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❖ Introduction:

Molecular interactions play a crucial role in determining the physical, chemical and electrical properties of substances across various states of matter, including solids, liquids and gases [1]. These interactions arise due to attractive and repulsive forces between molecules, significantly influencing material science, drug development, chemical engineering, nanotechnology and various industrial applications [2]. Understanding binary liquid mixtures is crucial for gaining insights into molecular structure, chemical reactivity, energy transfer in biological systems and phase transitions [3-6]. These studies help in deciphering solute-solvent interactions, hydrogen bonding networks and dipole-dipole interactions, which are essential for applications in chemical engineering, pharmaceuticals and materials science. Molecular interactions also play a key role in industrial processes, such as transportation, chemical manufacturing and energy storage, where optimizing these interactions enhances efficiency and performance [7]. Several advanced experimental techniques are used to investigate molecular interactions, including Dielectric Relaxation Spectroscopy (DRS), Nuclear Magnetic Resonance (NMR) Spectroscopy, Fourier-Transform Infrared (FTIR) Spectroscopy, Raman Spectroscopy, Infrared Spectroscopy. [8-17]. Among these, DRS is a particularly powerful method for analysing the dielectric properties of pure and mixed liquid systems, providing essential information on molecular polarization, dipolar interactions and charge transport mechanisms [18]. At low frequencies (20 Hz to 2 MHz), DRS provides insights into charge dynamics, ionic conductivity, and electrode polarization effects, while at higher frequencies (hundreds of MHz to 20 GHz), it reveals information about dipolar relaxation processes, dielectric loss and molecular orientation [19-28]. Studying these properties across different temperatures enables the determination of thermodynamic parameters, such as enthalpy (ΔH), entropy (ΔS) and activation energy (ΔEa), which help explain the energy landscape of molecular interactions [29-36]. Broadband DRS, which measures complex permittivity ($\varepsilon^*(f)$) over a frequency range from 1 μHz to several THz, offers a comprehensive understanding of molecular fluctuations, collective dipolar relaxation, charge transport, and polarization effects in liquid systems [35-42]. This technique is particularly valuable for investigating polar liquids, where electrode polarization, hydrogen bonding interactions, and ionic conduction play a significant role in defining the macroscopic electrical and physicochemical properties of the system [41-42].

Furthermore, the study of molecular interactions and their associated physico-chemical properties, such as density (ρ), viscosity (η) and other related parameters in liquid binary mixtures at varying concentrations and temperatures, is essential for both industrial and academic research. Understanding these properties provides valuable insights into the thermodynamic behavior of mixtures, which is crucial for designing, modeling, and optimizing equipment such as condensers, heat transfer devices, distillation columns, and other process systems [43].

A comprehensive understanding of the physico-chemical properties of multicomponent systems is particularly critical in industrial operations, especially in chemical engineering, where it plays a key role in developing new models for engineering applications. Additionally, optical and acoustic properties, such as the refractive index (n) and ultrasonic speed (U), provide further understanding of the physical nature and intermolecular forces in liquid systems [44]. To gain deeper insights into molecular interactions, it is necessary to examine the physico-chemical properties of liquids, including the formation and disruption of hydrogen bonds, dipole-dipole interactions, and dipole-induced dipole interactions [45-48].

In the present study it has been systematically planned to explore the molecular interaction study of binary mixtures of some higher order alcohols (n-Hexanol, n-Octanol) with amide (N, N dimethylformamide) at varying concentrations and temperatures over the wide frequency span using multi experimental approach.

