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STUDY OF MOLECULAR INTERACTIONS OF COUMARAN-3-ONES IN POLAR AND NON POLAR SOLVENTS USING ULTRASONIC INTERFEROMETER

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ABSTRACT

The molecular interaction between solute-solute and solute-solvent was studied with the help of acoustic properties determined by ultrasonic interferometer at 303.15 K in polar acetone and non polar dioxane solvents. The effect of introduction of metal ions viz Cu(II), Fe(III) in the same solution was found out. Also the effect of position of same group at ortho or para position on the molecular interaction was simultaneously examined.

Key words: Ultrasonic interferometry, Acoustic.

INTRODUCTION

To study the liquid theory the acoustic properties as ultrasonic velocity, adiabatic compressibility are very useful. The most precise property of sound wave is that it gives information of adiabatic properties of solution. Now-a-days molecular interactions are mostly studied by ultrasonic interferometer.[1-3] It is also useful in understanding physico-chemical behavior of liquid. The structural arrangements are influenced by the shape of the molecules as well as by their mutual interactions.

In view of the medicinal, agricultural, pharmaceutical significance of coumaran-3-ones[4-7] we have synthesized some new coumaran-3-ones. In continuation of our work[8-10] we intended to investigate the solute-solute, solute-solvent molecular interactions of these newly synthesized coumaran-3-ones in the polar acetone and non polar dioxane solvents. An attempt in this investigation is made to study the effect of substituent at ortho and para positions on the acoustic properties. The changes due to addition of Cu(II) or Fe(III) metal ion solutions were also noted. The acoustic properties like adiabatic compressibility (β), apparent molar compressibility (ϕK), acoustic impedance (Z), relative association (R_A), intermolecular free length (L_f) etc. are determined from the ultrasonic velocity and density measurements of the solutions at 303.15 K. From the observations of these properties the molecular interactions are predicted.

EXPERIMENTAL

All the chemicals used were of AR grade. 2-(2'-hydroxy)benzylidene-5-methylcoumaran-3-one (1), 2-(4'-hydroxy)benzylidene-5-methylcoumaran-3-one (2), 2-(2'-hydroxy)benzylidene-5-methyl-7-bromocoumaran-3-one (3), 2-(4'-hydroxy)benzyli-dene-5-methyl-7-bromocoumaran-3-one (4), 2-(2'-nitro)benzylidene-5-methylcoumaran-3-one (5), 2-(4'-nitro)benzylidene-5-methylcoumaran-3-one (6), 2-(2'- nitro)benzylidene-5-methyl-7-bromocoumaran-3-one (7), 2-(4'-nitro)benzylidene-5-methyl-7-bromocouma-ran-3-one (8) were synthesized in our laboratory[11] (Scheme-1). These compounds are recrystallized from acetone and water mixture. The solvents (1,4-dioxane and acetone) were purified by standard procedure.[12] The double distilled water was used for preparation of metal solutions of Cu(II) and Fe(III). Densities were measured with the help of bicapillary pyknometer. The pyknometer was calibrated at 303.15K with double distilled water and gave an estimated reproducibility of ±0.0001 gcm^{-3}. 0.01 M solutions of Cu(II) and Fe(III) in distilled water and 0.01 M solutions of compounds in acetone and dioxane solvents were prepared separately. Weighing was done on Roy CCB-4 Balance, (± 0.001 g). A special thermostatic water bath arrangement was made for density and ultrasonic velocity measurements, in which continuous stirring of water was carried out with the help of electric stirrer and temperature variation was maintained within ± 0.1°C. Single crystal interferometer (Mittal Enterprises, Model F-81) with accuracy of ± 0.03% and frequency 2 MHz was used in the present work. The working of the ultrasonic interferometer was checked by measuring ultrasonic velocity of pure water at 303.15 K. The measured value 1505 ms^{-1} is in good agreement with literature[13] value 1515 ms^{-1}. The densities and ultrasonic velocities of all compounds in acetone solvent were measured at 303.15 K. Then 10 ml of each compound solution in acetone was mixed with 2 ml metal ion [Cu(II)/Fe(III)] solution in water and densities, ultrasonic velocities of these solutions also were measured. Similar measurements were carried out in dioxane solvent also.

