CHAPTER - I

INTRODUCTION

Historically, it is interesting to read that the concept of liquid crystalline appeared in the science fiction literature of the eighteenth century in the "Narative of Arthur Gordon Pym" by the science fiction writer Edger Alen Poe. He describes a miraculous water that behaves in a strange manner, sometimes behaving with certain properties and some other times with certain other properties. It is as well interesting to note that the first liquid crystal was discovered by a botanist Friedrich Reinitzer [1,2], when he observed that Cholesteryl benzoate showed an unusual kind of melting behaviour. In his famous letter dated back to 14 March 1888 [2] he wrote to Lehman that 'The substance has two melting points, it can be described in such a manner. At 145.5° C it melts to a cloudy but fully liquid melt, which at 178.5° C suddenly becomes completely clear'. The subsequent investigations by a number of researchers established that these are distinct phases of the matter, in contrary to the convention, that when the solid crystalline with positional and orientational order converts into an isotropic liquid, when provided with the required amount of heat. This matter exhibits a cascade of phase transitions between the solid crystalline and the isotropic states, and adopts an intermediate state in which there is a simultaneous possession of solid-like character (molecular order) and liquid-like character (fluidity). The state of aggregation that exhibits a molecular order in size that is comparable to that of solid crystals yet act more or less as a viscous liquid. Hence the degree of long-range positional and orientational order lies intermediate between an almost perfect long-range order of solid crystalline and the statistical long range disorder as isotropic liquids. The molecules constituting the mesophase are usually elongated and rod-like, however Chandrasekhar and coworkers[1] reported that pure compounds of disc-shaped molecules may also form a stable mesophase. The terms liquid crystals or mesomorphic states are employed synonymously in the current usage. The unique optical, electrical and electro-optical properties of liquid crystals, and their importance in many areas of applications attracted major attention from several researchers. This can be seen from the good account of results of investigations carried out and also the important applications which the liquid crystals offer, incorporated in books on liquid crystals written by Caray[3], Sackmann and Demus[4], de Gennes[5], Brown[6], Chandrasekhar[7], Sackmann and Grabeier[8], Edward Williams[9], Gray and Goodby[10], Bahadur[11] and others. The international conferences which have been held regularly since over two decades, of which the proceedings of the results of investigations on various aspects of liquid crystals have been published in the well known journal 'Molecular Crystals and Liquid Crystals' and awarding the noble prize in Physics for the first time to a liquid crystal scientist, P.G de Gennes in

1991 had an added effects. A wide variety of experimental techniques including ultrasonic measurements, nuclear magnetic resonance, X-ray and linear and non-linear optics can fruitfully be used to study the mesophases and the phase transitions in liquid crystals.

TYPES OF LIQUID CRYSTALS:

Liquid crystals are classified into two main groups

See page 57.

I. Thermotropic liquid crystals and

II. Lyotropic Liquid crystals.

I. Thermotropic liquid crystals:

In these liquid crystals the transitions to the mesophases are brought about by purely thermal processes. Thermotropic liquid crystals are observed in pure compounds as well as in homogenous mixtures. They are further categorized as enantiotropic and monotropic. In the enantiotropic liquid crystals the mesophase is formed both while heating as well as cooling whereas monotropic liquid crystals exhibit mesophase only when the isotropic liquids cool.

II. Lyotropic liquid Crystals:

In Lyotropic liquid crystals, the mesophases are brought about by the influence of solvents. Such mesophases are observed in amphiphilic molecules such as soaps, when dissolved in a suitable solvent. For lyotropic substances, concentration is the main physical variable. Lyotropic liquid crystals occur in the living systems and hence are of great importance biologically.