❖ Brief Research Methodology:

In this study, binary mixtures of n-Hexanol with DMF and n-Octanol with DMF were prepared at eleven different volume fractions (0.0→1.0) in sealed glass vials to prevent contamination and ensure accurate composition. The volume fractions were converted into mole fractions using the respective densities and molecular weights. High-purity chemicals (n-Hexanol and DMF, AR grade; n-Octanol, synthesis grade) were obtained from Loba Chemie Pvt Ltd, India, and used without further purification. Purity was verified by measuring the refractive index using Abbe's refractometer, with an accuracy of ±0.001. Dielectric properties were measured across a wide frequency range using an Agilent Precision LCR meter (20 Hz–2 MHz) with a four-terminal liquid dielectric test fixture and an Anritsu Shockline Vector Network Analyzer (200 MHz–20 GHz) with a SPEAG-3.5 Dielectric Assessment Kit. Measurements were performed at four different temperatures (293.15 K, 303.15 K, and 313.15 K) with a constant temperature water bath (accuracy ±0.1 K). Additional physicochemical properties were measured as follows: density (using specific gravity bottle), viscosity with an Ostwald viscometer (accuracy ±0.1%), ultrasonic velocity using an ultrasonic interferometer operating at 2 MHz and refractive index with Abbe's refractometer.

These measurements provided a comprehensive understanding of the dielectric behavior, molecular interactions and structural dynamics of the binary mixtures, offering valuable insights into their thermophysical properties across different concentrations and temperatures.

The thesis entitles "**MOLECULAR INTERACTION STUDIES OF SOME AMIDES WITH ASSOCIATIVE MOLECULES**" consists of nine chapters.

- ***Chapter-1:*** Introduction

  The importance of study of dielectric relaxation spectroscopy and physico-chemical properties of liquid materials is given in this chapter. An overview of dielectric spectroscopy (BDS), refractometric and physiochemical methods are also given in this chapter. Major findings from the work done by various researchers on dielectric, refractometric, acoustic and volumetric study of various liquid systems have been given in brief. Applications of the studied compound (n-Hexanol, n-Octanol and N, N-Dimethylformamide (DMF)) in different areas and their some basic physico-chemical properties is provided in it. The aim and objective of the present study is also explained in the present chapter.

- ***Chapter-2:*** Theoretical Backgrounds

  This chapter mainly includes the fundamental theory of dielectrics, exploring dielectric properties such as dipole moments and types of polarization, as well as the theory of dielectric relaxation, permittivity, and the Clausius-Mossotti equation. It encompasses static permittivity theories like Debye, Onsager, Kirkwood, and Frohlich, along with dynamic permittivity theories, detailing various relaxation models including Havriliak-Negami, Cole-Cole, Debye and Cole- Davidsons. The chapter examines the relationship between dielectric parameters and molecular behavior, alongside thermodynamic and physico-chemical properties such as ultrasonic velocity (u), density (ρ), refractive index (n) and viscosity (η), and other related parameters. Basic theory of electrochemical impedance spectroscopy are also given in this chapter.

- ***Chapter-3:*** Experimental Procedures and Evaluation of Different Parameters

  This chapter provides detailed information on the materials and sample preparation processes. It describes the experimental methods and setups for measuring various dielectric and physico-chemical properties. Techniques for determining the complex permittivity (ε*) of liquid samples across different frequency ranges are explained, including the use of a Precision LCR meter for the 20 Hz to 2 MHz range and a Vector Network Analyzer (VNA) for the 200 MHz to 20 GHz range. The chapter also covers methods for measuring ultrasonic velocity (U) using an ultrasonic interferometer (2 MHz), density (ρ) using a specific gravity bottle, refractive index using Abb's refractometer, and viscosity (η) using an Ostwald viscometer. From the experimentally determined values of complex permittivity, refractive index, ultrasonic velocity, density, and viscosity of liquid mixtures, various other dielectric, electrical, physico-chemical, and thermodynamic parameters are evaluated. Brief details about the evaluation of these parameters are also provided in this chapter.

- ***Chapter-4:*** Dielectric Relaxation Study of Binary Mixtures (n-Hexanol+ N, N-Dimethylformamide)

  The detailed study of the dielectric relaxation spectroscopy (DRS) study conducted over broad range of frequencies (20 Hz to 2MHz) and (200 MHz to 20 GHz) at different temperatures (293.15 K, 303.15 K, 313.15 K) for binary mixture of n-Hexanol with N, N Dimethylformamide is given in this chapter.

