

Ion-Solvent Interaction Behaviour of Lithium Perchlorate in *N,N*-Dimethylacetamide and its Binary Mixtures with 2-Aminoethanol: FTIR Vibrational and NMR Spectroscopy Study

SURESH KUMAR^{1,2,*}, NARENDER SINGH¹ and HARDEEP ANAND¹

¹Department of Chemistry, Kurukshetra University, Kurukshetra-136119, India

²Department of Chemistry, Markanda National College, Shahabad Markanda-136135, India

*Corresponding author: E-mail: suresh980980@gmail.com

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FTIR vibrational spectra and NMR spectra were studied for different electrolytic concentrations of lithium perchlorate (LiClO_4) in pure *N,N*-dimethylacetamide (DMA) and its binary mixtures with 2-amino ethanol (AE) of the different salt concentration ranges from 0.25 to 1.0 M with a molar ratio of LiClO_4 :AE:DMA (4:4:4). The ^{13}C NMR and FTIR studies have been used to investigate the behaviour of O=C-N deformation vibration and change in chemical shift. Study revealed that the Li^+ ions coordinate to the nitrogen atom of NH_4^+ structure and O of the C=O group of DMA. The interaction between Li^+ ions and solvent molecules was confirmed by the carbonyl stretch symmetric ring deformation bands in DMA. From both studies it was found that the intensity increases, band split and chemical shift values of C atom of C=O of amide group also change with the increase in the concentrations of LiClO_4 in binary mixtures of AE + DMA, indicating a strong coordination between Li^+ ions and DMA.

Keywords: FTIR Spectra, NMR spectra, Lithium perchlorate, *N,N*-dimethylacetamide, 2-Aminoethanol.

INTRODUCTION

A great applicability of the aqueous solutions, organic polar and non-polar electrolytes in lithium ion batteries is having current interest [1-10]. Lithium ion batteries used the electrolyte of lithium salts such as LiPtF_6 , LiClO_4 , LiBF_4 and LiXs in the binary mixtures of organic polar and non-polar solvents such as in propylene carbonate, ethylene carbonate, acetonitrile, acetone, pyridine, DMF and other additives in past decades. These electrolytic solutions were used in batteries of small electronic devices such as mobile phones, laptops, tablets as well as in hybrid electric vehicles [1,2]. Therefore, from a strong, important and meaningful point of view, it becomes essential regarding the research and development of lithium ion battery with high performance.

Vibrational spectroscopy is a powerful technique for studying the ion-solvent and ion-ion interactions. It occurs through the changes of frequency, intensity and other band properties [8,9]. Several experimental results for the electrolyte solutions [3,4,6,10-15] explained the dependency of general properties and performances. Moon *et al.* [3] reported the solvation of

lithium ion in propylene carbonate through vibrational and ^1H NMR spectroscopy. In the Raman and IR spectra of $\text{LiBF}_4/\gamma\text{-BL}$, Xuan *et al.* [16] reported the significant characteristic peaks of propylene carbonate. Additionally, Xuan *et al.* [17] studied the infrared spectroscopy of LiBF_4 in propylene carbonate with different concentration the electrolytic salt and LiClO_4 in the binary mixtures of propylene carbonate + DMF. James & Mayes [17-19] studied the ion-ion and ion-solvent interactions in solutions of different concentrations of LiClO_4 in acetone and LiBF_4 in acetonitrile through IR & NMR and IR spectroscopy & DST methods, respectively.

According to the literature, several researchers reported a new solvent series for LiClO_4 in different solvents (both aqueous and non-aqueous) and their binary mixtures [19-23]. Therefore, the aim of the present study is about the interaction of LiClO_4 in DMA and its binary mixtures with 2-aminoethanol with different concentrations of the electrolyte in a definite molar ratio. The binary mixture of DMA with 2-aminoethanol (AE) is selected in the present study, due to its aprotic dipolar nature (dielectric constant value of 37.8 at 298.15 K) and has a number of conversion applications in lithium ion batteries. In order to

investigate, the interaction of LiClO_4 with the solutions of $\text{LiClO}_4/\text{DMA}$, $\text{LiClO}_4/\text{AE} + \text{DMA}$ in the molar ratio 4:4:4 with LiClO_4 concentration ranging from 0.25 M to 1.0 M at room temperature (298.15 K) and ambient pressure.

EXPERIMENTAL

2-Aminoethanol (AE) (99.5% GC grade, TCI, Japan) and *N,N*-dimethylacetamide (DMA) (99.5%, GC grade, TCI, Japan) were used without further purification. The salt LiClO_4 (Hi-Media, AR grade) was twice purified by recrystallization in acetonitrile, then dried under vacuum for 36 h at 170 °C. All the solutions were prepared by weight as molar ratio in a dry room and their concentration were expressed as molality (mol/kg).