CLASSIFICATION OF THEMOTROPIC LIQUID CRYSTALS:

Following the nomenclature proposed by Friendel[12], Thermotropic liquid crystals are classified into three phases:

1. Nematics
2. Cholesterics
3. Smectics

1. Nematic Liquid Crystals

Nematic liquid crystals possess a high degree of long range orientational order of the molecules but no long range transitional order. Nematic liquid crystals are optically anisotropic, hence all macroscopic physical properties are tensoral in nature, and the mesophase is strongly birefringent. The high degree of molecular orientational order of the molecules which quantitatively described by the order parameter is defined as the second degree Legender polynomial as

$$S=1/2\ (3\cos^2\theta - 1)$$

where θ is the angle between the long axis of the molecules and the preferred direction $\hat{n}$.

The molecules of the nematic mesophase are spontaneously oriented with their long axes approximately parallel. The orientation is however in the direction of the molecules labelled by a unit vector n which is called the dirctor as shown in Figure 1.1. The director is arbitrary in space, and it can be influenced by minor forces, a situation analogous to

Hisenberg ferromagnet, and can change from point to point within the system. The nematic phase is mobile and markedly affected by external fields viz. electrical and magnetic fields. The mesophase owes fluidity to the ease with which the molecules slide past one another, while still retaining their parallelism. X-ray studies, indicate that some nematics are composed of groups of molecules called cybotactic groups [13,14].

Nematic Liquid crystals show threaded, marbled, pseudo-isotropic, schlieren and homogeneous textures.

2. Cholesteric Liquid Crystals :

Because it was discovered in the derivatives of Cholesterol, and the fact that many Cholesteral esters exhibit cholesteric liquid crystalline, the name cholesteric was given, however some other compounds e.g. non-sterol derivatives exhibit a cholesteric mesophase provided that they are optically active. In cholesteric liquid crystals, a gradual twist or rotation of the director n perpendicular to the plane on moving away from the side of the rod like molecules, giving rise to a periodical helical structure as shown in Figure 1.2. The helical structure has the property of being handed, and hence the helical structure twist may be in clockwise or anticlockwise direction. If z-axis of the helical axis, the following equations describe the director $\hat{n}$.

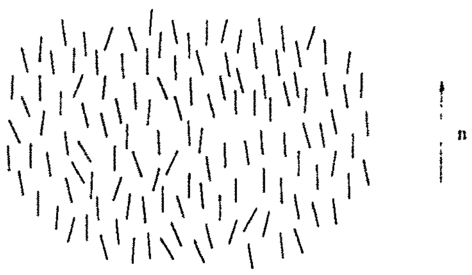


FIG 1 1 The arrangement of molecules in the *nematic* mesophase.

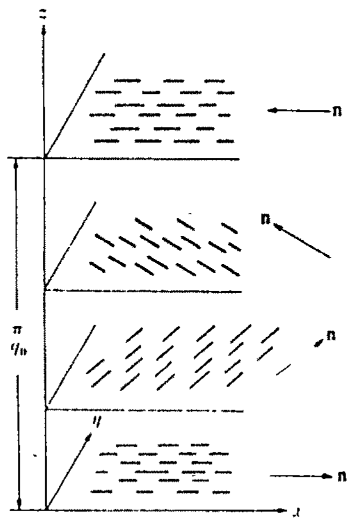


FIG. 1.2 The arrangement of molecules in the *cholesteric* mesophase.

$$n_x = \cos(q_o z + \phi)$$

$$n_y = \sin(q_o z + \phi)$$

$$n_z = 0$$

where z-axis and the value of ϕ are arbitrary, q_o is the pitch length with spatial period.

$$L = \pi / |q_o|$$

The value of the pitch length is comparable to the wavelength of light. The sign of q_o distinguishes between the right-handed and left-handed directions of the helix. This extraordinary mesophase was found to exhibit unusual optical properties. The optical properties are directly related to the pitch length, q_o, and it is also a strong temperature dependent. Cholesteric liquid crystals selectively reflect the light, and they have a great rotatory power [2]. Several other phases called blue phases occur in cholesteric systems at sufficiently low pitch. They exist over a narrow temperature range usually of the order of 1°C between the cholesteric mesophase and isotropic phase. Blue phases are further classified as BP-I, BP-II and BP-III according to their occurence with increasing temperature. All of them are optically active,though they may have colours other than blue, but still they are referred as blue phases [15]. X-ray studies indicate that BP-I is a body-centered cubic, BP-II is a simple cubic lattice and BP-III is amorphous and some times called blue fog.