  Dielectric and electrical properties of the binary mixtures of n-Hexanol and N, N-Dimethylformamide have been studied in the frequency range 20 Hz to 2 MHz at various temperatures. Different electrical parameters such as electrical modulus (M*(f)), electrical conductivity (σ*(f)) and complex impedance (Z*(f)) were derived from the complex permittivity spectra (ε*(f)). Ionic polarization relaxation time (τσ) and DC conductivity (σdc) were calculated from different dielectric and electrical formalism. With the help of dielectric parameters and DC conductivity, different parameters such as Debye length (λd), ion mobility (μ), mobile ion concentration (P0) and ion diffusivity (D) have been determined. Complex impedance spectra were also fitted to RC equivalent circuit using EC-Lab software. The Complex permittivity (ε*) spectra of liquid samples are obtained in frequency span (200 MHz to 20 GHz) using vector network analyser (VNA) at three different temperatures. The frequency dependence complex permittivity spectra of liquid samples were analyzed in the framework of Debye (DB), Cole-Cole and Cole-Davidson dielectric models using complex nonlinear least squares (CNLS) fitting (LEVMW Software) techniques. The critical comparison of the dielectric spectra obtained from these models are discussed and the best fit relaxation model is determined. Static dielectric permittivity (ε_0) and permittivity at optical frequency (ε_∞) of binary mixtures of n-Hexanol and DMF is determined at different temperatures. From these measured values, its excess static permittivity $(\varepsilon_0)^E$ and excess permittivity at optical frequency $(\varepsilon_\infty)^E$ are calculated and fitted to the Redlich-Kister equation. Effective correction factor (g_{eff}) and corrective correlation factor (g^f) are evaluated from the modified Kirkwood equation. Additionally, experimental data of static permittivity are fitted to the modified Bruggman equation to find Bruggman factor (F_B). The variations of these parameters are discussed in terms of molecular structures, inter molecular and dipole-dipole interactions. Further, the determined relaxation time at different temperatures were used to evaluate thermodynamic parameters, and these parameters are interpreted in terms of the force of hindrance experienced by the molecules and their orientation in the liquid mixtures.

- ***Chapter-5:*** Dielectric relaxation study and Electrochemical impedance Spectroscopy (EIS)of binary mixtures (n-Octanol + N, N Dimethylformamide) In this chapter, results of the DRS study conducted over broad range of frequencies (20 Hz to 2MHz) and (200 MHz to 20 GHz) at different temperatures for binary mixtures of n-Octanol and N, N-Dimethylformamide are reported. Different dielectric and electrical properties such as complex permittivity $\varepsilon^*(\omega)$, loss tangent ($\tan \delta$), complex electric modulus $M^*(\omega)$, complex conductivity $\sigma^*(\omega)$ and complex impedance $Z^*(\omega)$ have been investigated at different temperatures. Using these properties different parameters such as electrode polarization relaxation time (τ_{EP}), ionic conductivity relaxation time (τ_σ) and D.C conductivity (σdc) have been calculated. Complex impedance spectra have been fitted to RC equivalent circuit using EC-Lab software. Different equivalent circuit model parameters such as R1, R2, C1 and C2 were determined and reported. Geometric relaxation time (τg) was determined from different fitting parameters. Complex permittivity spectra for binary mixtures of n-Octanol and N, N Dimethylformamide with varying concentration (0.0→1.0) in microwave frequency range (200 MHz to 20 GHz) at different temperatures are also studied. Using CNSL fitting method, the complex permittivity data are fitted to different model (Debye (DB), Cole-Cole and Cole Davidson). Dipolar relaxation time for the mixtures was determined from the fitted parameters for all mixture concentrations. Static dielectric permittivity (ε_0) and permittivity at optical frequency (ε_∞) of binary mixtures of n-Octanol and N, N-Dimethylformamide (DMF) is determined at various temperatures (293.15 K, 303.15 K, 313.15 K). From these measured values, the critical comparison of the dielectric spectra obtained from these models are discussed and the best fit relaxation model is determined. The static dielectric constant (ε_0) and relaxation time (τ_0) are obtained. Dielectric constant at optical frequency (ε_∞) of liquid samples are determined. From these various parameters namely; excess static dielectric constant $(\varepsilon_0)^E$, excess dielectric constant at optical frequency $(\varepsilon_\infty)^E$, Kirkwood parameters (g_{eff} and g^F) and Bruggeman parameter (FB) are determined. Furthermore evaluated microwave radiation heating parameters like power reflected (pr), power transmitted (pt) and penetration depth (dp) at different temperature are investigated at general purpose and commercial microwave radiation 2.45 GHz. Variation of these parameters are discussed considering molecular interaction between molecular species. Further, the determined relaxation time at different temperatures were used to evaluate thermodynamic parameters, and these parameters are interpreted in terms of the force of hindrance experienced by the molecules and their orientation in the liquid mixtures.

