and structural arrangements within pure liquids and more commonly, liquid mixtures (e.g., binary or ternary systems). In this article, we have studied the density (ρ), viscosity (η), surface tension (γ), apparent molar volume (ϕ_m) and partial molar volume ($\bar{V}_m$) of the binary mixtures of the DAA-water system at 301 K.

2. Materials and Methods

All chemicals were obtained from SD Fine Chemicals and Loba Chemie, India, and used as received without further purification. Experimental studies were carried out on the binary liquid mixtures of diacetone alcohol (DAA) and water at a temperature of 301 K (28°C) and their pure components.

2.1. Density Measurement of the pure liquids/binary liquid mixtures

The specific gravity bottle was cleaned with water and then dried with acetone prior to the measurements. Binary mixtures of DAA and water were prepared in varying compositions as outlined in Table 1 [7,8]. The density of the binary liquid mixture was determined using a specific gravity bottle with an approximate volume of 10 mL. Initially, the empty specific gravity bottle and an empty beaker were weighed separately on an electronic balance. Pure liquids and their binary liquid mixtures containing 25 mL of diacetone alcohol and water at various concentrations were prepared (see Table 1). The mass of pure liquids/each prepared liquid binary mixture, along with the beaker, was recorded using an electronic balance PGB 200 (Wensar, India) (±0.001 g accuracy). The prepared liquid binary mixture was transferred into the density bottle, and the combined mass of the specific gravity bottle and the binary liquid mixture/s was measured. The binary solution mixture mass was obtained by subtracting the weight of the empty specific gravity bottle. Density of distilled water is 0.9962×10^3 kg m^{-3} at 301 K.

2.2. Viscosity Measurement of the pure liquids/binary liquid mixtures

The viscosity of the pure liquids/binary liquid mixtures was measured at 301 K. A clean specific gravity bottle was rinsed with distilled water and dried thoroughly. Both the empty bottle and the same bottle filled with distilled water were weighed on an electronic balance. The bottle was then filled with pure liquid/each liquid binary mixture/s at different concentrations, and their total mass was recorded to determine the density [7].

For viscosity measurement, a capillary U-shaped Ostwald viscometer (borosilicate glassware, A grade, Borosil, India) was cleaned and rinsed with distilled water before use. It was positioned vertically on a stand, and approximately 20 mL of each mixture was introduced into the viscometer. The viscometer was calibrated with distilled water, and flow time was recorded with an electronic stopwatch [22,23]. The flow time of each binary liquid mixture through the capillary was measured (two trials for each pure liquid/binary liquid mixture to ensure accuracy and reproducibility) and to calculate the viscosity under the above-mentioned conditions [44].

2.3. Surface Tension Measurement of the pure liquids/binary liquid mixtures

A stalagmometer (A grade 3758 Borosilicate glassware, JSGW, India) calibrated with pure water was used for surface tension measurements at 301 K [7]. To get a consistent drop rate of 10 to 15 drops per minute, the flow rate was adjusted by turning the knob. The stalagmometer was then clamped firmly, and ten to fifteen droplets of water were allowed to fall into a beaker. The procedure was carried out twice for consistency, and the number of drops was counted. Later, the stalagmometer was filled with pure liquids/each binary liquid mixture and the above process was repeated [7,45]. The surface tension for each binary liquid mixture was computed [7,46]. The apparent molar volume (ϕ_v) of the binary liquid mixture was calculated using equation (1).

$$\phi_v = \frac{M}{d_o} - \frac{1000(d - d_o)}{d_o c} \tag{1}$$

where d0 and d are the densities of the solvent and binary liquid mixture, respectively, c: concentration in mol L^{-1} and M: molecular weight of the solute. The partial molar volume ($\bar{V}m$) was determined using equation (2) [7,8]:

$$V_m = \frac{W}{d(x_1 + x_2)} \tag{2}$$

where W is the weight of the diacetone alcohol/water/diacetone alcohol-water liquid mixture taken in a beaker.