The cholesterics show focal conic, planer, batonnets and Grandjean step or threaded textures.

3. Smectics:

The name smectic was derived from the Greek Word 'Smectos' which means Soap-like [12]. The smectic mesophase structure consists of layers on which, the molecules lie parallel to one other with their long axis in the direction of the optical axis. Within each layer the molecules are arranged with different molecular packing, and hence accordingly, smectics were further classified into different modifications viz. S_A, S_B, S_C, S_D, S_E, S_F, S_G, S_J, S_I.

The characteristic representation following structural arrangement and molecular orientation of the long axis of the molecules with increasing degree of order can be described in the sequence [16] as $N \rightarrow S_A \rightarrow S_D \rightarrow S_C \rightarrow S_B \rightarrow S_E \rightarrow S_I \rightarrow S_F \rightarrow S_G \rightarrow S_H$. With decrease in temperature. In liquid crystals which are polymesomorphic, smectic mesophases form at lower temperatures than nematics and cholesterics. This is supported by the fact that smectics have higher orientational order, and more viscous at low temperatures than nematics and cholesterics.

Smetic A :

It is a lamellar structure in which the molecules are arranged parallel to their long axis and perpendicular to the layer and so lamellar spacing (d) is equal to the molecular length (L) [1]. The molecular lateral distribution between each layer is random as shown in Figure 1.3. This structure was confirmed by X-ray studies [17]. In any phase sequence which includes S_A, this preceedes all other smectic phases upon cooling either the isotropic liquid or the nematic phase since it possesses the least order. The S_A phase usually adopts one of the two textures namely, the homeotropic or the focal conic fan texture.

Smectic B :

The Smectic B (S_B) is a lamellar structure in which the constituent molecules are arranged within the layers in a hexagonal close-packing, with the molecular long axis perpendicular to the layer plane as shown in Figure 1.4.

The structures was confirmed by X-ray investigations [18-20]. Other investigations [21] show that, there are some cases in which the hexagonal close-packing net arrangement is correlated over large number of layers and not within a given layer only and so S_B is known to be capable of existing in three dimensional form. Smectic B usually exhibits homeotropic, mosaic and focal conic texture.

Smectic C :

It is a lamellar structure in which the constituent molecules are packed parallel to each other but tilted with respect to the normal to the layer; therefore the lamellar spacing (d) is less than the molecular free length (L) as shown in Figure 1.5. The lateral molecular distribution is random, so it is the tilted analog of S_A.

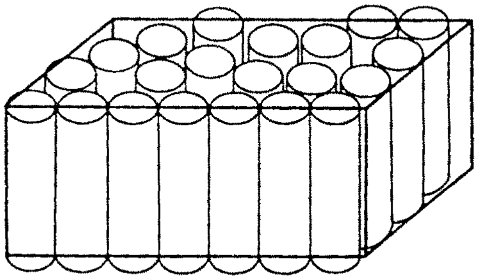
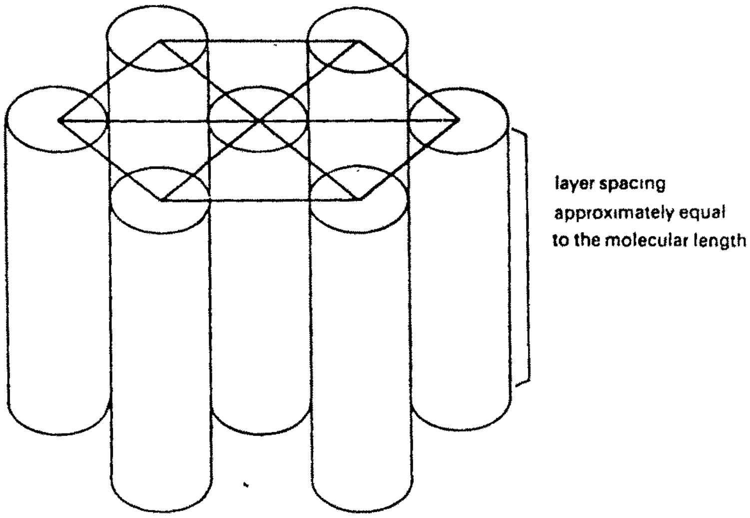