RESULTS AND DISCUSSION

In the present investigation, measurements of densities and ultrasonic velocities of compounds in solvent and compounds and metal ions in solvent have been made, and given in Table 1-6. The adiabatic compressibility (β) is evaluated by using equation.

$$\beta = 1 / v^2 . d \qquad (1)$$

Apparent molar compressibility (ϕK) is an acoustic property which is determined by measuring density and ultrasonic velocity and depends upon the molality of solution and molecular weight of the solute, by the relation,

$$\phi K = [1000 (\beta_s d_0 - \beta_0 d_s) / m\, d_s d_0] + (\beta_s M / d_s) \qquad (2)$$

where, d_0 = density of pure solvent, d_s = density of solution, m = molality of solution, M = molecular weight of solute, β_0 = adiabatic compressibility of pure solvent, and β_s = adiabatic compressibility of solution.

Specific acoustic impedance is determined from the measurement of ultrasonic velocity and density by formula,

$$Z = v_s . d_s \qquad (3)$$

The solute-solvent interactions may be interpreted in terms of acoustic impedance.

Relative association is a function of ultrasonic velocity and is computed by the equation,

$$R_A = d_s / d_0 [v_0 / v_s]^{1/3} \qquad (4)$$

where, v_0 and v_s are ultrasonic velocities in solvent and solution.

Intermolecular free length (L_f) is one of the important acoustic properties to study the intermolecular interactions.

According to Hildebrand[14] the center of attraction does not coincide with geometrical center of molecule of liquid as the distance between the centers of attraction is a property extremely difficult to define. The distance between the surfaces of molecules, on the other hand has a clear physical significance so it is used in defining free length.

Intermolecular free length has been evaluated from adiabatic compressibility (β) by Jacobson's[15-17] formula,

$$L_f = K . \sqrt{\beta_s} \qquad (5)$$

where, K is the temperature dependent constant known as Jacobson's constant and is independent of the nature of liquid. (at 303.15 K, K=631)

By comparing the values of β in acetone (Table-1) and dioxane (Table-2) it is clearly observed that the β values in dioxane medium are considerably and notably smaller than in acetone medium. This may be due to nature of solvents. The parameters of solvents, which directly affect the values of β, are non-polarity, low dielectric constant (2.2) and higher density of dioxane as compared with polarity, high dielectric constant (20.7) and lower density of acetone.

Similar trends (Table 3-6) and patterns (Fig. 1) of β values are seen after the addition of metal ion solutions [Cu(II) and Fe(III) in water] to each of the above solution. But the adiabatic compressibilities are lower for Cu(II) and lowest for Fe(III) as compared with compounds in both the solvents.

Thus it may be generalized that the β values for all compounds (1 to 8) in acetone solvent are higher than those in dioxane solvent and in both the solvents the values of compounds in pure solvent are higher than after addition of Cu(II) solution [Cu(II)-Comp-solvent] and lowest in case of Fe(III) solution [Fe(III)-Comp-solvent].

Comp-acetone > Comp-dioxane and
Comp-solvent > Cu(II)-Comp -solvent > Fe(III)-Comp-solvent

With solute in acetone, the combination forms polar-polar molecules and as such the interaction between solute and solvent is a polar-polar interaction with resulting compressibility which is of higher value.

As against this when the solvent contains Cu(II) and solute both solute being polar, the resulting interaction between polar solutes and polar solvent is bound to be an enhanced one, resulting in decreased compressibility as can be seen from Fig. 1. Further in case of Fe(III)-Comp, the Fe(III) is more polar the resulting combination with increased polarity is bound to reflect in greater polar-polar interaction, giving rise to a further decrement in compressibility. These physical occurrences of solute-solvent interactions are reflected in the decrease in compressibility.

The lower β values in case of Fe(III)-Comp-solvent than Cu(II)-Comp-solvent may also be interpreted in terms of smaller size and high charge by radius ratio of Fe(III) than Cu(II). Because of the smaller size and polar-polar interaction due to high positive charge on Fe(III) close packing will occur and compressibility will decrease. It may also be interpreted in terms of electrostriction.[18] When the compound is added to acetone electrostriction increases, since the compound is polar and solvent is also polar. Consequently the adiabatic compressibility will decrease.

Acetone is a polar solvent and when polar solutes are added the association of solute and solvent molecules occurs resulting in close packing and clinging of molecules. Therefore the solution becomes less compressible and thereby decreasing the β values.

On the other hand dioxane is a non-polar solvent having higher density. Therefore, already packing of dioxane molecules occurs in the structure and hence it becomes less compressible (Table-2). After the addition of polar solute the association, in other words, packing or clinging of molecules increases, resulting in less compressible nature of solution. Further addition of metal ions still increases the polar-polar interactions, thereby increasing the compactness of solution resulting in further decrease in compressibility, as is clearly indicated by inspection of Table 2 and Fig. 1 where the β values in dioxane solvent are considerably lower and go on decreasing with the addition of Cu(II) and Fe(III) solutions.

