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Research Article

THERMOCHEMICAL STUDY OF SOME CHALCONES IN DMF AND CHLOROFORM AT DIFFERENT TEMPERATURES

***Shipra Baluja and Asmita Hirapara**
Physical Chemistry Laboratory, Department of Chemistry, Saurashtra University,
Rajkot-360005, India
**Author for Correspondence: shipra_baluja@rediffmail.com*

ABSTRACT

Ultrasonic velocity, density and viscosity of some synthesized chalcones were measured in N, N-dimethyl formamide and chloroform at different temperatures (298.15 to 318.15 K). From these experimental data, various acoustical parameters such as specific impedance (Z), adiabatic compressibility (κ_s), Rao's molar sound function (R_m), inter molecular free path length (L_f), solvation number (S_n), internal pressure (π) have been calculated. The observed variation in these parameters with concentration highlights ion-solvent and ion-ion interactions present in the studied solutions. It is observed that in both chloroform and DMF, predominance of ion-solvent interactions exist for both the compounds. These interactions are higher in DMF. Further, interactions increases with increase in concentration but decreases with increase in temperature.

Keywords: *Chalcones, acoustical parameters, DMF, chloroform.*

INTRODUCTION

In recent years, the ultrasonic, volumetric and viscometric techniques have gained much importance in the study of solution thermodynamics and molecular interactions in solutions (Mokate and Ddamba, 2006; Kumar *et al.*, 2012; Canzonieri *et al.*, 2012; Mandakmare *et al.*, 2014) The viscosity can provides concerning the nature and kinetic behaviour of entities from which, ion-solvent and the molecular solvent-solute interactions can be inferred (Lofutar *et al.*, 2007). The knowledge of ion-solvent interactions play a vital role for separation of various component in petroleum refinery (Hu *et al.*, 2014), solvent used in paint and emulsion industries (Sollner *et al.*, 1944) crystallization industries (Ruecroft *et al.*, 2005) etc. It also gives information of bonding between molecules and formation of complex at different temperatures through various interactions (Raj *et al.*, 2009) occurring in the solutions. Ultrasonic techniques have also been useful in different fields such as engineering (Knorr *et al.*, 2004), food (Dolatozski *et al.*, 2007), chemical (Abou-Okeil *et al.*, 2010), water treatment (Doosti *et al.*, 2012), medicine (Staples *et al.*, 2010; Nikoonahad and Iravani, 1989), etc. Further, **s**onochemistry is attracting considerable research activity within the synthetic chemistry community, because it offers a new approach to the preparation of organic compounds.

Chalcones are an aromatic ketones, which contain active methylene group that forms the central core for variety of biological activities including antitumor (Kumar *et al.*, 2003), antioxidant activities (Shenvi *et al.*, 2013), anti-inflammatory (Ballesteros *et al.*, 1995), anti-HIV (Wu *et al.*, 2003), antiprotozoal (Salemand *et al.*, 2005), antimitotic (Edwards *et al.*, 1990) and antivirus (Trivedi *et al.*, 2007) properties. It is also important constituents of many products such as paint, food, plastic, explosive, petrochemical, pesticide etc. (Nakaoki *et al.*, 1955; Simpson *et al.*, 1957; Bathory *et al.*, 1973). Due to wide biological and pharmacological applications of chalcones, it is interesting to study acoustical properties of such compounds.

Thus, in the present work, some new chalcones are synthesized and their structure characterization was done by IR, ^{1}H NMR and mass spectral data. The density, ultrasonic velocity and viscosity of these synthesized compounds were measured in N, N-dimethyl formamide and chloroform over a wide range of concentrations at different temperatures. From these experimental data, various acoustical parameters such as specific impedance (Z), adiabatic compressibility (κ_s), Rao's molar sound function (R_m), inter molecular free path length (L_f), solvation number (S_n), internal pressure (π) have been calculated. The obtained results are discussed in term of ion-ion and ion-solvent interactions occurring in the studied solutions.

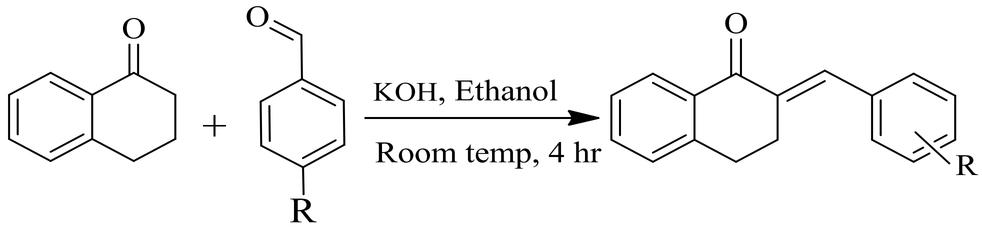
MATERIALS AND METHODS

Materials:

α-tetralone, methanol, 3-bromo benzaldehyde and 4-methoxy benzaldehyde used in the synthesis were supplied from Spectrochem Pvt. Ltd. (Mumbai, India). The solvents, DMF and chloroform used in the present work were of AR grade supplied by LOBA Chemie Pvt. Ltd. (Mumbai, India) and were purified according to the standard reported method (Riddick *et al.*, 1986). The purified solvents were kept over molecular sieves. The purities of solvents were checked by GC-MS (SHIMADZU-Model No.-QP-2010).