A series of concentrations of LiClO_4 salts in pure DMA and AE + DMA binary mixtures were prepared from the stock solutions of appropriate concentrations in the 4:4:4 ($\text{LiClO}_4/\text{AE}/\text{DMA}$) molar ratio. These solutions were kept in air tight stoppered glass bottles to avoid the absorption of atmospheric moisture. These solutions were prepared by molar ratio, using an A&D Company Limited electronic balance (Japan, Model GR-202) with precision of ± 0.01 mg. The measurements were repeated twice in order to obtain reproducible results.

^1H & ^{13}C NMR spectra data of the samples were conducted on a JEOL 400 MHz spectrometer with CDCl_3 as standard reference. The FT-IR spectra of binary mixture of EA + DMA (1:1), LiClO_4 in pure DMA (1:1) and $\text{LiClO}_4:\text{AE}:\text{DMA}$ (4:4:4) were recorded using the ABB Horizon (MB 3000) spectrometer. This spectrometer has resolution of the order of 4 cm^{-1} and signal to noise ratio 50,000:1. All the investigations were made at room temperature.

RESULTS AND DISCUSSION

Interaction behaviour between 2-aminoethanol (AE) and *N,N*-dimethylacetamide (DMA): The IR spectrum of binary mixtures of AE and DMA (1:1) was recorded at room temperature (Fig. 1). The peak at 3356 cm^{-1} is due to H-bonding between $\text{N}-\text{C}=\text{O}$ of DMA and $-\text{OH}$ and $-\text{NH}_2$ group of 2-aminoethanol. The other peaks were observed at 1636 cm^{-1} due to resonance of $\text{NH}-\text{C}=\text{O}$ group, C-N stretching at 1396 cm^{-1} and $\text{N}-\text{C}=\text{O}$ peak at 633 cm^{-1} . The peak of pure DMA was observed at 1690 cm^{-1} but decreases due to H-bonding. The study confirmed by observing the characteristic absorption band shifts of $-\text{OH}$ group at $3600\text{--}3000\text{ cm}^{-1}$ in 2-aminoethanol with mixtures of DPA, DEA and TEA [24]. The study confirmed about the form-

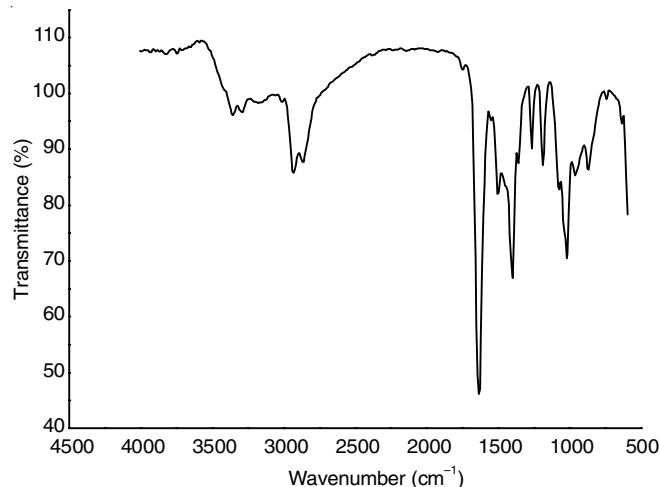


Fig. 1. IR spectra of AE and DMA in the hydrogen bonding formation (3356 cm^{-1}) and the $\text{O}=\text{C}-\text{N}$ deformation with $\text{C}=\text{O}$ stretching (1636 cm^{-1})

ation of hydrogen bond between $-\text{OH}$ groups of 2-aminoethanol molecules and *N*-atoms of DMA.

Interaction behaviour between Li^+ ions and *N,N*-dimethylacetamide (DMA): The IR spectra of $\text{LiClO}_4:\text{DMA}$ (1:1) solutions in pure DMA at room temperature are shown in Fig. 2. The peak of $\text{NH}-\text{C}=\text{O}$ was shifted towards lower wave number at 1628 cm^{-1} instead of 1636 cm^{-1} which is less than the peak observed in the binary mixture of AE + DMA (1:1), this shows that the structure formations between Li^+ ion and *N*-atom/ $\text{C}=\text{O}$ group of DMA molecule is due to formation of coordinated complex [25,26] and confirms the ion-solvent interactions [24]. Another peak was also shifted from 3356 cm^{-1} to 3448 cm^{-1} that is higher wavenumber in IR spectra at a molar ratio (1:1) of $\text{LiClO}_4/\text{DMA}$ than the binary mixed solution of AE + DMA. Other shifts in the peaks in the IR spectra indicated that the interaction of Li^+ ions with DMA in the structural formation. Besides, there is no other change in shifts in the IR spectral peaks, *i.e.* at 741 cm^{-1} , C-N stretching mode frequency were increased from 1396 cm^{-1} to 1404 cm^{-1} . The investigation shows the coordination behaviour of Li^+ ions and DMA, the bond exist between $\text{N}-\text{C}=\text{O}$ and Li^+ ion. Therefore, it strongly proved the ion-solvent and ion-ion interactions helps to identify the structure making process towards all the studied concentrations of LiClO_4 .