It is observed from Fig. 1 that when –OH group is attached at ortho position (R^2), β values are lower as compared to that at para position (R^3). But in case of $–NO_2$ group reverse trend is observed.

Like adiabatic compressibility (β) apparent molar compressibility (ϕK) is another important acoustic parameter, which explains the solute-solvent and solute-solute interactions in solutions. It not only depends upon density and ultrasonic velocity like β but also the molecular weight of solute and molarity of solution. Thus, the structure of solute will have direct effect on ϕK values.

From the Table 1-6 and Fig. 2, the general trend for apparent molar compressibilities is seen as - M-Comp-acetone < M-Comp-dioxane and Comp-solvent > Cu(II)-Comp-solvent > Fe(III)-Comp-solvent, where, M = Metal ion.

The variation in trend of ϕK values after the addition of metal ions in both the solvents is in agreement with the β values for the same solutions. This agreement is supportive of the observations predicted in case of adiabatic compressibility βs and as such the same argument can be valid in this case of apparent molar compressibility also.

The ϕK values for the compounds in acetone and compound + metal ion in acetone solutions are lower than those in dioxane solvent. This appears to be the reverse trend than which is observed for βs. Here the difference between the two properties can be clearly seen.

Table-1 : Acoustic parameters for compounds in acetone at 303.15 K

Compd. No.	v	d	β x 10^{-10}	ϕK x 10^{-9}	Z x 10^{4}	R_A	L_f
	(m sec^{-1})	(kg m^{-3})	(pa^{-1})	(m^{3}mol^{-1}pa^{-1})	(kg m^{-2}sec^{-1})		(A^0)
Acetone	1147.91	784.50	09.6736	--	--	--	--
1	1140.73	779.10	09.8637	03.6144	88.8742	0.9952	0.0198
2	1107.04	780.50	10.4544	10.9739	86.4045	1.0070	0.0204
3	1118.43	779.60	10.2545	08.6614	87.1925	1.0024	0.0202
4	1143.46	777.70	09.8344	03.5646	88.9267	0.9926	0.0198
5	1144.74	785.00	09.7211	00.8744	89.8623	1.0016	0.0197
6	1145.38	789.60	09.6537	-00.7052	90.4393	1.0072	0.0196
7	1118.72	788.30	10.1360	05.7353	88.1883	1.0135	0.0201
8	1147.46	785.50	09.6690	00.2277	90.1327	1.0014	0.0196

Table-2 : Acoustic parameters for compounds in dioxane at 303.15 K

Compd. No.	v	d	β x 10^{-10}	ϕK x 10^{-9}	Z x 10^{4}	R_A	L_f
	(m sec^{-1})	(kg m^{-3})	(pa^{-1})	(m^{3}mol^{-1}pa^{-1})	(kg m^{-2}sec^{-1})		(A^0)
Dioxane	1366.27	1022.42	5.2396	--	--	--	--
1	1432.48	1021.60	4.7703	-4.4370	146.3422	0.9836	0.0138
2	1455.13	1020.70	4.6270	-5.8023	148.5248	0.9776	0.0136
3	1322.76	1030.00	5.5488	2.8024	136.2440	1.0184	0.0149
4	1267.29	1022.10	6.0919	8.5508	129.5301	1.0251	0.0156
5	1309.90	1024.40	5.6893	4.4453	134.1860	1.0161	0.0151
6	1291.64	1040.40	5.7612	4.2824	134.3825	1.0368	0.0151
7	1293.08	1022.50	5.8490	6.1609	132.2177	1.0186	0.0153
8	1291.51	1040.80	5.7602	4.2946	134.4208	1.0373	0.0151

Table-3: Acoustic parameters for Cu(II)-compounds in acetone at 303.15 K

Compd. No.	v	d	β x 10^{-10}	ϕK x 10^{-8}	Z x 10^{4}	R_A	L_f
	(m sec^{-1})	(kg m^{-3})	(pa^{-1})	(m^{3}mol^{-1}pa^{-1})	(kg m^{-2}sec^{-1})		(A^0)
1	1239.46	838.90	7.7593	-3.0582	103.9785	1.0423	0.0176
2	1229.47	837.10	7.9029	-2.8663	102.9190	1.0429	0.0177
3	1323.35	835.20	6.8369	-4.1179	110.5265	1.0153	0.0165
4	1263.48	836.60	7.4876	-3.3512	105.7030	1.0329	0.0173
5	1234.61	842.10	7.7907	-3.0534	103.9668	1.0477	0.0176
6	1256.95	831.90	7.6084	-3.1594	104.5656	1.0288	0.0174
7	1260.97	842.60	7.4639	-3.4408	106.2495	1.0410	0.0172
8	1294.61	845.70	7.0552	-3.9585	109.4851	1.0357	0.0168

