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Determination of Viscosity of 4,5-Dihydro Substituted Oxazoles in DMF - Water Mixture

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Abstract

The present study focuses on the determination of viscosity of 4,5-Dihydro substituted oxazoles in DMF–water mixtures at various compositions and temperatures to understand solute–solvent interactions. The viscosity measurements were carried out using an Ostwald viscometer under controlled temperature conditions. The effect of solvent composition, concentration, and temperature on the viscosity of the substituted oxazoles was systematically studied. The experimental data were analyzed using Jones–Dole equations to obtain the viscosity A and B coefficients, which provide insight into the ion–ion and ion–solvent interactions, respectively. The results reveal that viscosity decreases with increasing DMF content due to a reduction in hydrogen bonding and cohesive forces in the medium. The positive values of the B-coefficient suggest strong solvation of the substituted oxazoles in the mixed solvent system. These findings contribute to a better understanding of the molecular interactions and structural behavior of oxazole derivatives in mixed solvent environments, which is significant for their physicochemical and biological studies.

Keywords: Viscosity, 4,5-Dihydro Substituted oxazoles, DMF–water mixture, Jones–Dole equation, Solute–solvent interaction.

Introduction

The study of viscosity is an important tool for understanding the molecular interactions and solution behavior of organic compounds in various solvent systems. Viscosity measurements provide valuable information about the nature and extent of solute–solute and solute–solvent interactions, which influence several physicochemical parameters such as diffusion, solvation, association, and reactivity [1-5]. In mixed solvent systems, the variations in dielectric constant, polarity, and hydrogen-bonding capacity allow for systematic investigation of molecular interactions and structural effects.

4,5-Dihydro Oxazoles, being five-membered heterocyclic compounds containing oxygen and nitrogen atoms, have attracted significant attention because of their biological, pharmacological, and chemical significance. Substituted oxazoles exhibit a wide range of biological activities, including antimicrobial, anti-inflammatory, and anticancer properties. Understanding their physicochemical behavior in solution helps predict their stability, solvation, and binding behavior, which are important for both chemical reactivity and biological activity [6-10].

Dimethylformamide (DMF) is an aprotic, polar solvent that exhibits strong solvation ability for both organic and inorganic solutes. It is completely miscible with water, and the DMF–water mixed solvent system offers a broad range of dielectric constants. The addition of DMF to water modifies the structure of the solvent, influencing viscosity, density, and dielectric behavior. Therefore, DMF–water mixtures are suitable media for studying solute–solvent interactions of organic molecules such as substituted oxazoles [11-14]. Several researchers have investigated the effect of solvent composition on the viscosity behavior of organic and bioactive compounds. Studies on amino acids, peptides, and heterocyclic compounds in mixed solvent systems have demonstrated that viscosity measurements serve as a reliable method for understanding molecular association and solvation dynamics. Researchers such as Jadhav *et al*. and Patil *et al*. [15-19] have reported that viscosity parameters vary significantly with solvent polarity and temperature, indicating changes in molecular interactions. Similar investigations on nitrogen- and oxygen-containing heterocycles have revealed positive B-coefficient values, suggesting strong solute–solvent interactions due to hydrogen bonding and dipole–dipole forces. However, limited data are available on substituted oxazoles in DMF–water media. Hence,

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the present study aims to fill this gap by systematically analyzing the viscosity behavior of substituted oxazoles in mixed solvent environments to elucidate their structural and interactional characteristics [20].

In the present work, the viscosity of 4,5-Dihydro substituted oxazoles was determined in DMF–water mixtures of different compositions and at various temperatures using an Ostwald viscometer. The data were analyzed with the Jones–Dole equation to evaluate the A and B coefficients, which provide insight into the ion–ion and ion–solvent interactions, respectively. These results help in understanding the solvation characteristics and structural influence of 4,5-Dihydro oxazole derivatives in mixed solvents.

Material and Methods

Ligands were prepared by advance chemical and microwave methods. The solvents used were of AR grade and doubly distilled water was used. Weighings were made on one pan digital balance (Adair Dutt 180) (± 0.001gm). Densities of solutions were determined by a bicarpellary pyknometer having a bulb volume of about 10cm^3 and capillary having an internal diameter of 1mm and calibrated with deionised doubly distilled water (0.994 X 10^{3}kgm^{-3} at 303.5°C). The viscosities were measured by means of Ostwald's Viscometer (± 0.11% kgm^{-1}s^{-1}) which was kept in equilibrium with Elite thermostatic water bath (± 0.1°C). Ligand solutions of different concentrations were prepared in DMF. For each measurement, sufficient time was allowed to attain thermal equilibrium in thermostat.