The excess volume (V^E) was calculated from the experimental density data using equation (3) [47-52]:

$$V_E = \frac{x_1M_1 + x_2M_2}{\rho_m} - \left(\frac{x_1M_1}{\rho_1} + \frac{x_2M_2}{\rho_2} \right) \tag{3}$$

where x_1 and x_2 are the mole fractions of components 1 and 2; M_1 and M_2 are the molar masses of pure components 1 and 2; ρ_1 and ρ_2 denote the densities of the pure components, and ρ_m: density of the binary mixture.

3. Results and Discussion

The density (ρ), viscosity (η), surface tension (γ), apparent molar volume (ϕ_v), partial molar volume ($\bar{V}_m$) and excess volume (V^E)for binary liquid mixtures of diacetone alcohol (DAA) and water (H_2O) at 301 K are given in Table 1.

Dissociation of an organic acid generates ono-oxalate anion, which participates in various ionic equilibria, leading to the formation of ion-pairs or ionic doublets. Viscosity data confirm that these carboxylic acids are structure-promoting solutes, and the capacity of these acids to promote solvent structure increases in the order: methanol < ethanol < butanol [9]. A significant difference exists between the molar volumes of the alkanol (1) + propan-2-amine (2) systems at 298.15 K and 308.15 K and suggests that the non-associated propan-2-amine molecules fit into the interstitial spaces of the alkanol clusters, resulting in the observed negative V^E values across all temperatures. This effect is further enhanced by a reduction in the self-association of the alkanol; the breaking of internal hydrogen bonds in alkanol is the dominant factor, overriding the weaker dispersive forces between the two components and leading to an increasingly negative V^E. Overall, these results indicate strong interactions between the -OH and $-NH_2$ functional groups [11]. Analysis of various water-methanol mixture models shows their ability to mix in the water-rich region. This structural evidence contrasts with other models by indicating no component segregation [10].

Table 1. Apparent molar volume, partial molar volume, viscosity and surface tension values for the different binary liquid mixtures (diacetone alcohol-water)

Volume (mL)		**Viscosity (η) (N sm^{-2})**	**Surface tension (γ) N m^{-1})**	**Partial molar volume ($\bar{V}_m$) (mL mol^{-1})**	**Apparent molar volume (ϕ_v) (mL mol^{-1})**
Diacetone alcohol	**Water**				
20	0	0.00207 (±0.00015)	0.02473 ($\pm 4.511 \times 10^{-4}$)	125.596 (±2.4988)	-
18	2	0.00468 (±0.00014)	0.02658 ($\pm 1.044 \times 10^{-3}$)	76.2044 (±1.2334)	113.75 (±3.3234)
16	4	0.00514 ($\pm 4.0639 \times 10^{-5}$)	0.02908 ($\pm 8.949 \times 10^{-4}$)	54.6301 (±0.2263)	119.247 (±8.277)
14	6	0.00484 ($\pm 5.159 \times 10^{-6}$)	0.03093 ($\pm 1.0125 \times 10^{-3}$)	43.3261 (±0.5417)	119.33 (±8.1600)
12	8	0.0046 ($\pm 4.7289 \times 10^{-6}$)	0.0318 ($\pm 6.884 \times 10^{-4}$)	35.5882 (±0.09065)	119.935 (±7.1629)
10	10	0.0036 ($\pm 9.0937 \times 10^{-5}$)	0.03316 ($\pm 1.115 \times 10^{-3}$)	30.224 (±0.2658)	120.725 (±5.9043)
8	12	0.00287 ($\pm 3.1672 \times 10^{-5}$)	0.03375 ($\pm 1.119 \times 10^{-3}$)	26.4578 (±0.0550)	120.78 (±5.9679)
6	14	0.00202 ($\pm 1.9600 \times 10^{-5}$)	0.03366 ($\pm 7.820 \times 10^{-3}$)	23.617 (±0.125)	120.95 (±5.798)
2	18	0.00104 ($\pm 1.9151 \times 10^{-5}$)	0.05059 ($\pm 5.32 \times 10^{-4}$)	19.3774 (±0.0971)	121 (±5.624)
0	20	8.38408×10^{-3} ($\pm 7.2644 \times 10^{-5}$)	0.0717 (1.39×10^{-5})	17.6825 (±4.61)	121.325 (±4.9143)

*Values in the brackets indicate standard deviation values.