- ***Chapter-6:*** Acoustic, Volumetric and IR spectra study of binary mixtures of n-Hexanol and N,N Dimethylformamide

  In this chapter experimentally measured values of ultrasonic velocity (u), Density (ρ), Viscosity (η) over temperature range of 293.15 K to 313.15 K (interval of 10 K) are reported. From the experimental data the physiochemical (Acoustic and Volumetric) parameters such as molar volume (V_m), adiabatic compressibility (β), intermolecular free length (L_f), acoustic impedance (Z), molar sound velocity (R), molar compressibility (B), surface tension (σ) internal pressure (Pint) and relaxation time (τ) were calculated, which are more useful to predict and confirm the molecular interaction in the binary liquid mixtures. Excess of measured acoustic parameters have been estimated and fitted in Redlich -Kister polynomial. Variations of these parameters are discussed in the light of molecular interaction in the mixture species. Their conformational study is supported by Fourier Transform Infrared (FTIR). The effect of concentration and temperature variation on the acoustical and volumetric parameters of the binary mixture of n-Hexanol and N, N-Dimethylformamide are investigated.

- ***Chapter-7:*** Acoustic, Volumetric and Thermodynamic study of binary mixtures of n-Octanol and N,N Dimethylformamide

  This chapter contains experimentally measured values of ultrasonic velocity (u), Density (ρ) and Viscosity (η) for binary mixtures of n-Octanol and N, N-Dimethylformamide at different temperatures (293.15 K, 303.15K, 313.15 K) are reported. From the experimental data the physiochemical parameters such as molar volume (Vm), adiabatic compressibility (β), intermolecular free length (Lf), acoustic impedance (Z), molar sound velocity (R), molar compressibility (B), relaxation strength (r), surface tension (σ), internal pressure (Pint), free volume (Vf) and relaxation time (τ), enthalpy (ΔH), entropy (ΔS), Gibbs free energy (ΔG) were calculated, which are more useful to predict and confirm the molecular interaction in the binary liquid mixtures. The Redlich-Kister equation has been associated with the excess and deviation functions that have been computed from experimental data. These functions were further discussed in terms of molecular interactions existing in the mixtures.

- ***Chapter-8:*** Study of Refractometric and various mixing relations of Binary mixtures (n-Hexanol + N, N-Dimethylformamide, n-Octanol + N, N-Dimethylformamide)

  This presents temperature dependent refractometric study of binary mixtures of n-Hexanol with N, N-Dimethylformamide over varying concentration of composition. Furthermore, the validation of various mixing relations for static dielectric constant (ε_0), refractive index (n), ultrasonic velocity (u) and viscosity (η) of binary mixtures of (n-Hexanol +N, N-Dimethylformamide, n-Octanol + N, N-Dimethylformamide) systems. A comparison of the predicted values of static dielectric constant (ε_0), refractive index (n), ultrasonic velocity (v) and viscosity (η) of the liquid mixtures using various mixing relations with the experimentally determined values have been assessed in terms of Average Percentage Deviation (APD).

