CHARACTERIZATION OF LIQUID CRYSTALS: A LITERATURE REVIEW

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Abstract. Liquid crystals (LCs) have been the source of vast attention of scientific community during last few decades. The unique properties of LCs and their wide applicability have been thoroughly investigated. However, the process of evaluating their applicability for different fields has been initiated from their synthesis and successful characterization. There are certain characterization techniques which assess the ability of a certain compound to exhibit liquid crystalline properties as all related compounds may not have shown LC behavior and hence not an ideal candidate for further applicability. In our current contribution, a brief literature review has been compiled to account for different characterizing techniques for LCs (only last 5-years literature has been considered).

1. INTRODUCTION

The term "liquid crystal", (LC, also called as mesophase) signifies a state of aggregation that is intermediate between the crystalline solid and the amorphous liquid. As a rule, a substance in this state is strongly anisotropic in some of its properties and yet exhibits a certain degree of fluidity which in some cases may be comparable of that of an ordinary liquid. There are several different types of systems exhibiting liquid crystalline properties, Fig. 1.

In addition to certain classes of organic molecules, micellar solutions of surfactants, main and side chain polymers and a large number of biological systems are known to be liquid crystalline. A typical LC molecule could be represented by two parts: i) the central rigid part called as mesogen and ii) the flexible side chains called as spacer. Fig. 2 shows the molecular structure of a typical rod-like liquid crystal molecule. It consists of two or more ring systems connected by a central linkage group. The liquid crystal molecules prefer to align parallel to each other because of the strong intermolecular attraction (π-π interactions).

The characteristic feature of LCs is the presence of long-range orientational order in the arrangement of constituent molecules, and sometimes one- or two- and even three dimensional long-range translational or positional order. LCs exhibit a great variety of phases, which differ one from another by their structures and physical properties. Although LCs combine the properties of a crystalline solid and an isotropic liquid, they exhibit very specific electro-optical phenomena, which have no equivalent analogues in solids or in liquids.

The physical properties of LC medium are governed by the orientational order of the constituent anisotropic molecules. Due to the anisotropic nature of the electrical and magnetic properties, the orientation of the LC molecules could be effectively monitored by weak electric or magnetic fields. Changing the LC molecules orientation, it is possible to change resultant optical and mechanical properties of the medium. All of these factors are important for the functioning of devices based on

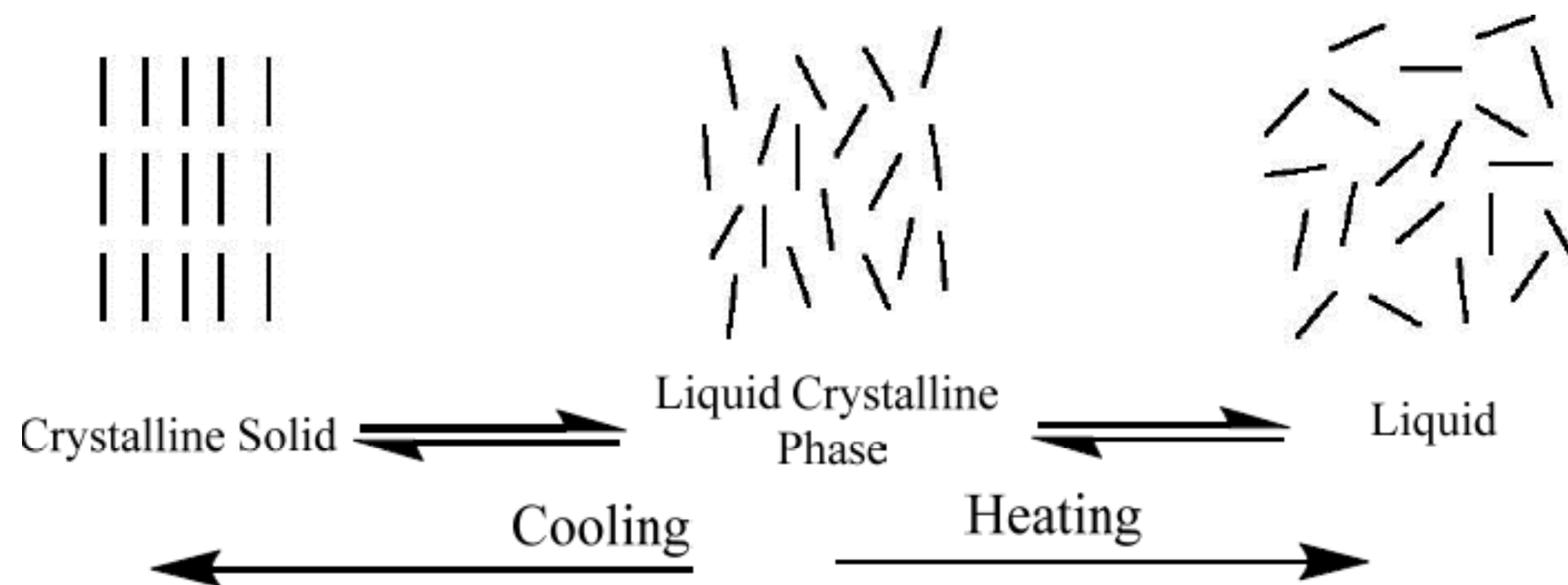Corresponding author: M. Xue, e-mail: minxue@zstu.edu.cn

