SYNTHESES OF SOME SCHIFF BASE NEMATIC MESOGENS AND THE STUDY OF THEIR BINARY PHASE DIAGRAMS BY MISCIBILITY EXPERIMENTS

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Abstract

The following four Schiff base compounds known to exhibit the nematic phase only have been prepared.(a)2-Hydroxy-4-Ethoxybenzylidene-4-Butylaniline(OH-EBBA).(b)4-Cyanobenzylidene-4-methoxyaniline (CNBMeOA). (c) 2-Hydroxy – 4 - Methoxybenzylidene-4-Butylaniline (OH-MBBA).(d) 4- Methylbenzylidene - 4- cyanoaniline (MeBCNA). The compounds have been characterized by UV/VIS, IR, and H' NMR spectra. Two binary systems were prepared. System 1, by mixing OH – EBBA and CNBMeOA; and system 2, by mixing OH –MBBA and MeBCNA. The liquid crystalline behavior of the prepared compounds and those of the mixtures of the two systems was studied by differential scanning calorimetry (DSC) and hot-stage polarized optical microscopy (POM). Comparative study on the binary phase diagrams of the two systems revealed that, the permanent dipole – induced dipole interactions responsible for the induced phases, semctic C (SmC) and smectic A (Sm A), over a long range of mole fractions, are much stronger in system 1 than in system 2. An explanation for this behavior has been put forward.

Introduction

There have been a large number of studies on the binary phase diagrams of mixtures which exhibit the induced smectic A phase (Sackmann and Demus, 1973- Dernus et al, 1971-Srikanta and Madhusudana 1983)

Such a phase is observed in some composition range of the mixtures when one of the components has the strongly polar cyano or nitro end group while the other component has no such group. Thus permanent dipole-induced dipole interactions appear to be important for the occurrence of the induced phase. In binary systems of the same series of compounds where both constituents do not have any strongly polar end groups, and hence no dipole - induced dipole interactions appear in such system. Instead the expected enlargement of the nematic phase temperature range occurs. This is indicated by the minimum (the eutectic mixture melting point) which is well below the melting point of the two constituents, for example the 2-Hydroxy-4binary system of Ethoxybenzylidene -4-Butylaniline and 2-Hydroxy-4-Methoxybenzylidene -4-Butylaniline (Haas et al, 1973). In another binary system made by mixing constituents A and B (chemical structures shown below) where both exhibit SmC and SmI phases (Hamad, 2001).



In this system the intermolecular interaction between the unlike molecules (through hydrogen bonding) leads to a lower temperature eutectic point as well as an appreciable enlargement of SmI phase temperature range at the expensive of that for the SmC phase. Miscibility experiments are widely used to obtain eutectic mixtures of liquid crystals (especially nematics) that have a wide nematic phase temperature range (preferable from -60 to well above room temperature) to be used in electro-optic- cells in liquid crystal displays (LCDS) (Hamad, 2001). The aim of this work is to study two binary systems where the two constituents of each system are all pure nematogens and may form induced phase like SmC and / or SmA

Material and Methods

(a) 4- Methylbenzylidene-4-Cyanoaniline (MeBCNA) (Ojala, 2007):

The compound was prepared by adding (0.02 mole) of the 4-methylbezaldehyde to (0.02 mole) of 4- aminobenzonitrile, then adding (50 ml) of absolute ethanol to the mixture with (2 drops) of glacial acetic acid. Refluxed the mixture for one hour. Then the mixture was cooled, filtered, washed with very cold ethanol and dried under vacuum over anhydrous CaCl2. After several recrystallisations from ethanol, the pure compound was obtained.