Figure 1.3 Disordered arrangement of molecular centres in a smectic A phase.



hexagonally close-packed smectic B phase

Figure 1.4 The molecules are orthogonal to the layers and packed in a hexagonal array. The rotating molecules are depicted as smooth cylinders

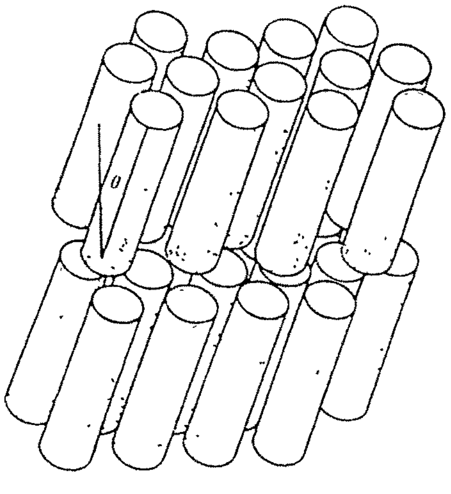


Figure 1·5 Structure of the smectic C phase: there is no regular arrangement of the molecular centres in the planes of the unstructured layers. Tilt angles (θ) may range upwards from $\lesssim 10$

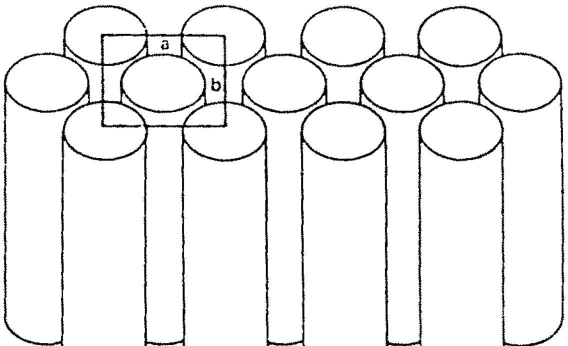


Figure 1·6 The packing arrangement of molecules in a layer in the smectic E phase ($b>a$)—see also Fig. 5.1

The tilt angle is constant over a considerable volume element. S_c is a biaxial. Chiral smectic C phase is exhibited, if the constituent molecules are chiral in nature and optically active. S_c phase exhibits schlieren and focal conic fan textures.

Smectic D :

According to Pelzel and Sackmann [22], the S_D phase has a cubic lattice and it is considerably structured. Since the S_D phase is optically isotropic, it has no texture in the real sense. However the phase is distinguished from the isotropic liquid or homeotropic phases by the fact that the black isotropic S_D nucleates in the birefringent S_c phase occuring at lower temperature in straight-edged squares, rhombus, hexagons and rectangles.

Smectic E :

Smectic E (S_E) phase is a lamellar phase in which the constituent molecules are orthogonal to the layer and are arranged in orthorhombic arrays within the layer as shown in Figure 1.6 [23]. Smectic E phase is highly ordered and is biaxial. Since the molecules are orthogonal to the layer, the lamellar spacing (d) was found to be closely equal to the molecular length (L). X-ray studies show that the layers in the smectic E plane are correlated with each other, giving rise to a 3-dimensional crystal like structure [24].