Fig. 1. Relationship of liquid crystalline phase with liquid and crystalline solid.
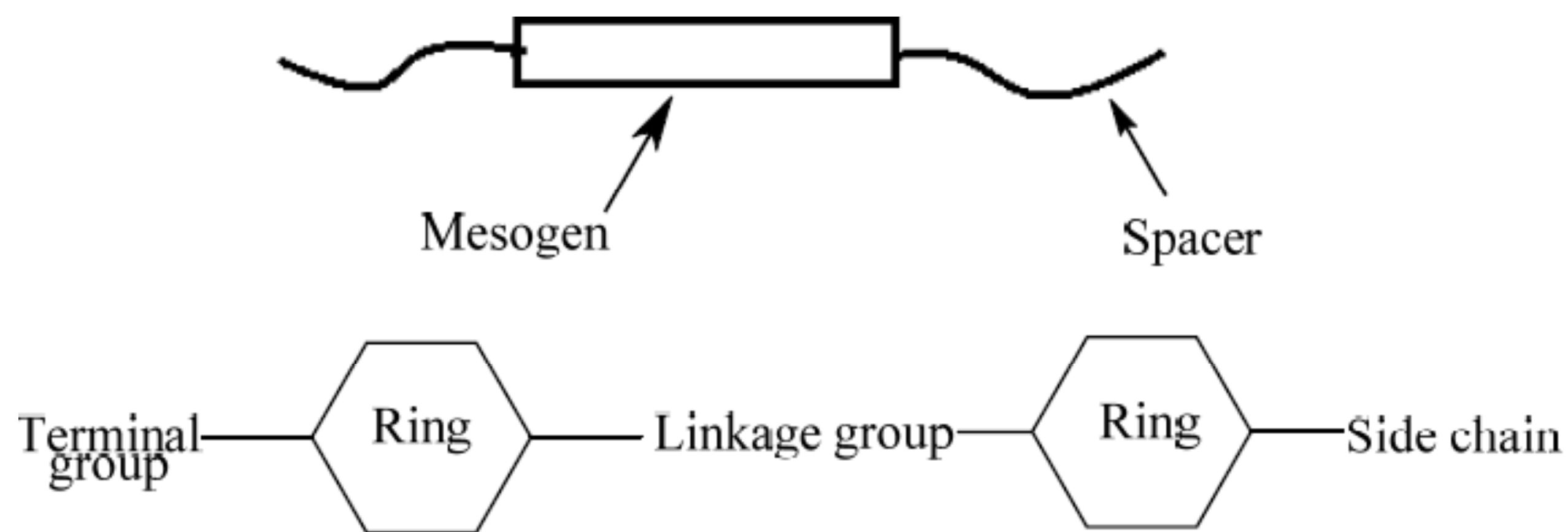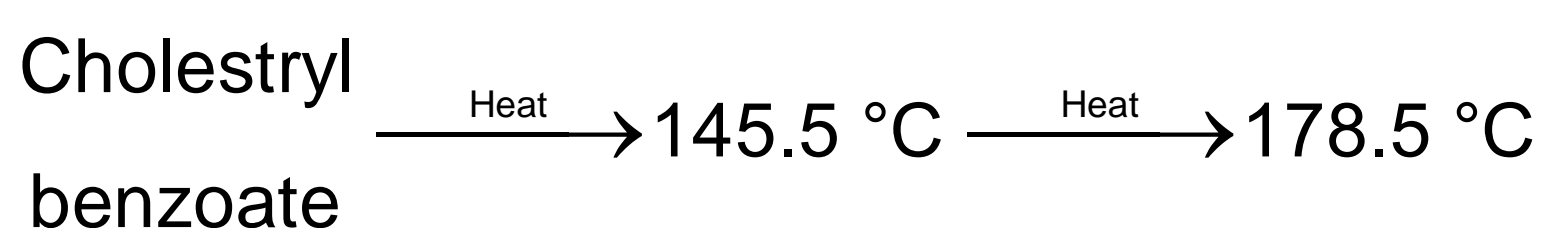