(b) 2-Hydroxy-4-Methoxybenzylidene -4-Butylaniline (OH-MBBA) and 2-Hydroxy-4-Ethoxybenzylidene -4-Butylaniline (OH-EBBA)(Liberko,2000-Patch,1985-Sadle,1979-Verbit ,1972- Keller and Liebert ,1978)

In each case the compound was prepared by dissolving (0.025 mole) of (2-hydroxyl-4methoxybenzaldehyde or 2-hydroxyl-4 ethoxybenzaldehyde) in (25 ml) of absolute ethanol, then added (0.025 mole) of (4- butyl aniline), followed by (3 drops) of glacial acetic acid and refluxed for 30 minutes. Then the mixture was cooled, filtered, (15 ml) of toluene added to the mixture in a separation funnel, then washed the mixtures with 5% solution of NaHCO3 in H2O, and then with (25 ml) of H2O. Then distilled the organic layer to remove the toluene completely, washed the solid with very cold ethanol and dried under vacuum over anhydrous CaCl2. After several recrystallisations from ethanol, the pure compounds were obtained.

(C) 4-Cyanobenzylidene -4-methoxyaniline (CNBMeOA)(Subodh, 2006):

This compound was prepared by dissolving (0.025 mole) of (4-cyanobenzaldehyde) in (25 ml) of absolute ethanol, then added (0.025 mole) of (4- methoxyaniline) and (3 drops) of glacial acetic acid. After reflux for 30 minutes, the mixture was cooled, filtered then (15 ml) of toluene added to the mixture in the separation funnel, washed the mixture with 5% solution of NaHCO3, and then with (25 ml) of H2O. Then distilled the organic layer to remove the toluene completely, washed the solid with very cold ethanol and dried under vacuum over anhydrous

CaCl2. After several recrystallisations from ethanol, the pure compound was obtained.

Preparation of binary mixtures

Binary mixtures (with mole fraction range 0.1 to 0.9) for both systems were prepared by taking the requisite amounts of each component, accurately weighed using a sensitive balance, to obtain the correct mole fraction for each component. Then each time the two components were mixed together, heated until a clear isotropic liquid was obtained, then cooled to room temperature, dried in a vacuum descecator and finally crushed into a powder.

System I:

2-Hydroxy-4-ethoxybenzylidene -4butylaniline (OH-EBBA) with 4-Cyanobenzylidene-4-methoxyaniline (CNBMeOA).

System II:

2-Hydroxy-4-methoxybenzylidene -4butylaniline (OH- MBBA) with

4-Methylbenzylidene-4-cyanoaniline (MeBCNA).

Results and discussion

All compounds exhibited intense IR (Perkin Elmer) absorptions of (C=C stretch) at between (1585 - 1598) (Subodh, 2006) cm-1, (C=N cyano stretch) between (1618 - 1627) (Silva et al, 1997) cm-1. (C – H) stretching aromatic between (3030 - 3065) cm-1. (CH=N) stretching unsaturated SP2 between (2920 - 2960) (Faeza, 2011) cm-1. (C-C) stretching between (1161 - 1190) (Subodh, 2006) cm-1. (C-H) bending out of plan between (822 - 846) (Govindarajan et al, 2010) cm-1.

The UV (Pye Unicam) absorption spectra in chloroform of prepared compounds were identical two bands with λ max between (245 – 279) nm (log ε = 4.12 and 4.22) respectively and λ max between (341 – 358) nm (log ε = 4.31 and 4.01) respectively.

1H NMR (Bruker 400 MHz) spectra of compounds. The samples were dissolved in deuterated chloroform, CDCl3, at room temperature (298 K).

1H NMR of OH-MBBA compound (t, 3H) at (0.94-0.97) ppm, (Sextet, 2H) at (1.36-1.59) ppm, (q, 2H) at (1.61-1.67) ppm, (t, 2H) at (2.63-2.67) ppm, (S, 3H) at (3.8) ppm, (m, 2H) at (6.48-6.53) ppm, (m, 5H) at (7.2-7.3) ppm, (S, 1H) at (8.5) ppm, (S, 1H)at (13.9) ppm.