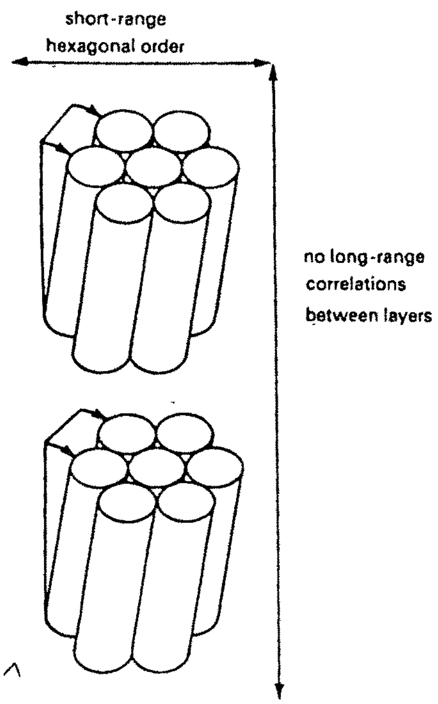


Figure 1.7 Structure of the S_F phase

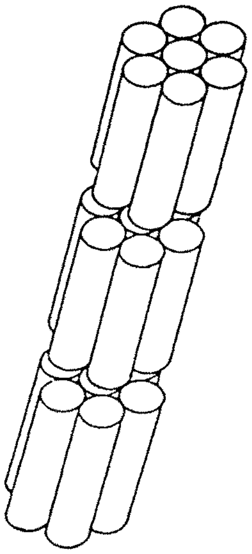


Figure 1.8 The correlated three-dimensional structure of the smectic G phase

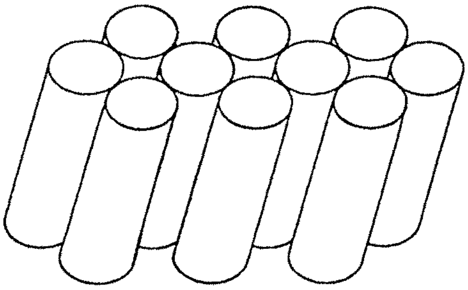
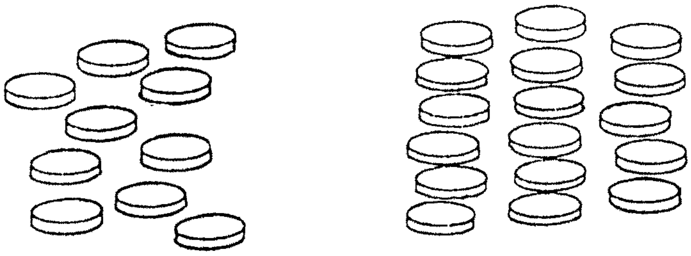


Figure 1·9 The layer structure of a smectic H type phase



(a) (b)

Fig 1·10 Illustration of the (a) nematic and (b) columnar discotic liquid crystal phases. The flat disks represent molecules.

Smectic F:

Smectic F (S_F) is a lamellar structured phase in which the molecules are tilted with respect to the layer planes. The packing in the layers is hexagonal as shown in Figure 1.7. Chiral smectic F phase can be exhibited if the molecules are optically active. Smetic F phase exhibits mosaic, Schlieren and fan type textures. Smetic F phase can also exhibit a large number of paramorphotic textures.

Smectic G :

Smectic G (S_G) is a lamellar structure in which the molecules are arranged in a pseudo-hexagonal packing within each layer as shown in Figure 1.8. The molecules are tilted with respect to the normal to the layer plane. This phase is of crystalline nature since it is a three dimensional structure. Chiral S_G can be exhibited provided that the molecules are optically active. The S_G phase exhibits usually mosaic texture and shows several paramorphic textures also.

Smectic H :

According to X-ray investigations, S_H phase is analogous to S_E phase except that the molecules have their long axis tilted with respect to the normal to the layer planes as shown in Figure 1.9. The phase adopts several paramorphic fan and mosaic textures.

Smectic I :

Smectic I phase is a lamellar structure which is equivalent to

S_F, the only difference is that, while in S_F phase the hexagon is tilted towards the edge, in S_I the tilt is towards the apex of the hexagon. The correlation length is larger than that of S_F phase. S_F is abiaxial phase. S_I phase exhibits two main textures, the broken focal-conic fan and Schlieren textures.

DISCOTIC LIQUID CRYSTALS :

Because the most common liquid crystal phases are those formed by rodlike molecules, the idea prevailed was that the mesophases are formed only by the molecules of this shape. However in 1977, Chandrasekhar and co-workers [1] discovered that disclike molecules can also form liquid crystals in which the long axis perpendicular to the plane of the disc tend to orient along specific direction. These phases and the molecules that form them are called discotic liquid crystals.