Table-4: Acoustic parameters for Cu(II)-compounds in dioxane at 303.15 K

Compd. No.	v	d	β x 10^{-10}	ϕK x 10^{-9}	Z x 10^{4}	R_A	L_f
	(m sec^{-1})	(kg m^{-3})	(pa^{-1})	(m^{3}mol^{-1}pa^{-1})	(kg m^{-2}sec^{-1})		(A^0)
1	1383.01	1034.50	5.0538	-2.2725	143.0725	1.0077	0.0142
2	1382.55	1029.90	5.0797	-1.8013	142.3891	1.0034	0.0142
3	1400.63	1028.50	4.9562	-2.8999	144.0545	0.9977	0.0140
4	1399.79	1032.70	4.9420	-3.2348	144.5560	1.0019	0.0140
5	1381.92	1024.80	5.1097	-1.2477	141.6192	0.9985	0.0143
6	1466.92	1040.40	4.4667	-8.1951	152.6184	0.9938	0.0133
7	1431.67	1029.40	4.7395	-5.0412	147.3758	0.9913	0.0137
8	1401.06	1040.80	4.8946	-4.0514	145.8223	1.0095	0.0140

Table -5: Acoustic parameters for Fe(III)-compounds in acetone at 303.15 K

Compd. No.	v	d	$\beta \times 10^{-10}$	$\phi K \times 10^{-8}$	$Z \times 10^{4}$	R_A	L_f
	$(m\ sec^{-1})$	$(kg\ m^{-3})$	(pa^{-1})	$(m^3 mol^{-1} pa^{-1})$	$(kg\ m^{-2} sec^{-1})$		(A^0)
1	1268.36	849.00	7.3216	-3.6853	107.6838	1.0468	0.0171
2	1267.51	847.20	7.3471	-3.6369	107.3832	1.0448	0.0171
3	1323.29	846.30	6.7479	-4.3311	111.9897	1.0288	0.0164
4	1229.56	849.00	7.7910	-3.1238	104.3894	1.0577	0.0176
5	1266.15	842.70	7.4021	-3.5224	106.6987	1.0397	0.0172
6	1292.93	827.30	7.2308	-3.5661	106.9640	1.0136	0.0170
7	1263.62	852.30	7.3480	-3.6784	107.6987	1.0522	0.0171
8	1291.00	844.80	7.1022	-3.8937	109.0639	1.0355	0.0168

Table -6: Acoustic parameters for Fe(III)-compounds in dioxane at 303.15 K

Comp No.	v	d	$\beta \times 10^{-10}$	$\phi K \times 10^{-9}$	$Z \times 10^{4}$	R_A	L_f
	$(m\ sec^{-1})$	$(kg\ m^{-3})$	(pa^{-1})	$(m^3 mol^{-1} pa^{-1})$	$(kg\ m^{-2} sec^{-1})$		(A^0)
1	1399.93	1026.20	4.9723	-2.6726	143.6605	0.9956	0.0141
2	1453.07	1026.20	4.6153	-6.1607	149.1140	0.9833	0.0136
3	1400.35	1032.20	4.9404	-3.2271	144.5445	1.0013	0.0140
4	1397.99	1030.30	4.9662	-2.8870	144.0353	1.0000	0.0141
5	1419.77	1029.90	4.8169	-4.3459	146.2218	0.9945	0.0138
6	1398.57	1027.00	4.9781	-2.6398	143.6328	0.9967	0.0141
7	1401.53	1029.00	4.9474	-2.9954	144.2178	0.9979	0.0140
8	1433.54	1044.10	4.6606	-6.4504	149.6759	1.0050	0.0136

The adiabatic compressibility may just explain the simple association or close packing or clinging of molecules. But on the contrary, apparent molar compressibility is a property with difference, which may explain the molecular interactions like structure making and structure breaking nature of solute. Apparent molar compressibility property is fairly sensitive to structural changes especially in highly structured solvent and is hence expected to throw interesting light.[19]

An addition of solute in dioxane solvent may produce weak interaction of the Vander Wall forces, which is expected to introduce structurdness in the solution i.e. specific arrangement of dioxane molecule may be occurring due to attached solute molecules. Thus spaces may be created making the solution more compressible as it appears from the higher apparent molar compressibility values in dioxane solvent. The adiabatic compressibility shows the increased association of molecules by lower β values, whereas, apparent molar compressibility also shows the increased association but at the same time structuredness of the solution by higher ϕK values.