1H NMR of OH-EBBA compound (t, 3H) at (0.94-0.98) ppm, (Sextet, 2H) at (1.36-1.47)

ppm, (t, 3H) at (1.44-1.47) ppm, (q, 2H) at (1.58-1.65) ppm, (t, 2H) at (2.63-2.67) ppm, (q, 2H) at (4.07-4.12) ppm, (m, 2H) at (6.47-6.51) ppm, (m, 5H) at (7.2-7.3) ppm, (S, 1H) at (8.5) ppm, (S, 1H)at (13.9) ppm.

1H NMR of CNBMeOA compound (S, 3H) at (3.87) ppm, (d, 2H) at (6.96 – 6.98) ppm, (d, 2H) at (7.28 – 7.32) ppm, (d, 2H) at (8 - 8.02) ppm, (d, 2H) at (7.76 - 7.78) ppm, (S, 1H) at (8.5) ppm.

1H NMR of MeBCNA compound (S, 3H) at (2.4) ppm, (d, 2H) at (7.22 – 7.24) ppm, (d, 2H) at (7.31 – 7.33) ppm, (d, 2H) at (7.68 – 7.69) ppm, (d, 2H) at (7.80 - 7.82) ppm, (S, 1H) at (8.3) ppm.

DSC (DSC-60 Shimadzu) analyses on the four pure nematogenic compounds that were prepared revealed, as expected two peaks, for each of three compounds (OH-EBBA), (CNBMeOA), and (OH-MBBA). In each case the first low temperature peak corresponds to the crystal to nematic (C - N) transition. In the case of the fourth compound, MeBCNA, the DSC curve displayed only one very large broad peak where the two transitions, (C -N) and (N - I), overlapped together due to the running of recorder chart speed at a very fast rate.

Polarized Optical Microscopy study (POM), by coupling a microscope (Labomed) to a heating stage (Linkam THM 600), was carried out on the above four compounds and revealed the expected two transitions in each case. Transition temperatures obtained from (POM) study were found to be higher than those obtained from DSC measurements. This is partly due to not calibrating the DSC instrument with indium before measurement. Our (POM) results agree very well with the data reported by other workers (Madhusudana and Moodithaya, 1983-Belarbi et al, 1991 - www.surechem.org, 1978).

The transition temperatures and the values of Δ H and Δ S for these transitions (where possible) are given in Tables (1 and 2). Transition temperatures obtained by hot-stage POM study were taken to be more precise than those from DSC measurements and were used in the calculation. Transition temperatures obtained by POM agree with the data reported by other workers [Madhusudana and Moodithaya, 1983-Belarbi et al, 1991 - Van der meulen

Zijlstra, 1983- www.surechem.org, 1978), and these values were used throughout this work.

Tuble (1). The number competition temperatures in (C) for the prepared competities					
Compounds	(C-	N)	(N	-I)	
	DSC	POM	DSC	РОМ	
OH-MBBA	40.61	44	58.29	65	
OH-EBBA	65.15	72	78.71	87	
CNBMeOA	110.27	112	120	123	
MeBCNA		110		124	

 Table (1): The transition temperatures in (°C) for the prepared compounds

Table (2): The values of enthalpy and entropy changes for the various transitions for the compounds studied

compounds	T _{C-N}	ΔH_{C-N}	ΔS_{C-N}	T _{N-I}	ΔH_{N-1}	ΔS_{N-1}
	in C	-1 J.mol	-1 -1 J.mol .K	in C	-1 J.mol	-1 -1 J.mol .K
OH-MBBA	44	17788.948	56.116	65	181.34639	0.536
OH-EBBA	72	26350.897	76.379	87	240.8783	0.669
CNBMeOA	112	17233.036	44.761	123		
MeBCNA	110	18178.815	47.464	124		

Study of Binary phase diagram of system I.