Synthesis

Equimolar mixture of α-tetralone and substituted benzaldehydes in ethanol was refluxed for 4 hr using potassium hydroxide as catalyst. The reaction progress was checked by analytical thin layer chromatography (TLC) using (4:1-Hexane: Ethyl acetate) as mobile phase. After completion of reaction, the temperature of reaction mass was allowed cool at room temperature. The resulting solid was filtered, washed with water and was dried under vacuum to give crude product. The obtained crude product was purified by washing with diethyl ether. The reaction scheme is given in Figure 1.



where R is 4-OCH_3 and 3-Br

Figure 1: Reaction Scheme for the synthesis of chalcones

Two compounds were synthesized and their IUPAC names are:
A-1:2-(4-methoxybenzylidine)-3, 4-dihydro napthalene-1, (2H)-one.
A-2:2-(3-bromobenzylidine)-3, 4-dihydro napthalene-1, (2H)-one.

Spectroscopy study

The structure of the synthesized compounds was confirmed by IR, ^{1}H NMR and mass spectral analysis. The IR spectra of compounds were taken on FT-IR (SHIMADZU Model-IR affinity-1S). ^{1}H NMR spectra were taken on a Bruker AVANCE III (400 MHz). For both compounds, ^{1}H NMR spectra were obtained in deuterated dimethyl sulfoxide (DMSO-d_6) using TMS as an internal standard. The NMR signals are reported in δ ppm. Mass spectra were determined using direct inlet probe on a GC-MS (SHIMADZU Model-QP 2010) mass spectrometer.

The melting points of compounds were measured by Different Scanning Calorimeter (SHIMADZU DSC-60) under nitrogen atmosphere.

Preparation of solutions:

The solutions of chalcones were prepared in DMF and chloroform in the concentration range from 0.01 to 0.10 mol. L^{-1} and were stored in bottles with PTFE septum until further use. The standard uncertainties of solutions are 0.0001 mol. L^{-1}. An electronic balance (Mettler Toledo Model-AB204-S) with accuracy of ±0.0001 g was used for weighing.

Measurements of Density and Ultrasound velocity:

The density and ultrasonic velocity of pure solvents and solutions were measured at different temperatures (298.15, 308.15 and 318.15K) using Anton Paar Density and Sound velocity meter (DSA

5000M). The temperature was controlled up to ± 0.01 K by a built-in Peltier device. Before measurements, the instrument DSA 5000M was calibrated by ultra-pure water in the experimental temperature range. The standard uncertainty of the density and ultrasonic velocity was found to be 0.5 kg m^{-3} and 0.5 m s^{-1}, respectively.

Measurement of viscosity

To determine the viscosity of pure solvents and solutions, Ubbelohde viscometer was used. The measured quantity of the distilled water / solvent / solution was placed in the viscometer, which was suspended in a constant temperature bath (NOVA Instruments Pvt. Ltd.-Ahmedabad) (NV-8550 E) at definite temperature. The accuracy of bath was ± 0.5° C. A digital stop watch (Hanhart-Germany) with accuracy of ±0.01 second was used to determine flow time of solutions.

RESULTS AND DISCUSSION

Table 1 shows the substitution groups and other physical properties of synthesized compounds.

Table1: Physical constants of the synthesized compounds

Compound	Mol. Formula	Mol. Wt.	% Yield	R_f^*value	Melting point (K)
A-1	$C_{18}H_{16}O_2$	264.32	80	0.47	382.09
A-2	$C_{18}H_{13}OBr$	113.19	78	0.36	396.65

**4:1-Hexane: Ethyl acetate*

Spectral data

A-1:

IR (cm^{-1}, KBr): 3005.20 (Ar-H asym. str.), 2944.44 (CH_2 stretching of cyclohexanone ring), 1665.50 (C=O stretching), 1252.81-1300.90 ($-CH_2$ bending), 1000.99 (ring stretching in cyclohexanone), 840 (C-H out of plane bending), 1177.58-1137.07 ($-OCH_3$ stretching).

^1H NMR ($CDCl_3$) δ(ppm) : 3.75-3.380 (3H, singlet) 2.80-2.90 (2H, triplet), 3.00-3.10 (2H, triplet), 6.80-6.90 (2H, doublet), 7.00-7.10 (2H, doublet), 7.27-7.28 (1H, doublet), 7.33-7.28 (1H, doublet), 7.30-7.38 (1H, doublet), 7.75-7.80 (1H, singlet), 7.40-7.45 (1H, doublet), 2.80-2.90 (2H, triplet).