Interaction behaviour between Li^+ ion and AE+DMA binary mixtures: The IR spectra of four different concentra-

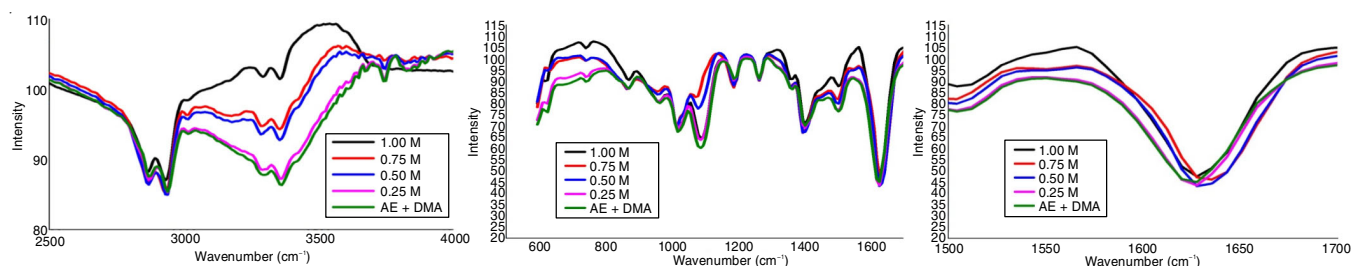


Fig. 2. IR spectra of AE and DMA in the hydrogen bonding formation (3364 cm^{-1}) and the $\text{O}=\text{C}-\text{N}$ deformation with $\text{C}=\text{O}$ stretching (1628 cm^{-1}) and C-N stretching at around 741 cm^{-1} for the solution possessing different concentrations of LiClO_4 in the molar ratio of $\text{LiClO}_4:\text{AE}:\text{DMA}$ of 4:4:4. (a) 0; (b) 0.25; (c) 0.50; (d) 0.75; (e) 1.0 M (from bottom to top)

tion solutions in the range of 0.25, 0.50, 0.75 and 1.0 M in the molar ratio of LiClO₄:AE:DMA (4:4:4) at room temperature were also analyzed (Fig. 2). As in binary solutions, the wave number of N-C=O stretching IR bands changes after 0.5 M concentration in fixed molar ratio with the increase in molarity as 1.0 M of LiClO₄. The peak of NH-C=O were shifted towards lower wave number at 1628 cm⁻¹ which is less than the peak observed of AE+DMA (1:1) binary mixture, this shows that the structure making formations between Li⁺ ion and DMA or AE molecule is due to ion-solvent interactions. There is no more shifting of frequency in IR spectra at low concentration, but as the concentration of lithium salt is increased up to 1.0 M, the shifts in the peaks of IR spectra were observed from 3356 cm⁻¹ to 3364 cm⁻¹ and 633 cm⁻¹ were shifted to 741 cm⁻¹, C-N stretching mode frequency were increased from 1396 cm⁻¹ to 1404 cm⁻¹. However, the IR characteristics bands are different for binary mixed solutions as compared to pure DMA and 0.25 and 0.50 M solution of LiClO₄. This strongly confirmed the interaction between Li⁺ ion and binary solvent. The shift in the peak is due to greater solvation of Li⁺ ion with the increase in concentration of lithium salt in binary mixed solutions. This also showed about the coordination behaviour of Li⁺ ion and DMA, the bond exists between N-C=O and Li⁺ ion. Therefore, it strongly suggested that the ion-solvent and ion-ion interaction helps to identify the structure making process at higher concentrations.