As mentioned earlier, both components (OH-EBBA and CNBMeOA) of this system exhibit, in the pure state, only the nematic phase. The first three binary mixtures with mole fractions (0.1, 0.2, and 0.3) of CNBMeOA exhibited the nematic phase only. A slight maximum at $(103^{\circ}C)$ in the (N-I transition) and minimum at $(62 \degree C)$ in the (C-N transition) was exhibited by the mixture having 0.2 mole fraction CNBMeOA. During the POM study the microscope was coupled to a computer via a camera to display the textures exhibited by nematic phase for these three mixtures. Again transitions obtained by hot-stage POM study were higher than those from DSC for the same reason mentioned earlier. DSC curve for the mixture having 0.3 mole fraction of CNBMeOA is shown in (Figure 1) and (Figure 2) shows the nematic texture exhibited the same mixture.



Figure 1: Heating DSC curves of 0.3 mole fraction of CNBMeOA



Figure 2: texture of nematic phase for 0.3 mole fraction of CNBMeOA at 96° (magnification 250)

The second three binary mixtures with mole fraction (0.4, 0.5, and 0.6) of CNBMeOA all exhibited the nematic phase as well as an induced SmC phase. DSC curve for the mixture

with 0.5 mole fraction (Figure 3) shows the transitions that are consistent with the results obtained from POM study, but again transitions temperatures obtained from POM were higher. The texture exhibited by the induced S_mC by this mixture is shown in (Figure 4).



Figure 3: Heating DSC curves of 0.5 mole fraction of CNBMeOA



Figure 4: Texture of S_mC phase for 0.5 mole fraction of CNBMeOA at 86°C (magnification 250)

The two binary mixtures with the mole fractions (0.7 and 0.8) of CNBMeOA both exhibited only two induced phases, S_mC and S_mA as was revealed by POM study. DSC curves for these two binary mixtures displayed only two peaks, again, due to fast chart speed of the DSC recorder, an overlap of the last two transitions, [(S_mC - S_mA) and (S_mA - I), has occurred. Textures exhibited by these two induced S_mC and S_mA phases, obtained from POM study, are displayed in (Figures 5 and 6



Figure 5: Texture of S_mA phase for 0.7 mole fraction of CNBMeOA at 107°C (magnification 250)



Figure 6: Texture of S_mC phase for 0.7 mole fraction of CNBMeOA at 100°C (magnification 250)

Binary mixture with mole fraction 0.9 of CNBMeOA was studied by hot-stage POM only and revealed three transitions, $(C - S_mC)$ at 70°C, $(S_mC - N)$ at 93 °C, and (N - I) at 127 °C. The complete phase diagram for system 1 is shown in (Figure 7). All transition temperatures for various phases exhibited by these binary mixtures of system 1 are given in Table (3). The changes in ΔH and ΔS (where possible) associated with the various transitions are reported in

The changes in ΔH and ΔS (where possible) associated with the various transitions are reported in Table (4).

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of	C-S _m C	C-N	S _m C- S _m A	S _m C-N	S _m A-I	N-I
CNBMeOA						
0		72				87
0.1		64				99
0.2		62				103
0.3		63				98
0.4	62			78		94
0.5	61			90		96
0.6	60			100		102
0.7	60		104		111	
0.8	62		102		120	
0.9	70			93		127
1		112				123

Table (3): The transition temperatures obtained by hot-stage POM in °C for binary system I Mole fraction

Mole fraction of CNBMeOA	T _{C-SmC} °C	∆H _{c-smc} J mol ⁻¹	∆S _{c-smc} J mol ⁻¹ K ⁻¹	Т _{С-N} ° С	∆H _{C-N} J mol ⁻¹	∆S _{C-N} J mol ⁻¹ K ⁻¹
0.2				62	24500.70	73.136421
0.3				63	14725.22	43.825074
0.4	62	15600.8	46.56981			
0.5	61	11115.8	33.28083			
0.6	60	10777.7	32.36564			
0.7	60	5237.08	15.72699			
0.8	62	5817.07	17.36439			

Table (4): The values of enthalpy and entropy for [(C-S_mC) and (C-N)] transition of system I



Figure 7: Phase diagram of the first binary system (I)

Study of Binary phase diagram of system II.