MS: *(m/z)* = 264.32

A-2:

IR (cm^{-1}, KBr): 3296.46 (Ar-H asym. stretching), 2902.96-2785.30 (CH_2 stretching of cyclohexanone ring), 1664.62(C=O stretching), 1296.21-1219.05($-CH_2$ bending), 958.66 (ring stretching in cyclohexanone), 742.62 (C-H out of plane bending), 500-600 (C-Br stretching).

^1H NMR ($CDCl_3$) δ(ppm) : 2.80-3.00 (2H, triplet), 3.00-3.10 (2H, triplet), 7.34-7.35 (1H, doublet), 7.38-7.42 (1H, doublet), 7.50-7.55 (1H, doublet), 7.56-7.58 (1H, doublet), 7.58-7.60 (1H, doublet), 7.70-7.68 (1H, doublet), 7.90-8.00 (1H, singlet), 7.70-7.68 (1H, doublet), 7.74-7.70 (1H, doublet).

MS: *(m/z)* = 113.19

Table 2 shows experimental density, ultrasonic velocity and viscosities of pure solvents and solutions of both the compounds. From the experimental data of density, viscosity and ultrasonic velocity, various acoustical parameters such as specific acoustical impedance (Z), adiabatic compressibility (κ_s), intermolecular free path length (L_f), Rao's molar sound function (R_m), free volume (V_f), internal pressure (π), solvation number (S_n) etc. are evaluated using equations reported earlier (Baluja and Karia, 2011). Some of these evaluated parameters are reported in Table 3.

Table 2: Experimental data of density, ultrasonic velocity and viscosity of chalcones in chloroform and DMF at different temperatures

Conc. (M)	*Density Kg/m^3*	*Velocity m/s*	*Viscosity m Pas*	*Density Kg/m^3*	*Velocity m/s*	*Viscosity m Pas*
A-1						
	Chloroform			**DMF**		
298.15 K						
0.00	1479.32	983.90	0.7678	943.94	1457.71	0.8158
0.01	1478.09	985.00	0.7535	945.70	1462.12	0.8435
0.02	1477.33	986.04	0.7555	946.35	1463.08	0.8510
0.04	1475.99	987.91	0.7572	947.80	1464.25	0.8619
0.06	1475.00	989.63	0.7613	948.77	1465.15	0.8715
0.08	1474.14	991.33	0.7632	948.51	1466.48	0.8773
0.10	1473.62	992.82	0.7653	949.16	1468.08	0.8897
308.15 K						
0.00	1460.15	949.94	0.6837	934.38	1418.8	0.7011
0.01	1458.99	951.38	0.6766	936.15	1425.31	0.7261
0.02	1458.10	952.71	0.6784	936.79	1425.89	0.7302
0.04	1457.01	954.02	0.6800	938.26	1426.93	0.7384
0.06	1456.09	955.77	0.6818	939.23	1427.79	0.7417
0.08	1455.31	957.35	0.6836	938.99	1428.61	0.7488
0.10	1454.85	958.89	0.6856	939.64	1429.77	0.7528
318.15 K						
0.00	1440.27	916.11	0.6129	924.77	1380.15	0.6337
0.01	1439.64	917.42	0.6089	926.54	1385.75	0.6461
0.02	1438.71	918.81	0.6110	927.20	1386.77	0.6537
0.04	1437.79	920.26	0.6158	928.67	1389.60	0.6607
0.06	1436.96	921.99	0.6179	929.65	1390.88	0.6648
0.08	1436.23	923.58	0.6196	929.41	1391.22	0.6738
0.10	1435.84	925.16	0.6215	930.08	1392.69	0.6771
A-2						
298.15 K						
0.00	1479.32	983.90	0.7678	943.94	1457.71	0.8158
0.01	1478.99	984.86	0.7564	945.58	1461.27	0.8609
0.02	1478.94	985.85	0.7596	946.43	1462.67	0.8571
0.04	1478.80	987.67	0.7624	947.65	1463.34	0.8552
0.06	1478.73	989.22	0.7661	948.70	1463.94	0.8549
0.08	1478.71	990.72	07694	949.54	1464.95	0.8511
0.10	1478.64	992.34	0.7726	949.77	1465.81	0.8396
308.15 K						
0.00	1460.15	949.94	0.6837	934.38	1418.8	0.7011
0.01	1459.89	950.98	0.6766	936.02	1422.74	0.7496
0.02	1459.86	951.95	0.6796	936.88	1424.11	0.7472
0.04	1459.80	953.77	0.6827	938.98	1424.83	0.7418
0.06	1459.81	955.37	0.6857	939.16	1425.46	0.7391
0.08	1459.83	956.82	0.6892	939.99	1426.26	0.7319
0.10	1459.82	958.44	0.6923	940.224	1427.28	0.7295