The most simple discotic phase is called nematic phase, because there is orientational order but no positional order as shown in Figure 1.10. The molecules move about randomly, but on an average the axis perpendicular to the plane of each molecule tends to orient along a preferred direction called the director n. Another phase called the columnar or smectic phase is formed by discotic molecules in which high orientational order is present and the molecules tend to position themselves in columns as shown in Figure 1.10. The columns are arranged in a hexagonal lattice. Chiral mesophase can also be exhibited in which the director rotates in a helical fashion through out the sample just as in the case of rodlike cholesteric mesophase.

PHYSICAL PROPERTIES OF LIQUID CRYSTALS:

1. Thermal properties:

Thermodynamic properties such as heat capacity, enthalpy and entropy provide much information about the mesomorphic transition and are usefull for comprehensive understanding of the relationship between the mesomorphic transition and molecular dynamics. The Differential Thermal Analysis (DTA) and Differential Scanning Calorimerry (DSC) methods have gained great interest in determining phase transition temperature, heats of transition and heat capacities of liquid crystals. Arnold etal [25-28] have reported several investigations on the thermal properties of liquid crystals. They calculated heat capacities and latent heats in several liquid crystals. It has been found out that the entropy changes at nematic-isotropic and cholesteric -isotropic phase transitions are of the same order and magnitude. A Schiff's base series have been studied by Leclereq etal [29].

Arzotko and Demus [30] have investigated transition enthalpies of six homologous polymesomorphic series. They have shown that the clearing enthalpies of S_A, S_C and S_D phases generally lie in a higher region than nematic clearing enthalpies, while the S-N phase transition in several cases are very low. Navarand and Cox [31] using DSC measurements have shown that S_A-N phase transition of octyl-cynobiphenyl is of the second order while it is found that it is of first order in nonyl-cynobiphenyl. Sorai etal (32) have examined the thermal behaviour of a series of (N-p-alkoxybenzylidine)-p-n-alkylanilines by DTA and showed for the first time that a glassy state of the smectic is exhibited. Chandra and Jaiswal (33) studied the phase transition behaviour of highly purified cholesteryl acetate by DTA and thermal optical microscopy. They found that the appearance of the cholesteric phase depends on both, time period and the temperature at which annealing is performed.

2. X-ray investigations:

X-ray investigations in liquid crystals is valuable in investigating the phase transition, structural studies, classification and estimating the degree of orientational molecular order in liquid crystals. X-ray studies also provide a good tool in detecting the new smectic modifications. A detailed account of the research on X-ray diffraction studies is given in the book of Gray[3] and the review by Brown and Show [34]. Vainstein has investigated long chain molecules and provided a quantitative interpretation of equatorial diffraction by oriented nematic or smectic, by use of cylindrical distribution function. The variation in thickness of smectic layers in polymesomorphic liquid crystals was studied by Guillon et al [35]. The liquid crystalline structure of p-n-alkoxybenzoic acids and p-n-alkoxycinnamic acids was studied by Bryan et al [36-38]. The solid-S_B phase transition of TBBA and other compounds were studied by Levelut, Doucet and Lambert [39-40]. de Vries [41-44] reported important investigations on the behaviour of the mosophase at phase transition using free standing liquid crystal films technique. Pindak et al [45] found that the non-crystalline B phase has short range, in-plane positional correlations. They have interpreted their results in terms of a system of interacting two dimensional hexatic layers. The order parameter in S_A, S_C and nematic phases of N-(n-n-butyloxybenxylidine)-4-octylaniline), TBBA and isobutyl-4-(4'-phenylbenzylidenecmino) cinamate (IBPBAC), using X-ray diffraction technique by Leadbetter and Wrighton was estimated [46]. The structural phase transition of MBBA, DOBAMBC have been studied by energy dispersive X-ray diffraction [47,48]. Pretransitional effects have been observed in 4-propionyl-4'-n-heptanoy- loxyazobenzene [49].