- ***Chapter-9:*** The comprehensive summary of the research work is provided in this chapter. The investigation's major findings are reported. The scope for further investigation based on the findings and experience of the current study is given in this chapter.

❖ Key Findings:

The study revealed important insights into the molecular interactions and physical properties of n-Hexanol + DMF and n-Octanol + DMF binary mixtures. Dielectric measurements across a wide frequency range showed how the Both systems (n-Hexanol + DMF and n-Octanol + DMF) responded in high frequency varying fields. DRS study helped to understand the relaxation mechanism of constituents of molecular species in different molecular environments. EIS study with a six-element equivalent RC circuit confirms various electrical processes in the bulk sample and at the electrode surface. The observed perfect scaling in dielectric properties (tanδ, M″ and Z″) with concentration variation indicates ability for tuning the electrical and dielectric properties of binary mixtures (n-Hexanol + DMF, n-Octanol + DMF). The variation of DRS parameters, EIS parameters, refractometric parameters and acoustics parameters suggests the formation of molecular complexion with different molecular stoichiometric ratios in both the studied systems. Thermodynamic parameters such as Gibbs free energy, entropy, and internal pressure provided further insights into molecular rearrangements. Finally, experimental data were compared with theoretical models, and the Average Percentage Deviation (APD) analysis validated the accuracy of the study.

These findings contribute to a better understanding of dielectric relaxation, solute-solvent interactions, and molecular dynamics, which are essential for applications in chemical, pharmaceutical and material sciences.

❖ Conclusion:

This study comprehensively investigates the dielectric, ultrasonic, volumetric, and refractometric properties of binary mixtures of n-Hexanol + DMF and n-Octanol + DMF across varying temperatures and frequencies to elucidate molecular interactions and relaxation dynamics. Dielectric spectroscopy revealed significant frequency-dependent polarization effects, with ionic conduction and electrode polarization dominating at low frequencies and dipolar relaxation processes governing the microwave region. The Kirkwood correlation factor and Bruggeman factor confirmed the presence of hydrogen bonding and dipole-dipole interactions, with concentration-dependent variations indicating structural rearrangements. Ultrasonic and volumetric studies demonstrated nonlinear variations in compressibility, free volume, and acoustic impedance, further supporting the role of intermolecular forces in determining the system's thermophysical behavior. Refractometric analysis indicated negative deviations in excess refractive index and molar volume, suggesting hydrogen bond disruption and weaker molecular associations at elevated temperatures. The applicability of various mixing models was assessed, with the Arrhenius-Eyring model providing the most accurate viscosity predictions and the Lorentz-Lorentz relation best describing refractive index trends.

Overall, this study provides critical insights into solute-solvent interactions, dielectric relaxation mechanisms, and thermodynamic stability, contributing to the fundamental understanding of amide-alcohol systems with implications for material science, pharmaceuticals and industrial applications.

❖ Recommendation and Suggestions:

Future research should extend the study to binary mixtures of DMF with long-chain alcohols like n-Nonanol and n-Decanol to examine how alkyl chain length and molecular structure affect dielectric relaxation, viscosity and ultrasonic velocity. Advanced spectroscopic techniques such as Raman, FTIR, and NMR (^{1}H-NMR, ^{13}C-NMR) should be used to analyze hydrogen bonding, molecular conformation, and solvation dynamics at a molecular level. Molecular dynamics (MD) simulations can complement experimental findings by providing insights into hydrogen bonding networks, diffusion behavior and solvation structures under different conditions. Expanding dielectric spectroscopy to high-frequency microwave and terahertz ranges would allow a better understanding of fast molecular dynamics and short-range interactions. Additionally, calorimetric techniques; heat capacity and thermal conductivity measurements should be used to quantify enthalpy and entropy contributions to molecular interactions. These approaches will provide a comprehensive understanding of physicochemical properties, optimizing applications in chemical, pharmaceutical and material science industries.

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