The densities and viscosities of four binary liquid mixtures containing diethyl oxalate (DEO) with the organic solvents ethanol, ethyl acetate (EA), tetrahydrofuran (THF) and toluene at temperatures ranging from 303.15 to 323.15 K and at atmospheric pressure have been examined. Ethanol or THF addition to DEO led to a positive deviation of excess molar volume (V^E), whereas EA or toluene addition to DEO led to a negative deviation. These results indicated that the molecular packing of DEO with EA and toluene was more efficient than that with ethanol and THF. By contrast, the $\Delta\eta$ values of the four binary mixtures were negative, indicating that the viscosity was markedly lower than that of an ideal mixture [13]. Viscosities and densities for 1,4-dioxane + carbon tetrachloride, carbon tetrachloride + butanol, and 1,4-dioxane + butanol have been measured as a function of mole fraction at 303.15 K, 308.15 K, and 313.15 K, respectively [15].

The viscosities of the binary liquid mixtures measured at 301 K are summarized in Table 1. Viscosity of (DAA-H_2O) the binary liquid mixtures is influenced by intermolecular forces such as electrostatic interactions, dipole-dipole interactions, induction forces and dispersion forces [53,54]. The viscosity reaches a maximum at the mole fraction of 0.6 for the DAA-H_2O liquid mixture and then gradually decreases [see Figure 1(a)]. Excess viscosity (η^E) or viscosity deviation ($\Delta\eta^E$) reflects the ease of flow compared to an ideal mixture. Positive viscosity deviation suggests the formation of strong intermolecular interactions (new hydrogen bonds), which restrict the molecular movement. Negative viscosity deviation indicates weaker interactions or the breaking up of the structure of the pure components. The viscosity deviation for the binary liquid mixtures is shown in Figure 1(b). Positive viscosity deviations are observed in the systems with strong specific interactions between DAA-H_2O molecules in the mole fraction range of 0.0 to 0.9 and negative viscosity deviations at higher mole fractions of DAA-H_2O due to weak interactions between the molecules [see Figure 1(b)]. The changes in enthalpy, entropy of mixing, size and shape of molecules also significantly influence the viscosity deviations [55,56].

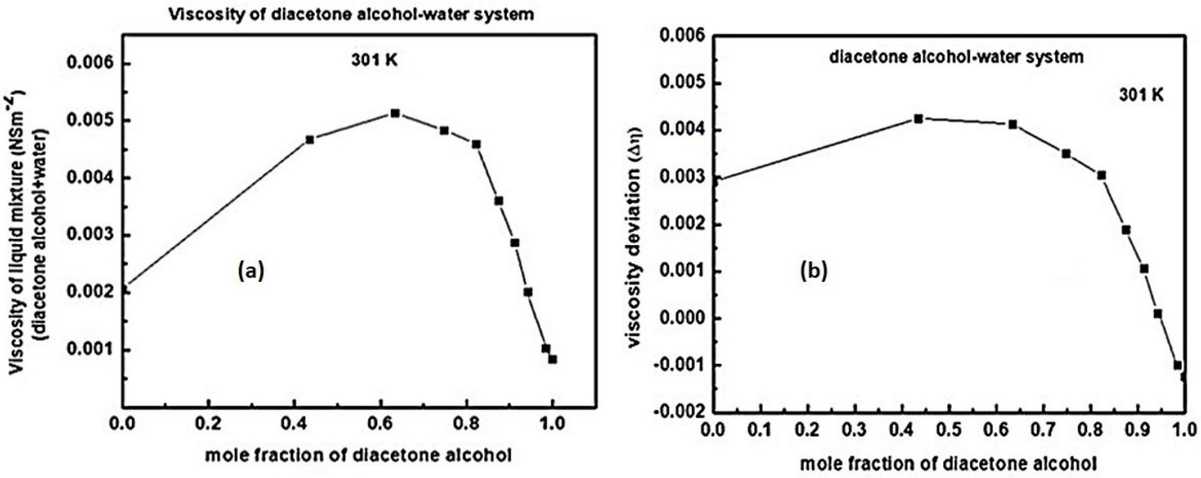

Figure 1. a) viscosity of liquid mixture (DAA+water system), b) viscosity deviation (Δη) v/s mole fraction of diacetone alcohol

The results of surface tension of the binary liquid mixtures measured at 301 K are given in Table 1. As shown in Figure 2(a), surface tension increases gradually with the increase in mole fraction of DAA. The increase in surface tension with an increase in the mole fraction of DAA concentration indicates strong intermolecular forces, a more homogeneous and stable binary liquid mixture/s [39,57].