Results and Discussion

The relative viscosity of each solution is determined by following empirical formula-

$$\eta_r = (d_s \times t_s) / (d_o \times t_o) \qquad (2)$$

Where,

η_r is relative viscosity of ligand solution.
d_s is density of ligand solution.
d_o is density of dimethylformamide
t_o is time of flow for dimethylformamide
t_s is time of flow for ligand solution.

The relative viscosity and density data for different ligands at different concentration are presented in Table 1 to 5.

The relative viscosities have been analyzed by Jones-Dole equation[17]

$$(\eta_r - 1) / \sqrt{c} = A + B\sqrt{c} \qquad (3)$$

Where, C is the molar concentration of the ligand solution.

A is the Falkenhagen coefficient which is the measure of solute-solute interactions and B is the Jones-Dole coefficient which is the measure of solute-solvent interactions.

The graphs are plotted between $(\eta_r - 1) / \sqrt{c}$ versus $\sqrt{c}$. The graph for each system gives linear straight line showing validity of Jones-Dole equation. The slope of straight line gives value of β-coefficient. The plots of $(\eta_r - 1) / \sqrt{c}$ versus $\sqrt{c}$ for some systems are shown in Fig 1 to 4.

In the present study, relative viscosity of ligand solution decreases with increase in the concentration of ligand. The decrease in viscosity with increase in concentration may be attributed to the decrease in solute-solvent interactions. From the graph of $(\eta_r - 1) / \sqrt{c}$ versus $\sqrt{c}$, 'A' which is the measure of solute-solute interactions and 'B' which is the measure of solute-solvent interactions has been calculated. The large and small values of 'A' show the stronger and weaker solute-solute interactions respectively as listed in Table 6.

When the solute is introduced into solvent of organic-water mixture, it will interfere with the ordered structure of water in solute's co sphere. It is evident from Table 6, that the β-coefficient is an adjustable parameter, which may be either positive or negative and it is a measure of hydrodynamic volume of solute which accounts for the solute-solvent interactions. It is known as a measure of order or disorder introduced by the solute into the solvent. Solutes with positive viscosity β-coefficients are characterized "Structure formers" and will impose a new order by reorientation of the adjacent water molecules indicating strong solute-solvent interactions. Solutes with negative viscosity β-coefficient are characterized as "Structure breakers" indicating weak solute-solvent interactions. Such type of results is also shown [26].

Table 1: System - Ligand - L_1

Temp: 27 ± 0.1^{O}C
Medium: 70% DMF-Water

Conc (C) mole/lit	$\sqrt{C}$ x 10^{-2} mole$^{-1/2}$lit$^{-1/2}$	Density gm/cc	Time Flow (Secs)	Relative Viscosity $\eta_r = \eta_l / \eta_w$	Sp. Viscosity $\eta_{sp} = \eta_r - 1/\sqrt{C}$
0.0025	5.00	0.9478	31.00	1.3877	7.7542
0.0035	5.91	0.9471	30.30	1.3553	6.0131
0.0055	7.41	0.9453	29.30	1.3081	4.1586
0.0075	8.66	0.9439	28.30	1.2616	3.0212
0.0100	10.00	0.9429	27.30	1.2157	2.1570

Table 2: System - Ligand – L2

Temp: 27 ± 0.1^{O}C
Medium: 70% DMF-Water

Conc (C) mole/lit	$\sqrt{C}$ x 10^{-2} mole$^{-1/2}$lit$^{-1/2}$	Density gm/cc	Time Flow (Secs)	Relative Viscosity $\eta_r = \eta_l / \eta_w$	Sp. Viscosity $\eta_{sp} = \eta_r - 1/\sqrt{C}$
0.0025	5.00	0.9980	30.30	1.4282	8.5643
0.0035	5.91	0.9974	29.60	1.3943	6.6731
0.0055	7.41	0.9949	27.30	1.2828	3.8166
0.0075	8.66	0.9928	26.30	1.2332	2.6930
0.0100	10.00	0.9910	26.00	1.2169	2.1693