Similarly (as mentioned earlier) both components (OH-MBBA and MeBCNA) of this system exhibit, in the pure state, only the nematic phase. This system was studied by (POM) only and the first three binary mixtures with mole fractions (0.1, 0.2, and 0.3) of MeBCNA exhibited the nematic phase only. A slight minimum at $(35^{\circ}C)$ in the (C -N transition) was exhibited by the binary mixture with mole fraction of 0.1 of MeBCNA, while a slight maximum at (82 °C) in the (N -I transition) was exhibited by the binary mixture having mole fraction 0.2 of MeBCNA. Some regions of biphase (during the second heating of this mixture were detected. During the second heating of the mixture having mole fraction 0.3 of MeBCNA, dark patch increased above 55 °C (indicating homeotropic molecular orientation) and approaching 83 °C all the sample looked completely dark under crossed polars. On cooling, nematic droplets appeared at 83 °C. Again, some regions of biphase (immiscibility, the existence of solid and nematic phases together) were detected, see (Figure 8).



Figure 8: Textures of nematic phase 0.3 mole fraction of MeBCNA at 81°C (magnification 250)



Figure 9: Texture of (C-I) transition for 0.6 mole fraction of MeBCNA at 101° C (magnification 250)

Then the next four binary mixtures having mole fractions (0.4, 0.5, 0.6, and 0.7) of MeBCNA did not exhibit any liquid crystalline phase and only one phase transition, namely (C-I) was detected by hot – stage POM study. (Figure 9) Shows the texture exhibited by the mixture with mole fraction 0.6 of MeBCNA.

Hot- stage POM study on the mixture having mole fraction 0.8 of MeBCNA revealed only an induced S_mC phase at (100 °C) and a phase transition (S_mC –I) at (119 °C).

Finally, when the study of the mixture with mole fraction 0.9 of MeBCNA was carried out, it displayed an induced S_mC phase at (95 °C) as well as a nematic phase at (105 °C) and turned into the isotropic liquid at (130 °C). A slight minimum in the induced (C – S_mC) transition at (95 °C) and a slight maximum in the (N –I) transition are exhibited by this mixture. All the transition temperatures of the various phases exhibited by the binary mixtures of system 2 are given in Table (5). The complete phase diagram for system 2 is shown in (Figure 10).

	1	<i>γ</i> υ		
Mole fraction of	C-S _m C	C-N	S _m C-N	Transition to
MBCNA				isotropic liquid
0		44		N – I 65
0.1		35		N – I 75
0.2		39		N – I 82
0.3		57		N – I 83
0.4				C-I 90
0.5				C-I 96
0.6				C-I 101
0.7				C-I 106
0.8	100			S _m C-I 119
0.9	95		105	N – I 130
1		110		N – I 124





Figure 10: Phase diagram of the second binary system (II)

It is a well known fact that permanent dipole -induced dipole interactions are not only very important but they are essential for the occurrence of the induced phase. Experimental evidence indicated that a charge transfer complex formation takes place between the molecules of the two components, the highly polar component acting as the acceptor while the other component acts as the donor. (Srikanta and Madhusudana, 1983 - Sharma et al, 1980- Fukai and Matsunaga, 1981)

The magnitude, μ , of the permanent dipole moment and its angle with long molecular axis depends on the sign of the dielectric anisotropy, $\Delta \varepsilon$, of the uniaxial nematic liquid crystal. The value of $\Delta \varepsilon$ is defined by the relation: $\Delta \varepsilon = \varepsilon \parallel$ $-\varepsilon^{\perp}$, where $\varepsilon \parallel$ and ε^{\perp} are the dielectric constants in the directions parallel and perpendicular to the nematic director respectively. The determination of the dielectric properties of these nematic LC'S is very essential for their potential development in display and nonlinear optics device technologies. Dielectric measurements may also be used to determine. with accuracy, the transition temperatures in LC'S and the technique permits to detect structure changes within the mesophase involving very small enthalpy changes (Belarbi - Massouras et al, 1991 - Somerton, 1988-Belarbi et al, 1988). Nematic compounds with cyano group substituents along their molecular axis possess very high polarizabilities which make them suitable for second optical harmonic generation.(Madhusudana and Moodithaya, 1983)

During this study thin films of (pure components and of their mixtures, for both systems) were placed between untreated glass surfaces of ordinary microscope slides and their cover slips. In such studies, without the use of any magnetic or electric fields ,any strong tendency of the director to prefer homogeneous (parallel) or homeotropic (perpendicular) molecular alignment will be dictated by the dielectric anisotropy of the mixture and the temperature.