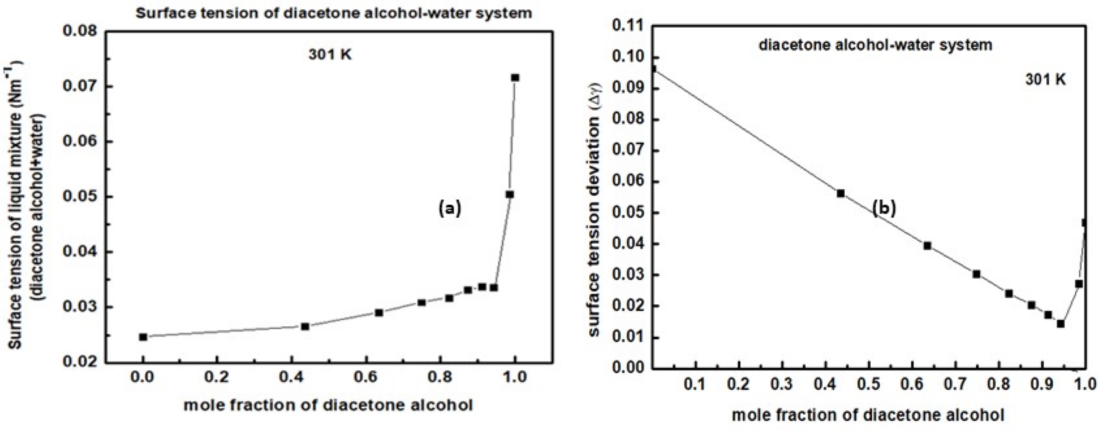

Figure 2. Surface tension of a) liquid mixture (DAA+water), b) deviation (Δγ) of liquid mixture (DAA+water system)

Figure 2(b) shows the surface tension deviations observed for binary liquid mixtures. The value of $\Delta\gamma$ provides insights into the molecular interactions and the structure of the liquid-vapour interface, information related to the difference in the activity components between the bulk phase and the surface phase at the liquid-vapour interface. This ratio provides deep insights into the non-ideal behavior resulting from intermolecular forces upon mixing.

The magnitude and sign of surface tension deviation directly reflect the behavior of the components at the interface compared to the bulk solution. The liquid with the lower surface tension is considered surface active and will preferentially migrate (adsorb) to the surface to minimize the total surface energy of the system. The decrease in the surface tension deviation with an increase in the mole fraction indicates that the binary liquid mixtures have lower surface tension than that of pure liquids/ideal surface tension. It indicates that the one with lower surface tension is highly effective at minimizing the surface energy by strongly accumulating at the interface, often associated with minimal surface energy.

Figure 3 shows the variation in apparent molar volume (ϕ_V) with composition, and the results have been given in Table 1. The decrease in ϕ_V with an increase in the mole fraction of water indicates strong intermolecular interactions between DAA and water in the binary liquid mixtures, resulting in volume contraction upon mixing. This indicates a more compact structure in the binary liquid mixture compared to the pure components.

Excess volume (V^E) indicates volume change upon mixing, and the negative V^E suggests volume contraction and strong attractive forces (hydrogen bonding/strong dipole-dipole interactions) between the molecules that allow closer packing. A positive V^E indicates volume expansion and weak repulsive forces or disruption of the structure of the pure liquids. Density and viscosity were measured for pure components and across the entire composition range of the trichloromethane (1) + 2-butanone (2) binary system at various temperatures. Vapor-liquid equilibrium (VLE) data collected at T = 303.15 K have been used to calculate the activity coefficients and the excess molar Gibbs function. This system exhibits negative deviations from ideal behavior and forms a minimum pressure azeotrope at a trichloromethane mole fraction of x=0.19 and a pressure p =15.3 kPa [16].