3. **Nuclear Magnetic Resonance:**

Nuclear Magnetic Resonance (NMR) and Electron Paramagnetic Resonance (EPR) studies provide information about the order parameter and molecular dynamics of liquid crystals. A detailed accont of NMR investigations is given in the review by Brown, Dowane and Nelf [50] and Diehl and Khetrapal [51]. Jain and co-workers reported NMR studies on the nematic phase of p-azoxyanisole and interpreted their results in terms of phase of p-azoxyanisole and interperated their results in terms of order resulting from parallel orientation [52,53]. The order parameter S, was estimated in the nematic liquid crystal 4-4'-bis(pentyloxy) azoxybenzene from wide line NMR spectra using doublet splitting; arising from dipole-dipole interaction of the protons [54]. Bose et al [55] have measured the order parameter S_{zz} and $(S_{xx} - S_{yy})$ and their temperature dependence in nematic and smectic phases of p'-(heptyloxy)-azoxybenzene (HOAB). Ruessink B.H. et al have observed pretransitional effect in the isotropic phase using NMR spectroscopy in the presence of electric field [56]. Rai et al [57] have studied the angular variation of the spectra, order parameter S, the rotation correlation time T_R and the anisotropic diffusion parameter N at different temperatures in MPOOB and OBT. Employing EPR, several researchers [58,59] have estimated the order parameter in liquid crystals.

4. **Electrical Properties**:

Liquid crystals exhibit strong anisotropy with a high degree of deformability, which give rise to many effects. If an electric field is applied to a thin layer of nematics contained between two glass plates having transparent conductive coatings on their inner faces, two electrical orientations effects are exhibited, they are described by the terms 'domains' [60-65] and dynamatic scattering [66-68]. By applying electric field of sufficient strength it is possible to convert the amorphous isotropic liquid to nematic at a temperature slightly above the isotropic-nematic phase. A detailed discussion of the electrical properties of liquid crystals is given in a book by Gray and Winsor[69]. The most useful application in the displays is due to field alignment. Heilmeir has done a pioneering research in this area [70]. Kirsanov [71] has studied various domain patterns in nematic phases of MBBA, PAA and p-n-butoxybenzoic acid above the electrodynamic stability threshold voltage. Above the threshold voltage of 7 volts the main pattern in MBBA is converted into 'hexagonal domains'

APPLICATIONS OF LIQUID CRYSTALS:

Due to their unique physical properties, liquid crystals have been exploited in many areas of applications. Optical properties such as birefringence, circular dichroism, optical rotation and colour are found to be sensitive to the changes in environment such as temperature, pressure and presence of an electric field or magnetic field. Due to the colour sensitivity of liquid crystals they have been used for temperature measurements also. Cholesteric liquid crystals provide means for measuring microwave energy, infrared and visible light and laser output.

Cholesteric liquid crystals are used in the electronic industry as non-destructive testing tools. They are also used in medical field to detect the vascular disease and subcontinuous tumours. The use of cholesteric liquid crystals for thermal mapping of the surfaces of aircraft models in the wind tunnel tests has been explored. The quality of bonding the laminates (those used in aircraft structures) can be established by applying a film of cholesteric liquid crystal to one side of the laminate and observing the pattern of heat flow to the other surface. Currently, large number of devices utilizing cholesteric and nematic liquid crystals have appeared. They have been used in all areas of display devices and digital readout for many instruments and microcomputers. Liquid crystals have been exploited in fiber-optic components where they serve as low-voltage, low-power optical switches and attenuators for fibre-optic instruments and communication networks. Liquid crystals have been used as a stationary phases in gas liquid chromatography (g.l.c) as they provide a convenient experimental method for investigating the solvent and certain other properties of the mesophase. Nematic liquid crystals have proved to be useful in orientating solvents in NMR, ESR and infrared spectroscopy. Smectics have shown a potential for use as storage media. Moreover, new uses for liquid crystals are rapidly emerging in different areas of applications.

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