For sytem 1, OH-EBBA has a negative dielectric anisotropy (Srikanta and Madhusudana,1983)

(NDA), $\Delta \epsilon = -1.45$; while CNBMeOA has a positive dielectric anisotropy (Belarbi – Massouras et al, 1991) (PDA), $\Delta \epsilon = 10.4$. Hence, for the first three mixtures that are rich in OH-EBBA only the nematic phase is exhibited.

As the mole fraction of CNBMeOA (with strong permanent dipole moment of the cyano group parallel to the molecular axis) increases to 0.4, an induced SmC (titled smectic) appears and further increases in mole fraction enhance the stability and temperature range of this tilted phase. Mixtures having mole fractions (0.7 and 0.8) of CNBMeOA exhibit (at and above 104 and 102°C respectively) a second induced phase, the SmA phase (with orthogonal molecular alignment). Thus the region of maximum interaction of permanent dipole – induced dipole for system 1 is in mixtures with mole fractions in the range (0.4 to 0.9) of CNBMeOA.

Since for nematic liquid crystals, the anisotropy of polarizability is always positive, a negative dielectric anisotropy (NDA) exhibited by OH – MBBA [($\Delta \epsilon = -0.45$) compared with that of OH – EBBA ($\Delta \epsilon = -1.45$)] (Srikanta

and Madhusudana, 1983-Arora and Agarwal, 1978)

is thus caused by the dipole moment of these two compounds which has a perceptible component in the direction perpendicular to the long molecular axis.

The two compunds, 4- cyanobenzylidene -4methoxyaniline (CNBMeOA) and 4-methylbenzylidene 4 _ cyanoaniline (MeBCNA), both possess strong permanent dipole moment of the cyano group parallel to the molecular axis. Therefore both of these compounds should possess high and positive dielectric anisotropies (PDA). The dielectric constant of CNBMeOA (used in system 1) is available in literature, ($\Delta \epsilon = 10.4$) [23], that for MeBCNA (used in system 2) is not available in literature but it is expected to be in the same range.

As can be clearly seen, the permanent dipole – induced dipole interactions responsible for the induced phases (SmC and SmA), over along range of mole fractions, are much stronger in system 1 than those in system 2. This could be due to the dipole of the methoxy group at the other end of CNBMeOA molecule which may interact with the glass surface of the microscope slide and this will enhance the formation of tilted alignment necessary for the molecules in the (SmC) phase ,and orthogonal alignment necessary for the (SmA) phase at higher temperatures.

The methyl group at the other end of MeBCNA compound (in system 2) cannot provide such facilities. However, measurements of dielectric anisotropies of these mixtures at the required temperature ranges are necessary to prove this.

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تحضير بعض من قواعد شف ودراسة مخططات الاطوار

الخلاصه

السائل للمركبات المحضره وكذلك للانظمه ثنائيه المكون بوساطة جهاز المسح التفاضلي المسعري (DSC) والجهر ذي الضوء المستقطب المزود بلوحة التسخين (POM). واظهرت دراسة مخططات الاطوار الثنائيه للنظامين الاول والثاني بأن التداخلات بين عزم ثنائي القطب- وعزم ثنائي القطب المحتث هي المسؤله عن ظهور الطور السمكتي (SmC) والطور السمكتي (SmA) بمدى واسع من الكسر المولى والتي تكون بالنسبه للنظام الاول أقوى مما في النظام الثاني .