This has been inferred from the shifts in lower value of C=O stretching vibration in the IR spectra [24,27] confirmed the solvation of Li⁺ ion with the oxygen in O=C-N group of DMF due to the rotation of C-N bond [5,18]. This was also due to the presence of low-energy, infrared active vibrations [24]. The binding modes of SCN [25-27] indicated that the CX group's stretching vibration values are binded through X, which affected by lowering in energy and increase in intensity [28]. Thus, it has been predictable that the (O=C-N) group in DMA will behaved as analogous to DMF. The shifts in IR vibrational band of O=C-N deformation in DMA appeared at 633 cm⁻¹ with the increase in concentration of LiClO₄ in binary mixtures, another band observed at around 741 cm⁻¹. As the concentration of the solution changes from 0.25 M to 1.0 M, the intensity of the band increases. Since, the N-atom of amide group is highly confined due to the presence of two bigger alkyl groups. So,

the solvation of Li⁺ ion in binary mixture through the N-atom of amide group would cause a considerable change in the constraint and as well as small increase in the band parameters. Moon *et al.* [3] & Alia *et al.* [29] studied the interaction between ClO₄⁻ and a non-aqueous solvent such as DMF and describe that the ClO₄⁻ has a little solvation in non-aqueous solutions with the shift in too small change in frequencies of the IR vibrational spectra. Therefore, the intensity and change in frequency of vibrational modes of the -N-C bond in DMA should be pointed out towards the association between Li⁺ and the N-atom on the -N-C bond in DMA. This is due to empty 2s orbital of Li⁺ ions of comparable energy with 2s orbital of N-atom having a lone pair of electrons on it, hence, it is probably because Li⁺ ions can interact easily with the N-atom and form the complex of [LiNMe₂CO-CH₃]⁺. Therefore on the basis of the above assumptions, the change in frequencies of IR spectra of O=C-N deformation mode was most possibly the joint effect of two interactions.

According to the above discussion, change in vibrational frequencies and intensities of the correlated complexes is most dependable on the combination of two interactions. (i) Li⁺ ions may associate with the O of the C=O group and (ii) N-atom of -N-C bond in DMA. The relative intensity of the bond appeared at 633 cm⁻¹ band indicates about the involvement of 'free' DMA in binary mixtures and has no longer power towards the intensity of the collected band.

As on the above results, Li⁺ ions can interact with O-atoms of the C=O bond or N- atoms of -N-C structural bond of DMA, the change in vibrational frequencies and intensities of the associated complexes were most dependable on the two above association. When Li⁺ ions associated with the O or N atoms of the O=C or N-C structures, their binding energy is expected to be lowered to some degree, which will produce the decrease in the vibrational frequencies of the groups.

NMR analysis: NMR techniques are very helpful to determine the solvent characteristics of the solvated ions having coordinated solvent spheres and large amount of ion pairs [18,23]. Several researchers [30-33] studied the coordination behaviour of lithium ions to the carbonyl groups (C=O) of the carbonate-based solvents using ¹³C NMR spectroscopic technique. All the ¹³C & ¹H NMR data are reported in Table-1. The results suggested that the LiClO₄ salt concentrations increase in

TABLE-1
¹H NMR AND ¹³C NMR DATA OF BINARY MIXTURES, SOLVENT AND SOLUTIONS OF DIFFERENT CONCENTRATIONS OF LITHIUM PERCHLORATE IN DMA AND AE + DMA (4:4:4) MOLAR RATIO

Conc. molar ratio	¹ H NMR		¹³ C NMR			
	C ¹ (δ)-N-(CH ₃) ₂ (A)	C ² (δ)-CH ₃ -C=O (B)	C ¹ (δ)-CH ₂	C ² (δ)-CH ₂	C ³ (δ)-CH ₂	C ⁴ (δ)-C=O
Pure DMA	2.98	2.08	21.51	35.03	38.00	170.49
LiClO ₄ :DMA (1:1)	2.388, 2.536	1.551	20.01	33.85	36.84	170.23
AE + DMA (1:1)	6.095 (H-bonding)	6.723, 6.737	20.33	33.88	36.89	169.56
Binary mixture						
LiClO ₄ :AE + DMA (0.25:4:4)	2.536	1.327	20.02	33.58	36.61	169.45
LiClO ₄ :AE + DMA (0.50:4:4)	2.043, 2.195	1.205	19.30	32.88	35.91	168.84
LiClO ₄ :AE + DMA (0.75:4:4)	2.494, 2.416	1.430	19.84 (20.19)	33.70 (33.57)	36.75 (36.57)	169.63
LiClO ₄ :AE + DMA (1.00:4:4)	2.446, 2.269	1.385	20.20 (19.89)	33.72 (33.58)	36.77 (36.58)	169.60
Pure AE	1.989 (-NH ₂ , singlet)	1.854 (-OH)	62.247 (-CH ₂ -OH)	43.12 (-CH ₂ -NH ₂)		

binary mixture systems, the shifts in the ^{13}C resonance of DMA were recorded with small change in chemical shift in the HN-C=O group. FTIR spectroscopy measures the relatively slow stretch of the carbonyl group, while the NMR measures the relatively rapid change in spin states, for this reason, it is possible to observe two distinct absorptions *via* FTIR, while only one signal appears on the NMR spectrum.