318.15 K							
0.00	1440.27	916.11	0.6129	924.77	1380.15	0.6337
0.01	1440.53	917.19	0.6101	926.42	1384.89	0.6745
0.02	1440.54	918.16	0.6122	927.27	1385.99	0.6722
0.04	1440.57	920.01	0.6142	928.50	1387.11	0.6640
0.06	1440.63	921.61	0.6163	929.57	1387.15	0.6613
0.08	1440.71	923.06	0.6183	930.415	1388.46	0.6559
0.10	1440.76	924.84	0.6204	930.641	1389.11	0.6490

Table 3: Some acoustical parameters of chalcones in chloroform and DMF at different temperatures

Conc. (M)	***$Z \cdot 10^{-6}$ Kg m^{-2} s^{-1}***	***$V_f \cdot 10^{12}$ m^3***	***$R_m \cdot 10^4$ $m^{-8/3}s^{-1/3}$***	***$\pi \cdot 10^{-10}$ Pa***	***$Z \cdot 10^{-6}$ Kg m^{-2} s^{-1}***	***$V_f \cdot 10^{12}$ m^3***	***$R_m \cdot 10^4$ $m^{-8/3}s^{-1/3}$***	***$\pi \cdot 10^{-10}$ Pa***
A-1								
Chloroform					**DMF**			
298.15K								
0.00	1.4555	6.7576	8.0260	1.4042	1.3769	5.3299	8.7795	1.5623
0.01	1.4559	6.9978	8.0637	1.3840	1.3827	5.1430	8.8293	1.5761
0.02	1.4567	7.0176	8.0984	1.3791	1.3846	5.1297	8.8824	1.5714
0.04	1.4581	7.0867	8.1664	1.3675	1.3880	5.1369	8.9855	1.5589
0.06	1.4597	7.1189	8.2323	1.3587	1.3901	5.1519	9.0914	1.5455
0.08	1.4614	7.1830	8.2976	1.3480	1.3910	5.2043	9.2107	1.5273
0.10	1.4630	7.2424	8.3605	1.3380	1.3935	5.1993	9.3213	1.5159
308.15K								
0.00	1.3871	7.6293	8.0370	1.3817	1.3257	6.4239	8.7897	1.5069
0.01	1.3881	7.8081	8.0755	1.3672	1.3343	6.1987	8.8445	1.5203
0.02	1.3891	7.8345	8.1124	1.3619	1.3358	6.2101	8.8974	1.5135
0.04	1.3900	7.9032	8.1785	1.3511	1.3388	6.2332	9.0009	1.5002
0.06	1.3917	7.9761	8.2451	1.3402	1.3410	6.3164	9.1084	1.4820
0.08	1.3932	8.0461	8.3107	1.3298	1.3415	6.3505	9.2279	1.4668
0.10	1.3950	8.1129	8.3744	1.3198	1.3435	6.4271	9.3389	1.4496
318.15K								
0.00	1.3195	8.5137	8.0501	1.3628	1.2763	7.1725	8.7996	1.4895
0.01	1.3208	8.6600	8.0859	1.3515	1.2840	7.0792	8.8533	1.4913
0.02	1.3219	8.6821	8.1238	1.3466	1.2858	7.0345	8.9077	1.4886
0.04	1.3231	8.6905	8.1904	1.3394	1.2905	7.0804	9.0163	1.4741
0.06	1.3249	8.7623	8.2576	1.3290	1.2931	7.1613	9.1259	1.4570
0.08	1.3265	8.8399	8.3239	1.3186	1.2930	7.1561	9.2457	1.4448
0.10	1.3284	8.9144	8.3884	1.3086	1.2950	7.2488	9.3585	1.4274
A-2								
298.15 K								
0.00	1.4555	6.7576	8.0264	1.4042	1.3760	5.3299	8.7795	1.5623
0.01	1.4566	6.9573	8.0584	1.3873	1.3817	4.9843	8.8288	1.5925
0.02	1.4580	6.9586	8.0890	1.3839	1.3843	5.0730	8.8808	1.5773
0.04	1.4605	7.0114	8.1500	1.3742	1.3867	5.1919	8.9847	1.5533

0.06	1.4628	7.0479	8.2100	1.3656	1.3888	5.2965	9.0896	1.5312
0.08	1.4649	7.0900	8.2696	1.3568	1.3910	5.4375	9.1970	1.5063
0.10	1.4673	7.1335	8.3299	1.3479	1.3922	5.6583	9.3101	1.4745
308.15 K								
0.00	1.3871	7.6292	8.0371	1.3817	1.3257	6.4239	8.7898	1.5069
0.01	1.3883	7.8036	8.0695	1.3680	1.3317	5.8902	8.8404	1.5462
0.02	1.3897	7.8033	8.1004	1.3649	1.3342	5.9889	8.8929	1.5319
0.04	1.3923	7.8543	8.1619	1.3556	1.3379	6.1767	8.9900	1.5056
0.06	1.3947	7.9020	8.2226	1.3467	1.3387	6.3343	9.1042	1.4805
0.08	1.3968	7.9401	8.2827	1.3385	1.3407	6.5559	9.2125	1.4524
0.10	1.3991	7.9880	8.3437	1.3298	1.3420	6.7186	9.3273	1.4289
318.15 K								
0.00	1.3194	8.5137	8.0501	1.3628	1.2763	7.1725	8.7996	1.4895
0.01	1.3212	8.6316	8.0803	1.3536	1.2829	6.6317	8.8527	1.5239
0.02	1.3226	8.6477	8.1115	1.3496	1.2852	6.7392	8.9053	1.5101
0.04	1.3253	8.7216	8.1737	1.3395	1.2879	7.0092	9.0126	1.4789
0.06	1.3277	8.7917	8.2350	1.3298	1.2895	7.1885	9.1185	1.4551
0.08	1.3299	8.8594	8.2958	1.3204	1.2918	7.4278	9.2291	1.4281
0.10	1.3325	8.9322	8.3578	1.3108	1.2928	7.6951	9.3445	1.3998