Table 3: System - Ligand – L4

Temp: 27 ± 0.1^{O}C
Medium: 70% DMF-Water

Conc (C) mole/lit	$\sqrt{C}$ x 10^{-2} mole$^{-1/2}$lit$^{-1/2}$	Density gm/cc	Time Flow (Secs)	Relative Viscosity $\eta_r = \eta_l / \eta_w$	Sp. Viscosity $\eta_{sp} = \eta_r - 1/\sqrt{C}$
0.0025	5.00	0.9878	28.30	1.3203	6.4060
0.0035	5.91	0.9873	27.66	1.2898	4.9035
0.0055	7.41	0.9860	27.00	1.2573	3.4723
0.0075	8.66	0.9837	26.00	1.2079	2.4006
0.0100	10.00	0.9825	25.00	1.1600	1.6000

Table 4: System - Ligand – L5

Temp: 27 ± 0.1^{O}C
Medium: 70% DMF-Water

Conc (C) mole/lit	√C x 10^{-2} mole$^{-1/2}$lit$^{-1/2}$	Density gm/cc	Time Flow (Secs)	Relative Viscosity $\eta_r = \eta_l / \eta_w$	Sp. Viscosity $\eta_{sp} = \eta_r - 1/\sqrt{C}$
0.0025	5.00	0.9874	33.33	1.5543	11.0870
0.0035	5.91	0.9871	32.66	1.5226	8.8426
0.0055	7.41	0.9854	31.66	1.4734	6.3897
0.0075	8.66	0.9834	30.66	1.4240	4.8966
0.0100	10.00	0.9827	30.00	1.3923	3.9239

Table 5: System - Ligand – L6

Temp: 27 ± 0.1^{O}C
Medium: 70% DMF-Water

Conc (C) mole/lit	√C x 10^{-2} mole$^{-1/2}$ lit$^{-1/2}$	Density gm/cc	Time Flow (Secs)	Relative Viscosity $\eta_r = \eta_l / \eta_w$	Sp. Viscosity $\eta_{sp} = \eta_r - 1/\sqrt{C}$
0.0025	5.00	0.9846	28.30	1.3160	6.3207
0.0035	5.91	0.9844	28.00	1.3018	5.1069
0.0055	7.41	0.9840	27.30	1.2687	3.6269
0.0075	8.66	0.9826	26.60	1.2344	2.7074
0.0100	10.00	0.9804	25.60	1.1854	1.8540

Table 6: A and B coefficient values

Systems	A	B (Lit / mole)
L_1	12.7836	-0.0110
L_2	14.4451	-0.0130
L_4	10.7259	-0.0094
L_5	17.5405	-0.0142
L_6	10.4607	-0.0088