In acetone the addition of polar solute may break the structuredness of the solvent and form bulk of solute-solvent, as is seen from the lower apparent molar compressibility value. Thus it may be predicted that adiabatic compressibility can detect gross changes in interaction but minute changes due to change in structure may only be noticed by apparent molar compressibility.

Notable change is not seen in ϕK values whether any of the –OH or –NO_2 groups are present at ortho or para position.

Specific acoustic impedance (Z) also makes the contribution in explaining molecular interactions. Literature survey shows that the impedance approach to explain the molecular interactions in liquid mixtures has been rather less commonly employed.[20] This is one of the reasons why the impedance approach has been adopted here, to examine the behaviour of the solutions regarding molecular interactions. The mathematical relation for specific acoustic impedance $Z = v \cdot d$ and adiabatic compressibility $\beta = 1/v^2 \cdot d$ shows that their behaviour is opposite.

The conventional approach based on compressibility is both useful and fundamental; however, acoustic impedance constitutes an additional probe for studying molecular interactions. Specific acoustic impedance is the complex ratio of the effective sound pressure at a point to the effective particle velocity at that point.[21]

It can be seen from Table 1-6 and Fig 3 that the values of Z in dioxane solvents are much higher than in acetone solvent. This can happen only when the effective particle velocity increases; this in turn means that dispersion forces should be active in mixture, a result anticipated in the absence of any specific interactions such as hydrogen bonding[20] etc.

Further the data analysis (Table 1-6 and Fig. 3) shows the reverseness in trend as compared with β, that is the values for acetone are lower than dioxane and addition of metal ion increases acoustic impedance, meaning thereby the solute-solvent interactions are increasing with the increasing polarity of solute and solute-solute interactions are also increasing. This also indicates the molecular packing in the medium increases with polarity of solutes. The variations in Z values are, Comp-dioxane > Comp - acetone and Fe(III)-Comp-solvent > Cu(II)- Comp-solvent > Comp-solvent. Thus, the specific acoustic impedance depends upon the various structures of the liquid and the molecular packing in the medium.

Exactly opposite trends than that of β values are seen in Z values on moving the group from ortho to para position or changing the group from –OH to –NO_2.

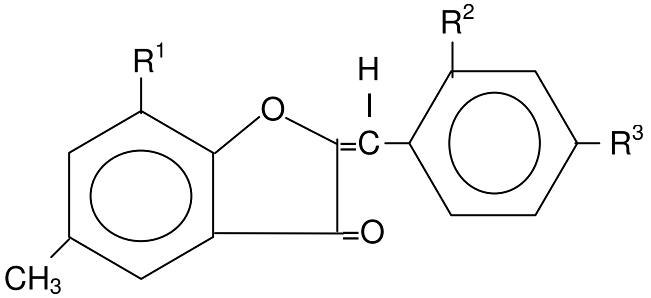
Relative association is an acoustic property of understanding interaction, which is influenced[22] by two opposing factors:

(i) Breaking of solvent structure on addition of solute to it and
(ii) Solvation of the solutes, those are simultaneously present, by the free solvent molecules.

The former effect results in the decrease in R_A values while the latter resulting in increase of R_A values. From the Table 1-6 and Fig. 4, it can be easily seen that the R_A values in dioxane are higher than in acetone for the compounds, which is a reverse trend than that is observed for β. The general trend can be shown as -

Comp-dioxane > Comp-acetone;
Comp-dioxane > Cu(II)-Comp-dioxane > Fe(III)-Comp-dioxane and
Comp-acetone < Cu(II)-Comp-acetone < Fe(III)-Comp-acetone.

In acetone, increase in R_A on addition of M ion suggests that solvation of solute is predominant over the breaking of solvent structure, whereas, in dioxane decrease in R_A suggests that breaking of solvent structure predominates over the solvation of solute due to non-polar nature of dioxane.