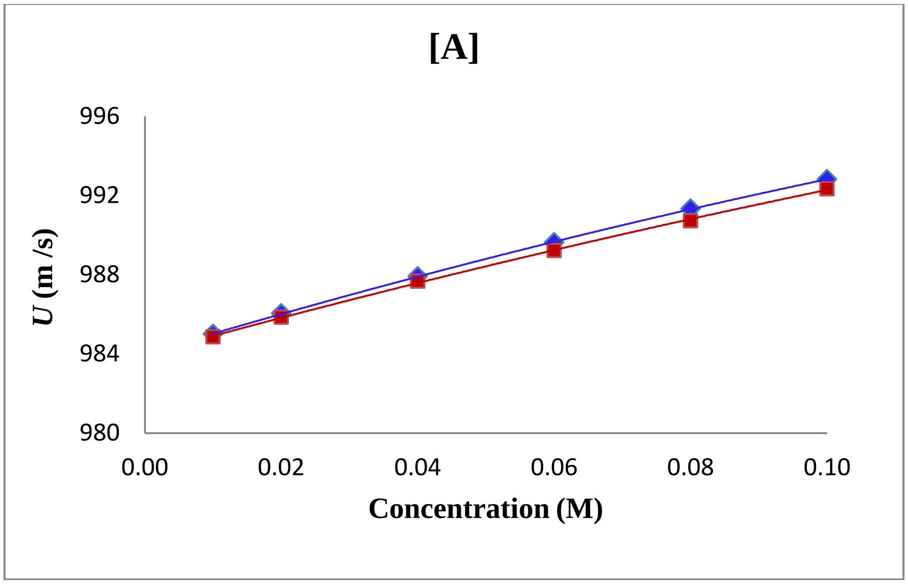
Figure 2 shows the variation of ultrasonic velocity with concentration for the studied compounds in both the solvents. It is observed from Figure 2 that the velocity increases almost linearly with concentration for both the compounds in both chloroform and N, N-dimethylformamide. Further, ultrasonic velocity is higher in DMF than that in chloroform. As temperature increases, ultrasonic velocity is found to decrease in both the solvents. The velocity is related to intermolecular free path length (L_f). Figure 3 shows that intermolecular free path length (L_f) decreases with concentration in both the solvents for both compounds. Thus, velocity is reciprocal of intermolecular free path length. As the name suggests, intermolecular free path length is a measure of distance between solute (compound) and solvent molecules. The decrease of L_f suggests that distance between compound and solvent molecules decrease which means compound and solvent molecules interact with each other i.e., compound-solvent interactions takes place in solution. This decrease in distance between compound and solvent molecules causes an increase in velocity.

As temperature increases, solution is disturbed due to increase in agitation i.e., increase in molecular motion. This causes decrease in compound-solvent interaction so ultrasonic velocity decrease with temperature.

The interaction depends on the nature of solvent and structure of compounds. In DMF, these interactions are higher than that in chloroform. Table 1 shows that compound A-1 contains 3-bromo group whereas 4-methoxy group is present in A-2. In both the solvents, interaction is higher in A-1 solutions. This suggests that although compound-solvent interactions predominant in both the solutions, 3-bromo group which is electron withdrawing cause's strong ion-solvent interactions than 4-methoxy group which is electron donating group.

The presence of compound-solvent interactions in studied solutions is further supported by adiabatic compressibility. Figure 4 shows that the variation of adiabatic compressibility (κ_s) with concentration which is found to decrease with concentration for both compounds in both the solvents. The compressibility of the solution is mainly due to free solvent molecules around compound molecules. So, when there is strong interaction between solvent and compound molecules, compressibility decreases which is the case in present study. By increase in concentration of solute (compound), molecular associations are enhanced and newly formed aggregates cause adiabatic compressibility to decrease. Furthermore, κ_s also increases with increase in temperature.

The predominance of ion-solvent interaction is further proved by increase of specific impedance (*Z*) (Table 3). Similar behaviour was also observed by Aswale *et al.*, (2013). The specific impedance also decreases with temperature due to decrease in ion-solvent interactions, which is expected.