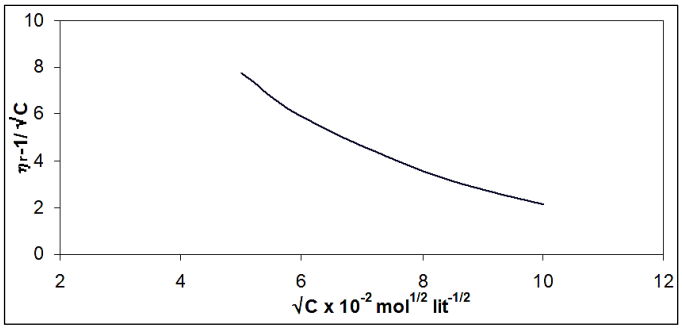

Fig 1:
Plot of √C Vs ηr - 1/ √C
System – L1 in 70% DMF-Water Medium

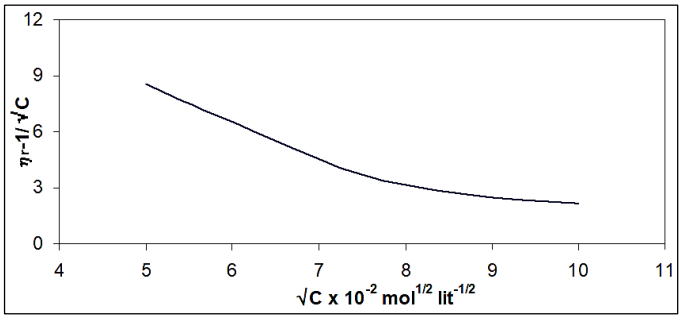

Fig 2
Plot of √C Vs ηr - 1/ √C
System – L2 in 70% DMF-Water Medium

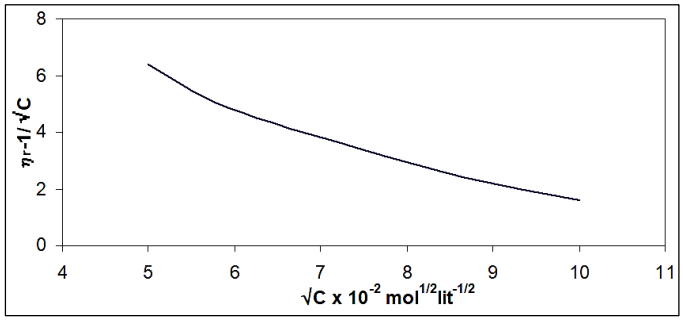

Fig 3
Plot of √C Vs ηr - 1/ √C
System – L4 in 70% DMF-Water Medium

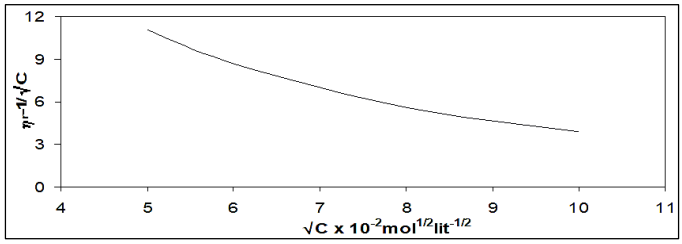

Fig 4:
Plot of √C Vs ηr - 1/ √C
System – L5 in 70% DMF-Water Medium

Viscosity and Thermodynamic Study

Liquid possess viscosity which implies resistance to flow of liquid. It is developed in liquids because of shearing effect of moving one layer of liquid past another. The structure breaking and making properties of liquids have been considered as a measure of solute-solute and solute-solvent interactions [21], Bary and Irving [21] determined the viscosities of concentrated aqueous electrolyte solution at various concentrations. The relative viscosities of ternary aqueous, mixed electrolytic solutions for the systems KBr-NaBr, KBr-Bu_4NBr, NaCl-NaBr and NaCl-Bu_4NBr at various ionic strengths with varying electrolytes mole fractions (at 25°C) have been determined by Patil [22]. Further, he used the data to evaluate the viscosity β-coefficient of the total electrolyte as a function of solute mole fraction. Pandey, Yasmin [17] have measured viscosities and densities of aqueous binary electrolyte solution of different molalities. Mahajan [18] have studied viscosity β-coefficient of sulphonic acid ligands in different percentages of dioxane-water mixtures. Sondawale [22] has also studied the viscosity at different temperature using 20% dioxane-water and methanol-water mixtures. Recently Agrawal [1] has determined the viscosity and some thermodynamic parameters of methyl-5-carboxylate and 1, 3, 4-thiadiazoles in 70% acetone-water mixture.

In the present work relative viscosity have been determined at different temperatures. From the slope of graph between logη_r and 1/T, different thermodynamic parameters like free energy change (ΔG), enthalpy change (ΔH) and entropy change (ΔS) have been determined to study various interactions taking place in solutions of pure electrolytes. All the materials and methods used were as described in Part (a). The temperature was maintained constant with the help of Elite thermostatic water bath (± 0.1°C). For each measurement, sufficient time was allowed to attain thermal equilibrium. Ligands used in present investigation are.

a) L_1 = 3-(2-hydroxy -5-methyl)-5-(2- chlorophenyl)-4,5-dihydro-1,2- oxazole

b) L_2 = 3-(2-hydroxy -5-methyl)-5-(3- chlorophenyl)-4,5-dihydro-1,2- oxazole
c) L_3 = 3-(2-hydroxy -5-methyl)-5-(1H-indol-3-yl)-4,5-dihydro-1,2- oxazole
d) L_4 = 3-(2-hydroxy -5-methyl-3-nitrophenyl)-5-(2-chlorophenyl)-4,5- dihydro-1,2- oxazole
e) L_5 = 3-(2-hydroxy -5-methyl-3-nitrophenyl)-5-(3-chlorophenyl)-4,5- dihydro-1,2- oxazole
f) L_6 = 3-(2-hydroxy-5-methyl-3nitrophenyl)-5-(1H-indol-3-yl)-4,5-dihydro-1,2- oxazole

Results and Discussion

The viscosity of a liquid generally decreases with rise in temperature. The decrease is appreciable, being about two percent per degree rise of temperature in many cases. This has been explained in terms of 'hole theory' of liquids. According to this theory, there are vacancies or holes in a liquid. The liquid molecules keep on moving continuously into these vacancies.