Coumaran-3-ones (1-8)

Comp.		R^1	R^2	R^3
(1)	:	H	OH	H
(2)	:	H	H	OH
(3)	:	Br	OH	H
(4)	:	Br	H	OH
(5)	:	H	NO_2	H
(6)	:	H	H	NO_2
(7)	:	Br	NO_2	H
(8)	:	Br	H	NO_2

Scheme-1

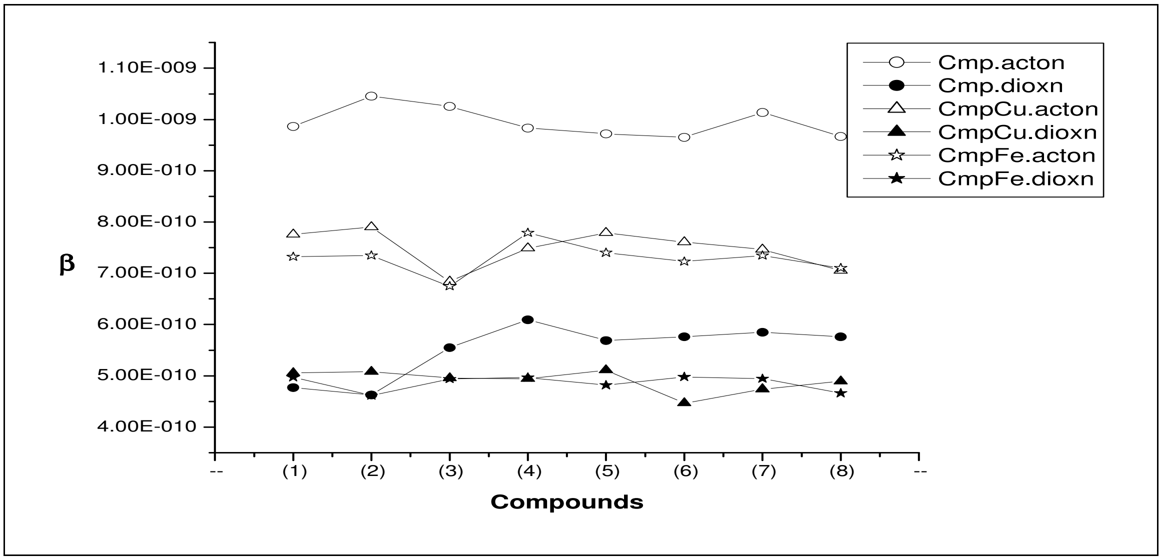


Fig.-1: Adiabatic compressibility (β) of coumaran-3-ones

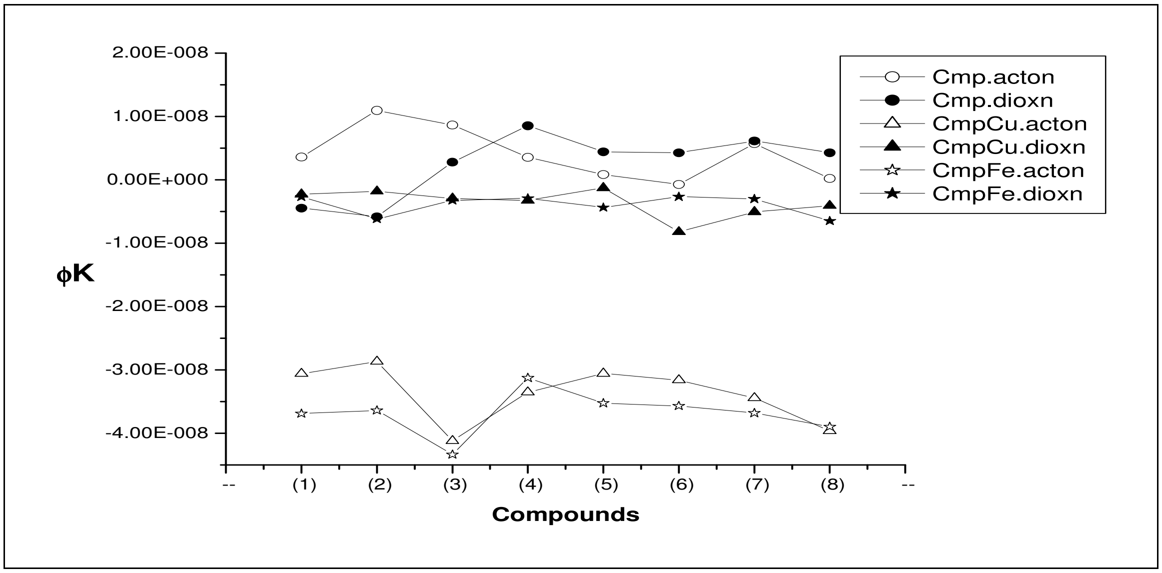


Fig.-2: Apparent molar compressibility (ϕK) of coumaran-3-ones

With the addition of Fe(III) and Cu(II) to acetone naturally the tendency of association should increase which is evident from our experimental observations, where R_A is increased as compared to that with the solvent alone. Similar effect is observed in case of compressibility.