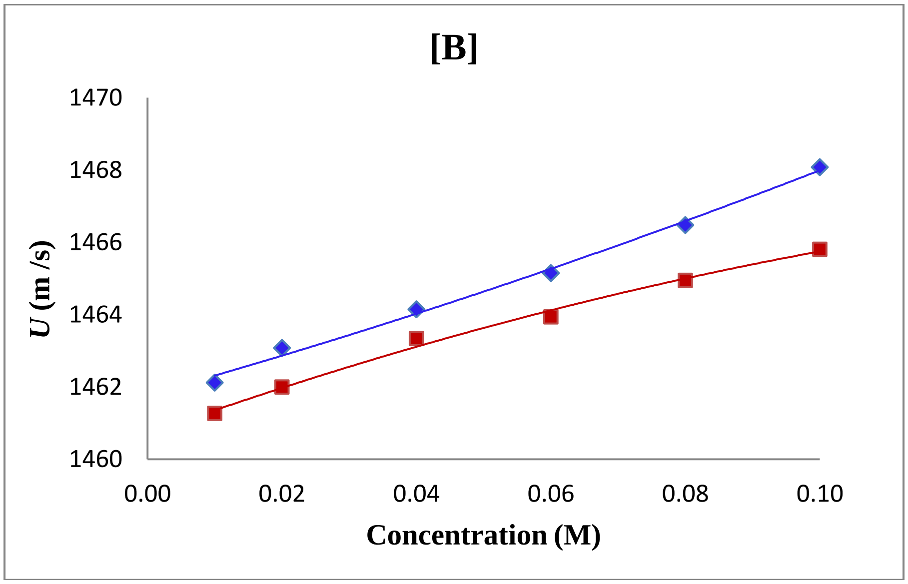
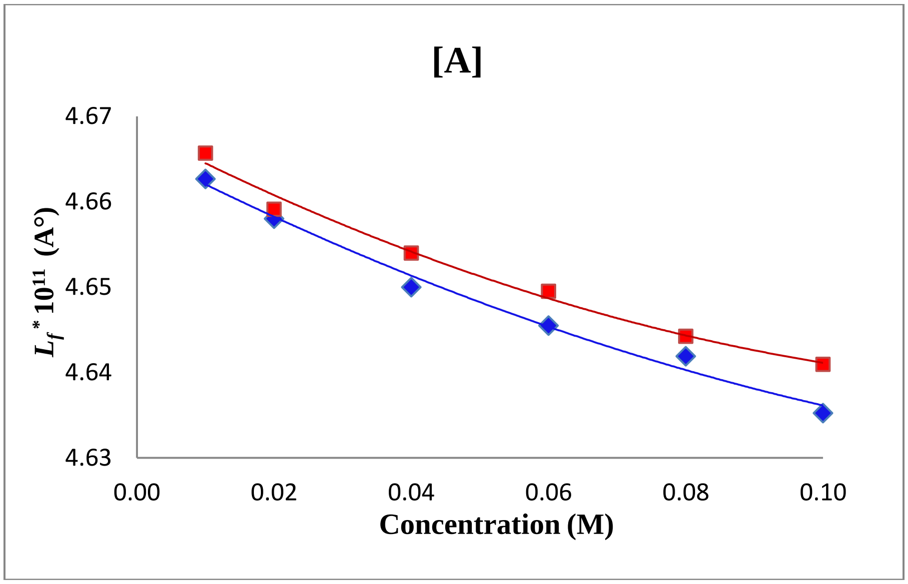


Figure 2B: The variation of ultrasonic velocity (*U*) with concentration for the studied compounds in [A] chloroform and [B] DMF at 298.15 K.
A-1, (♦); A-2, (■)