As a consequence, the vacancies also keep on moving around as otherwise the liquid will not be able to flow. This process however, requires energy. A liquid molecule, therefore, needs some energy to move into hole. As the energy becomes increasingly available at increasing temperature, a liquid can flow more easily at higher temperature. The coefficient of viscosity thus falls appreciably with rise in temperature as presented in Table 7 to 11.

The relationship between coefficient of viscosity of a liquid and temperature is expressed mathematically as –

$$\eta = Ae^{-\Delta G/RT} \quad (4)$$

The graphs are plotted between log η and 1/T and are presented in Fig 5 to 9. The graph for each system gives linear straight line showing the validity of equation.

The thermodynamic parameters were calculated by using following expressions-

$$\Delta G = -2.303R \times \text{slope} \quad (5)$$

$$\log\eta_{r1} - \log\eta_{r2} = [\Delta H/2.303]\,[1/T1 - 1/T2] \quad (6)$$

&

$$\Delta S = (\Delta G - \Delta H) / T \quad (7)$$

These thermodynamic parameters for different systems are presented in Table 12.

Table 7: System - Ligand L_1

Temp (°K)	1/ T x 10^{-3}	Density x 10^{-3} kgm^{-3}	Time Flow (Secs)	Relative Viscosity η_r
313.5	3.1897	0.9429	34.6	1.5408
323.5	3.0911	0.9415	32.0	1.4229
333.5	2.9985	0.9385	31.0	1.3740
343.5	2.9112	0.9335	29.0	1.2785

Table 8: System - Ligand L_2

Temp (°K)	1/ T x 10^{-3}	Density x 10^{-3} kgm^{-3}	Time Flow (Secs)	Relative Viscosity η_r
313.5	3.1897	0.9435	32.0	1.4529
323.5	3.0911	0.9402	31.3	1.3899
333.5	2.9985	0.9362	29.3	1.2955
343.5	2.9112	0.9311	28.0	1.2313

Table 9: System - Ligand L_4

Temp (°K)	1/ T x 10^{-3}	Density x 10^{-3} kgm^{-3}	Time Flow (Secs)	Relative Viscosity η_r
313.5	3.1897	0.9498	33.0	1.4803
323.5	3.0911	0.9484	31.0	1.3885
333.5	2.9985	0.9449	30.0	1.3388
343.5	2.9112	0.9415	29.0	1.2895

Table 10: System - Ligand L_5

Temp (°K)	1/ T x 10^{-3}	Density x 10^{-3} kgm^{-3}	Time Flow (Secs)	Relative Viscosity η_r
313.5	3.1897	0..9761	36.0	1.6596
323.5	3.0911	0.9668	34.0	1.5525
333.5	2.9985	0.9551	32.0	1.4435
343.5	2.9112	0.9459	30.3	1.3550

Table 11: System - Ligand L_6

Temp (°K)	1/ T x 10^{-3}	Density x 10^{-3} kgm^{-3}	Time Flow (Secs)	Relative Viscosity η_r
313.5	3.1897	0.9445	33.0	1.4721
323.5	3.0911	0.9420	31.6	1.4059
333.5	2.9985	0.9395	30.6	1.3578
343.5	2.9112	0.9350	29.3	1.2939