The lithium ions have coordination number of four on an estimated average in the binary mixed systems of amides, esters and carbonates [34]. Coordination of Li^+ ions in solvents *i.e.*, propylene carbonate and ethylene carbonate is achieved through the carbonyl groups, as evident from the enhanced deshielding of the carbonyl carbons in the ^{13}C NMR spectra [35]. Using gradient field NMR and conductivity studies, it was shown that 1 M LiBF_4 is dissociated to 30% extent in propylene carbonate solutions [30]. It was also shown that about 1.8 propylene carbonate molecules are coordinated to each of the Li^+ ions at comparatively high concentration [31,32].

The solvation of different salt concentrations of LiClO_4 in amide and binary mixture of alcohol + amine solvents (EA and DMA) was investigated *via* ^1H & ^{13}C NMR spectroscopy. The subsequent additions of LiClO_4 into a binary solvent mixture shows a change in the chemical shift as shielding of C-atoms of amide of C=O functional group of DMA than AE, it has been suggested that DMA binds Li^+ more strongly than AE. When the addition of electropositive metal to the binary mixtures of organic solvents or of the single solvent, the electrons push by the metal ion, results give indication of the binding of metal ion with any donor atoms of the particular functional group of the solvent or solvent systems through coordinate bond.

This change in the electronic environment of solvent's carbonyl-carbon due to Li^+ ion coordination may also be detected from the ^{13}C NMR spectroscopy as these solvents possess characteristic carbonyl resonances in ^{13}C NMR 170.23 and 169.45 ppm for $\text{LiClO}_4/\text{DMA}$ and $\text{LiClO}_4/\text{AE} + \text{DMA}$, respectively. With the increase in the concentration of LiClO_4 in binary solvent system, the coordination of the ion-dipole of the dissociated Li^+ ion effectively directs a shielding effect and the carbonyl carbon experiences upfield. From measurement of the degree of the experimental chemical shift, as a result, determination of the degree of coordination of the solvent to ion is possible. The results from IR were confirmed by comparing with ^1H & ^{13}C resonance spectra of the same solutions. Upon Li^+ coordination, the ^{13}C resonance of the solvent's carbonyl carbon shifts in the proportion with respect to the quantity in which salt is present in solution, indicates the degree of solvent coordination.

At high concentrations, additional solvation effects may dominate and minimal preferential solvation may exist. The value changes in chemical shifts of the protons as a shielding effect up to 0.5 M but deshielding shows beyond this concentration with increasing lithium perchlorate concentration in the binary mixture system. With increase in the concentration of lithium perchlorate in binary mixture system, the chemical shift values are not clear due to the overlapping of peaks in the same region, so the values cannot be measured with accuracy.

Changes in the chemical shifts of ^1H & ^{13}C NMR spectra provide extra evidences for the extent of solvation and results

are reported in Table-1. It is evident that the carbon atom of C=O in $\text{H}_2\text{N}-\text{C}=\text{O}$ has greater deshielding than the other two methyl groups present in DMA. It has explained the polarization nature of Li^+ ion by shifting of the electron density towards the carbon and results in deshielding will be most evident towards less electronegative carbon atom. A similar deshielding effect in all three carbon atoms were shown by the solvation through N-atom of amide group. The large change in amide carbon indicates that the Li^+ ions coordinate with the O atom of $\text{H}_2\text{N}-\text{C}=\text{O}$ through solvation.

Conclusion

The IR and NMR spectral studies revealed that the interaction of Li^+ ion with O- or N-atom of amide group of *N,N*-dimethylacetamide (DMA) or O atom of ClO_4^- , which confirmed the hydrogen bonding in the binary mixtures of AE-DMA (1:1), $\text{LiClO}_4:\text{DMA}$ and $\text{LiClO}_4:\text{AE}:\text{DMA}$ (4:4:4) fixed molar solution. Therefore, it can be concluded that the interaction of Li^+ ion with 2-amino ethanol (AE), DMA and in their binary mixtures were characterized by IR and NMR spectroscopic studies. Interactions of Li^+ with DMA are stronger in $\text{LiClO}_4:\text{DMA}$ than in the $\text{LiClO}_4:\text{AE}:\text{DMA}$ solutions. The specific solvation of Li^+ was distinguished in the AE:DMA systems, where DMA preferentially coordinates to Li^+ .

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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