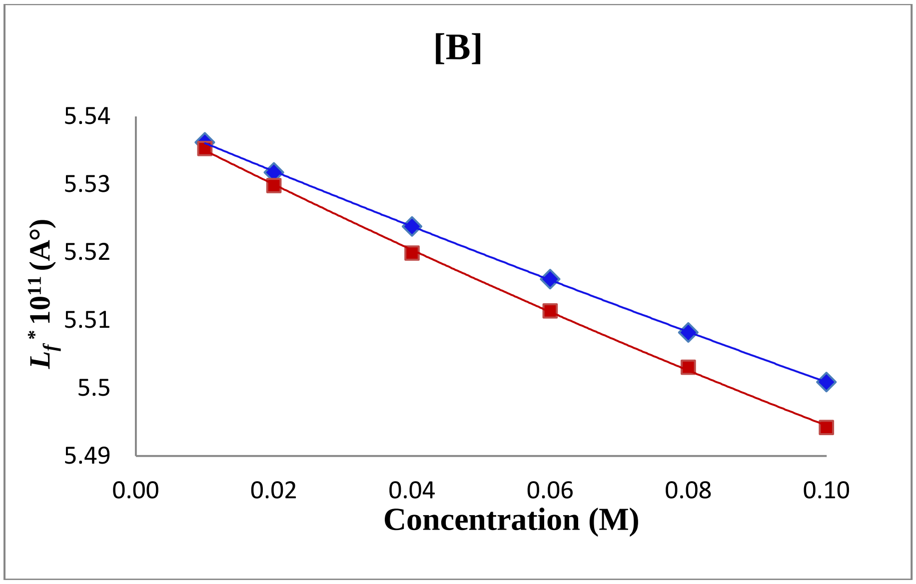
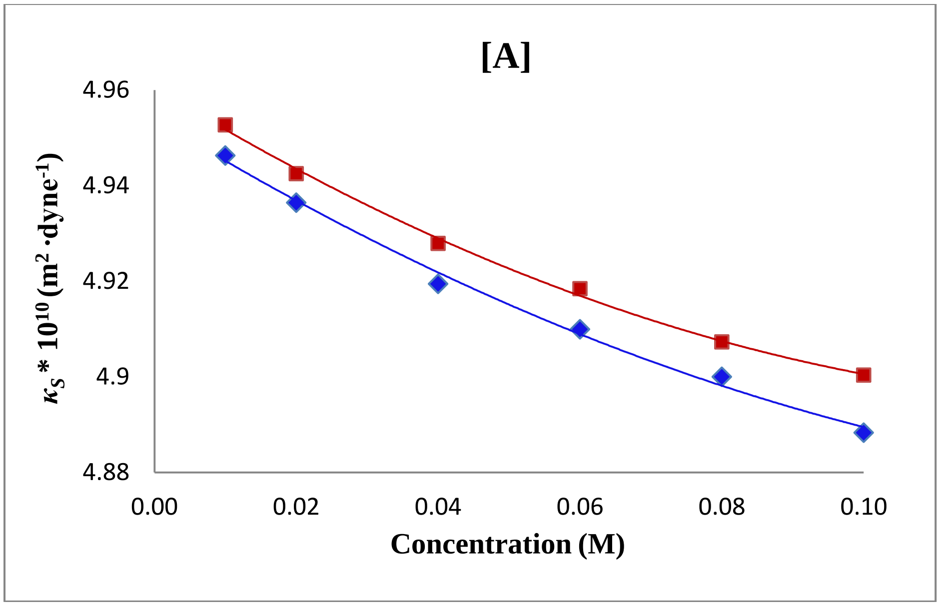


Figure 3: The variation of free path length (L_f) with concentration of the studied compounds in [A] chloroform and [B] DMF at 298.15 K.
A-1, (♦); A-2, (■)



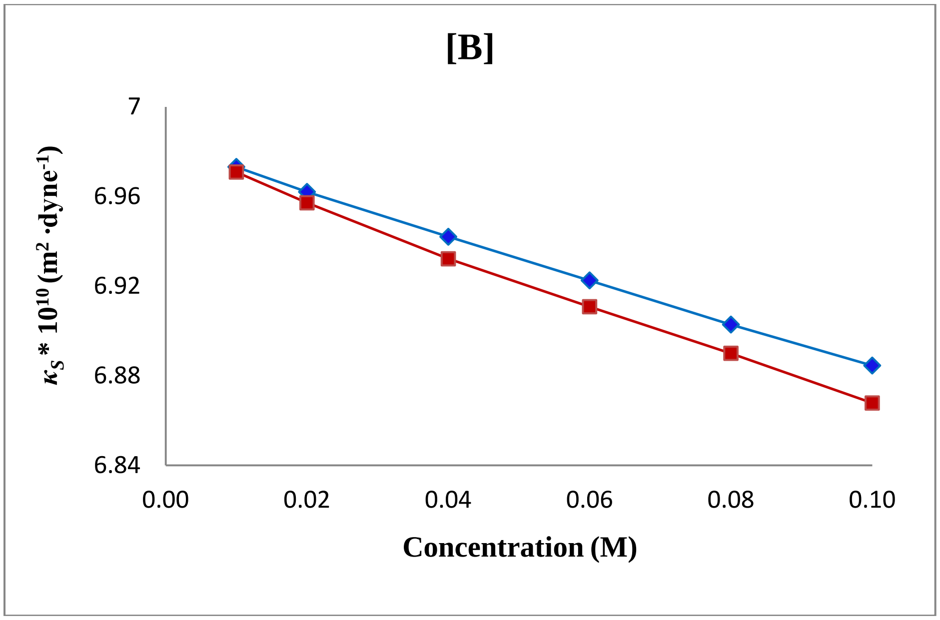
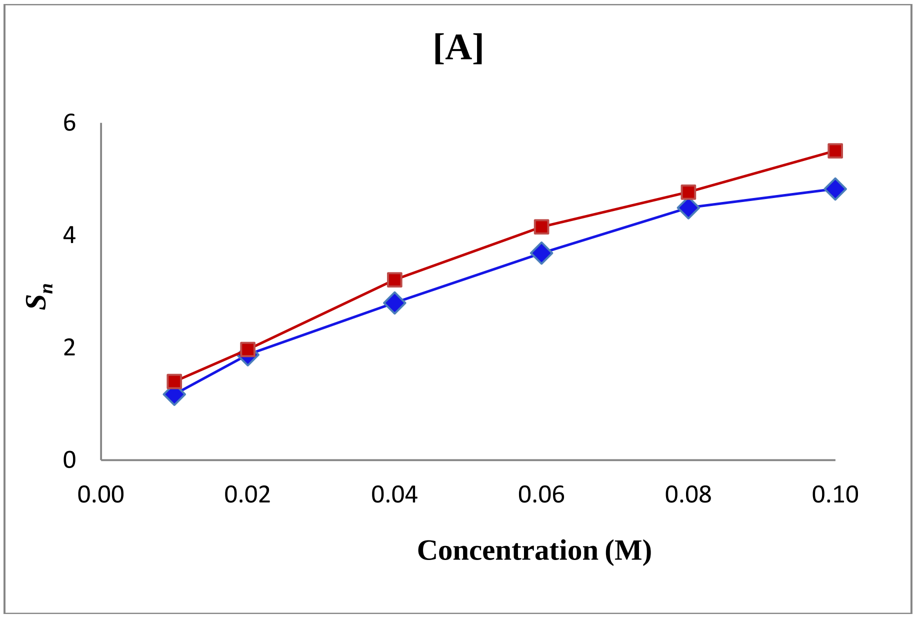