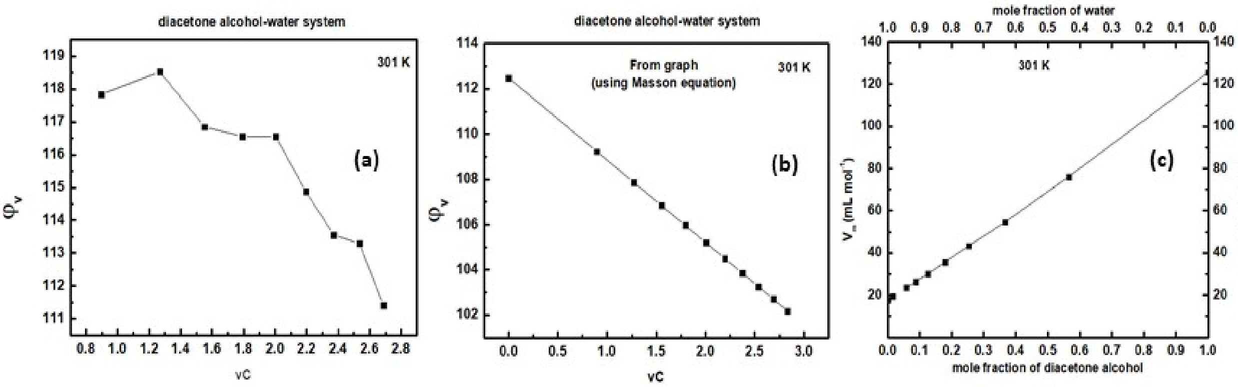

Figure 3. a) apparent molar volume of a binary liquid mixture, b) apparent molar volume of a binary liquid mixture [data from Figure 1(a) used to fit Masson equation], c) partial molar volume ($\bar{V}_m$) liquid mixture

The mixtures of triethyl phosphate (TEP) with N-methylformamide (NMF), N,N-dimethylformamide (DMF) and N,N-dimethylacetamide (DMA) all exhibited positive viscosity deviations and excess Gibbs energy. However, excess volume and excess isentropic compressibility were negative for the DMA and DMF mixtures (attributed to dipole-dipole interactions), but positive for the NMF mixture, indicating TEP acts as a structure breaker for the self-associated NMF [17]. Measurements of excess molar volume and viscosity for binary mixtures of ethylenediamine and water at various temperatures were conducted. All mixtures consistently displayed negative values, which is attributed to the enhanced specific interactions occurring between the amino and hydroxyl groups of the two components [38]. Density and viscosity data for binary mixtures of diacetone alcohol with both benzene and chlorobenzene across the full composition range at temperatures of 303.15 K, 313.15 K and 323.15 K have been reported. A systematic variation in the viscosity and excess property values with increasing temperature was observed, which reflects a decrease in the strength of intermolecular interactions between the molecules [39]. The density and viscosity of four chlorides (magnesium chloride, ammonium chloride, cupric chloride, aluminium chloride) were measured in a methanol-water solvent across three temperatures 303.15, 308.15 and 313.15 K. Calculations of the apparent specific volume and the Jones-Dole B-coefficient showed that magnesium chloride is a structure maker and ammonium chloride is a structure breaker. Importantly, both cupric chloride and aluminium chloride were also characterized as structure-making salts in the mixed solvent system [50]. The solubility of diacetone acrylamide (DAAM) was measured gravimetrically in sixteen distinct organic solvents (including various alcohols, ketones, and esters) at ambient pressure across temperatures from 272.05 K to 323.65 K. The data were successfully correlated using the NRTL and Wilson models, with the Apelblat equation providing the best agreement, yielding maximum deviations (relative average deviation) and (root-mean-square deviation) of approximately 2.4×10^{-2} [52]. The diacetone alcohol and chlorobenzene mixture exhibits its strongest interaction at lower frequencies, and the changes in the sign and magnitude of the excess parameters reveal the decrease in the extent of interaction as the frequency is increased [57]. The binary liquid mixtures of different types of alcohols with propan-2-amine at different compositions and temperatures exhibit negative V^E values due to strong interactions and have been fitted with Redlich-Kister type polynomial equation [58].