In case of solute Fe(III) and Cu(II) added to dioxane the interaction/association of dioxane molecules is almost absent and Fe(III) and Cu(II) will have almost negligible association with dioxane molecules due to the charged neutrality and zero dipole moment of the dioxane molecule. Thus overall association of solute in dioxane will be further reduced as compared to pure dioxane.

Relative association decreases on changing the –OH group from ortho to para position in both acetone and dioxane solvents. But interestingly in compounds no. 7 and 8 where $–NO_2$ group is changing from ortho to para position R_A increases in dioxane solvent whereas decreases in acetone solvent.

Jacobson[17] pointed out that certain properties of liquid state could be studied to an advantage as a function of free length between molecules. These properties include those, which are mainly dependent on the forces between the molecules such as compressibility, surface tension, viscosity etc. Intermolecular free length for these studies is chosen because; intermolecular forces, which determine the property of liquids, consist of attractive and repulsive forces. The attractive forces depend on the distance between the centers of attraction of molecules, while the repulsive forces are dependent on the distance between the surfaces of molecules.

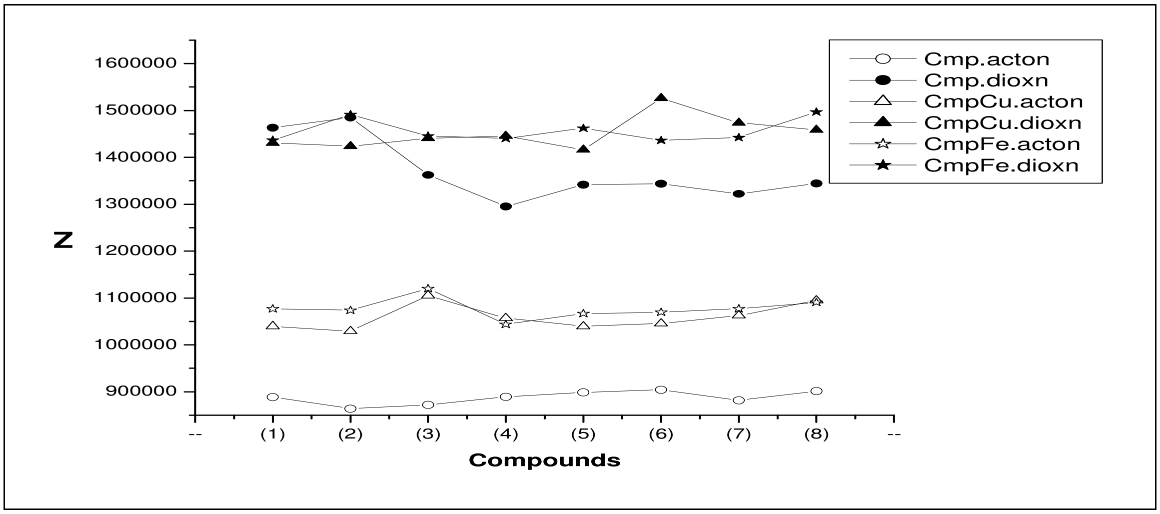


Fig.-3: Specific acoustic impedance (Z) of coumaran-3-ones

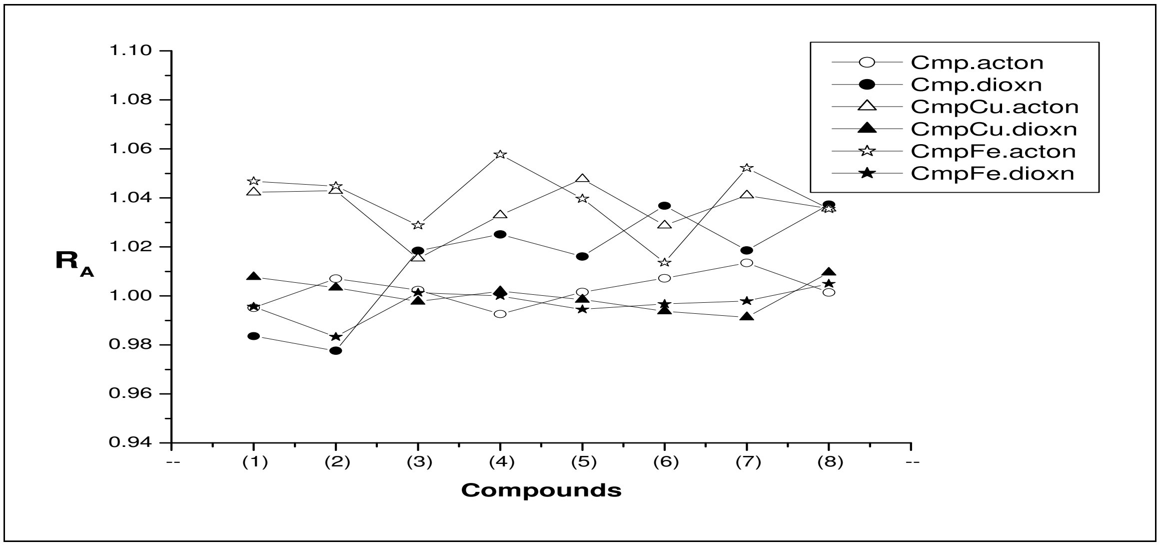


Fig.-4: Relative Association (R_A) of coumaran-3-ones

The values of L_f for the compounds in acetone (Table 1) are much higher which may be because of polar and volatile nature of acetone resulting in more free length available. When a polar solute is added it also gets associated in the structure by electrostriction thus decreasing the free space available.