Figure 4: The variation of adiabatic compressibility (κ_s) with concentration for studied compound in [A] Chloroform and [B] DMF at 298.15 K.
A-1, (♦); A-2, (■)



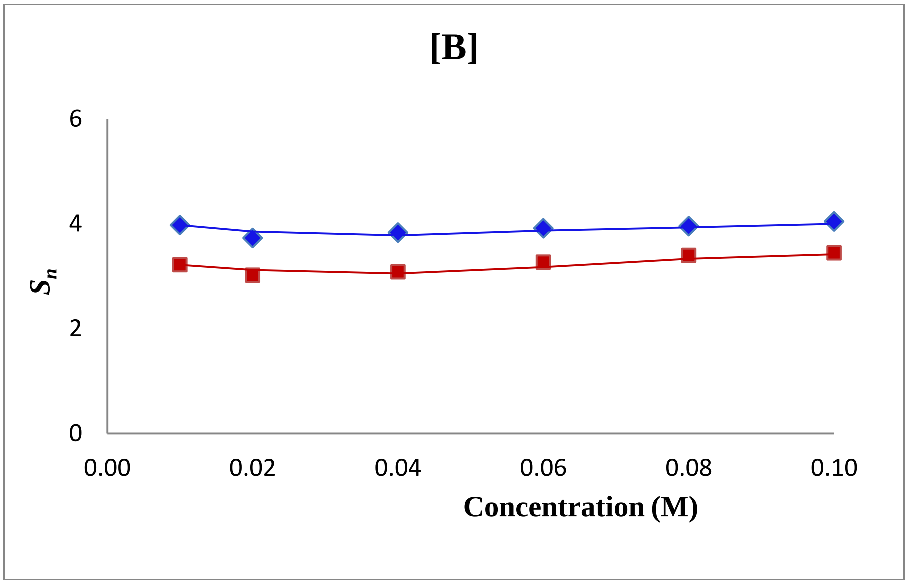


Figure 5: The variation of solvation number (S_n) with concentration for the studied compounds in [A] Chloroform and [B] DMF at 298.15 K.
A-1, (♦); A-2, (■)

It is observed from the Table 3 that the Rao's molar function increase linearly with concentration indicating thereby that there is no complex formation in the studied solutions. The internal pressure (π) is a measure of cohesive energy in the solution which is found to decrease. This suggests that in the studied systems, ion-ion interactions also exist. This is again suggested by increase in free volume which is found to increase. However, the decrease of internal pressure and increase in free volume is in very small magnitude. Thus, in the studied systems, both ion- solvent and ion-ion interactions are present but their magnitudes are much different.

The interactions in a solution can be confirmed by the solvation number (S_n), which is a measure of structure forming or structure breaking tendency of a solute in a solution. Figure 5 shows that for both the compounds, solvation number (S_n) nonlinearly increases with concentration. Further, solvation numbers are positive for both the compounds in both the solvents. The positive S_n values suggest structure forming tendency of compounds in solution. This further confirms the predominance of strong ion-solvent interactions in studied solutions of both the compounds the variation in solvation number is more pronounced in chloroform than that in DMF solutions for both the compounds. In DMF, there is slight change in salvation number and at higher concentrations, it is almost constant.

Conclusion

On the bases of the evaluated data, it is concluded that in both chloroform and DMF, predominance of ion-solvent interactions exist for both the compounds. Further, interactions increase with increase in concentration but decrease with increase in temperature. The interaction depends on the nature of solvent and structure of compounds. In DMF, these interactions are higher than that in chloroform. The electron withdrawing bromo group exhibits strong ion-solvent interactions than electron donating 4-methoxy group.

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