The limiting apparent molar volume (ϕ_V^O) and the experimental slope (S_V) were calculated from the Masson equation (4) [47,48]:

$$\phi_v = \phi_v^o + S_v\sqrt{c} \quad (4)$$

where ϕ_V: the apparent molar volume; ϕ_v^o: the apparent molar volume at infinite dilution; S_V is the experimental slope, and c: the molar concentration of the DAA-water in the binary liquid mixtures. The calculated values for ϕ_v^o are tabulated in Table 1, and the corresponding values of S_v were obtained by the least squares method, which yielded negative values for all the liquid binary mixtures (DAA-water), indicating strong intermolecular interactions due to volume contraction and enhanced structural ordering. The partial molar volumes of the binary liquid mixtures lie between the molar volumes of pure water and diacetone alcohol (DAA), indicating the extensive intermolecular interactions between DAA and water molecules (Figure 3). These interactions were also supported by excess volume (V^E), and the deviations are shown in Figure 4 [56]. The negative V^E values at all the compositions of DAA-H_2O mixture are due to volume contraction and dominant associative forces in these compared to their pure components, thus forming a more compact and stable mixture [16,29].

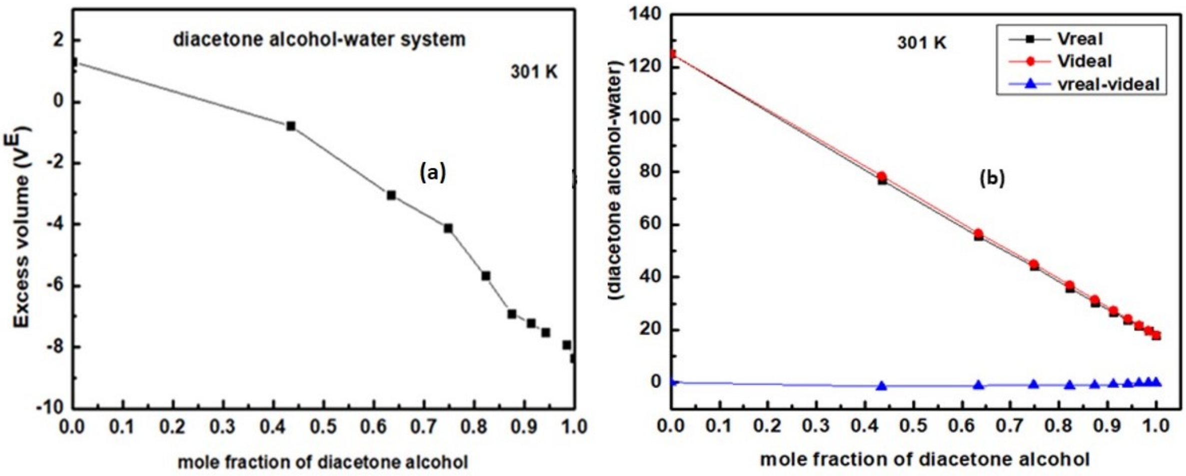

Figure 4. a) excess volume (V^E) v/s mole fraction of diacetone alcohol, b) volume changes ($\Delta\bar{V}_m$) in liquid mixture (DAA+water system)

From the experimental results, the partial molar volumes for each component mixture were calculated, and the data are shown in Figure 3(c). The volume changes of the liquid binary mixtures decrease with an increase in the mole fraction of the diacetone alcohol. This is shown in Figure 4(b). This indicates negative deviation from Raoult's law, further confirming the strong dipole-dipole intermolecular interactions.

4. Conclusion

Density, viscosity, limiting apparent molar volume, and partial molar volume studies of the liquid binary mixtures (DAA-H_2O) have been examined. Strong intermolecular interactions exist in the DAA-water system, which is evident from the experimental data. The negative excess volume change has been observed with an increase in the DAA mole fraction at 301 K, indicating stronger interactions due to volume contraction. Thus, the volumetric, viscometric and surface tension data provide a comprehensive understanding of the non-ideal behavior of the liquid binary mixtures (DAA-H_2O). The experimental data play an important role in the optimization of conditions and provide better insights into the procedures adopted in polymer coating technologies, solvent formulation and separation techniques.

Conflict of Interest

We declare no conflict regarding the publication of the study.

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