In case of dioxane, because of its non-polar nature the compact packing of molecules is already there and when polar solute is added because of its association again free space decreases. Therefore, the L_f values in dioxane must be smaller (Table 2).

When the metal ions are added, the polar-polar associations still increase and the L_f decreases (Fig. 5). Ultrasonic velocity depends on intermolecular free length L_f, with decrease in free length velocity increases or vice versa.[23] The trend in change of L_f values is as,

Comp-acetone > Comp-dioxane and
Comp-solvent > Comp-Cu(II)-solvent > Comp-Fe(III)-solvent

In solvent acetone L_f values increase from ortho to para for –OH group whereas opposite sequence is seen for $–NO_2$ group.

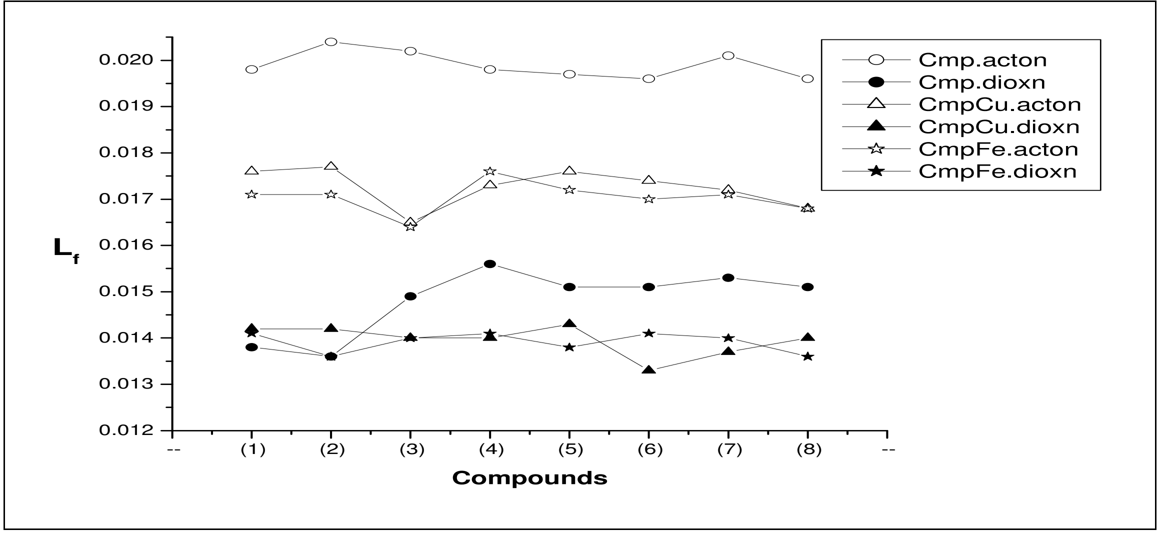


Fig.-5: Intermolecular Free Length (L_f) of coumaran-3-one

Thus it can be generalized that except Z the other acoustic properties increase when –OH group moves from ortho to para position. This may be due to the optimum size of –OH group. When it is at ortho position the compactness of molecule is there and therefore compressibility is less. Whereas, when it is at para position the molecule may be relaxed some what which may increase the spaces and thereby making the solution more compressible.

Generally opposite trends are observed in case of $–NO_2$ group. This may be attributed to the bigger size of $–NO_2$ group, which when present at ortho position orients to increase void spaces that give more compressibility to the solution but at para position the molecule in solution must be oriented compactly thereby decreasing the compressibility of solution.

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