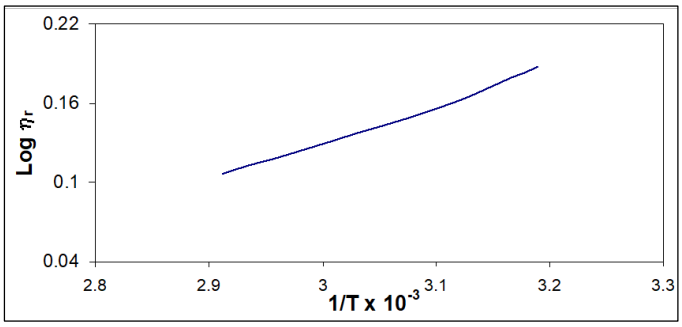

Fig 5
Plot of log η_r Vs 1/ T
System – L_1

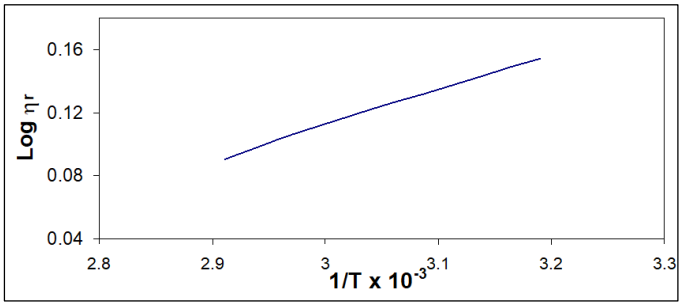

Fig 6
Plot of log η_r Vs 1/ T
System – L_2

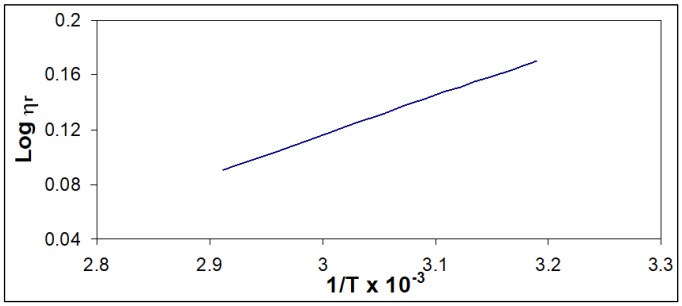

Fig 7:
Plot of log η_r Vs 1/ T
System – L_4

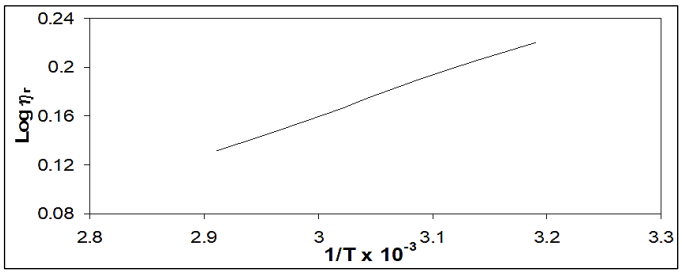

Fig 8
Plot of log η_r Vs 1/ T
System – L_5

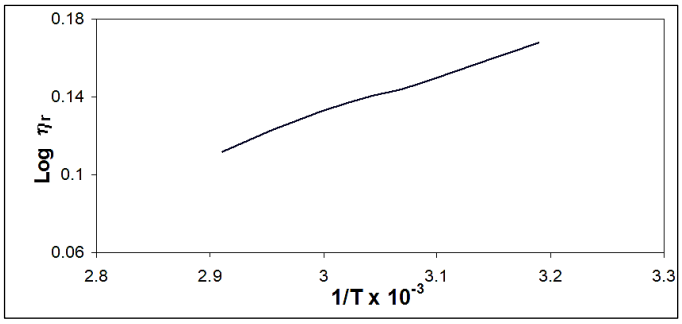

Fig 9
Plot of log η_r Vs 1/ T
System – L_6

Table 12: Values of Thermodynamic Parameters

System	ΔG (J mole^{-1} K^{-1})	ΔH (J mole^{-1} K^{-1})	ΔS (J mole^{-1} K^{-1})
L_1	-5491.7700	808.1521	-20.0951
L_2	-4351.9794	583.0045	-14.7995
L_4	-5506.8833	649.3245	-19.6370
L_5	-6101.8100	677.3529	-21.6241
L_6	-3773.6765	375.5431	-12.8260

The positive and negative values of entropy indicate that the reaction may be exothermic and endothermic respectively. Burghate *et al* [22] have also studied thermodynamic parameters of chalcones in 70% dioxane-water mixtures. Many workers [5-6] have also studied viscosity at different temperatures.

Conclusion

The viscosity studies of 4,5-Dihydro substituted oxazoles in DMF–water mixtures have provided valuable insight into the solute–solvent and solute–solute interactions in mixed solvent systems. The results indicate that viscosity decreases with increasing DMF content, which can be attributed to the reduction in hydrogen bonding and cohesive forces within the solvent medium. The analysis of the data using the Jones–Dole equation yielded positive B-coefficient values, suggesting strong solvation of the substituted oxazoles and the structure-making tendency of the solutes in the mixed solvent. It is evident that the viscosity behavior is influenced by both the nature of the substituent on the oxazole ring and the composition of the solvent mixture. The observed trends demonstrate that solvent polarity and hydrogen-bonding capability play a crucial role in determining molecular interactions and solution dynamics. Overall, the study confirms that viscosity measurements serve as an effective and simple technique for evaluating the molecular interactions and solvation behavior of heterocyclic compounds. The findings contribute to a better understanding of the physicochemical properties of substituted oxazoles and provide a foundation for further studies involving their complex formation, reactivity, and biological applications in different solvent environments.

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