Fig. 2. Typical rod-like shape of a liquid crystal molecule.

LCs, called LC displays, such as digital watches, calculators, panel TVs and thermometers [1-8].

2. HISTORY OF LIQUID CRYSTALS

The discovery of an intermediate, liquid crystalline, state of matter is credited to Friedrich Reinitzer [9]. In 1888, during the experiments on a cholesterol based substance trying to figure out the correct formula and molecular weight of cholesterol, he was struck by the fact that this substance seemed to have two melting points. At 145.5 °C the solid crystal melted into a cloudy liquid which existed until 178.5 °C where the cloudiness suddenly disappeared, giving way to a clear transparent liquid.

$$\text{Cholestryl benzoate} \xrightarrow{\text{Heat}} 145.5\ ^\circ\text{C} \xrightarrow{\text{Heat}} 178.5\ ^\circ\text{C}$$

Later this cloudy liquid was named as "liquid crystal" by Otto Lehmann [10]. In 1904, liquid crystals were firstly commercialized by Merck-AG. Liquid crystals have been classified in different phases (nematic, smectic and cholesteric) based on their structural aspects by Georges Friedel in 1922 [11]. It was not until in the 1950s that further progress was made in understanding liquid crystals (by F.C. Frank and others).

At the end of 1960s, cholesteric LCs were being suggested as temperature indicators, analytical metrology, cancer diagnostics and non-destructive testing of materials. Today, LCs have broad applications in display technology, high modulus fibres and opto-electronic devices [12].

3. CLASSIFICATION OF LIQUID CRYSTALS

The liquid crystals are classified in two major types i.e. thermotropic and lyotropic. These types are further distinguished into different phases depending on the changes in their orientational or positional order under influence of external factors such as temperature. Under appropriate conditions, the molecules of LCs exhibit orientational order such that all the axes line up and form a so-called nematic liquid crystal. The molecules are still able to move around in the fluid, but their orientation remains the same. It is the least ordered LC phase. On the contrary, smectic (Sm) phase exhibits not only orientational order but also positional. In smectic phase, the molecular centers of mass are arranged in layers and the movement is mainly limited inside the layers. In cholesteric LC phase, molecules show intermolecular forces that favor alignment between molecules at a slight angle to one another. The brief classification of liquid crystal has been represented in Fig. 3, see [13].
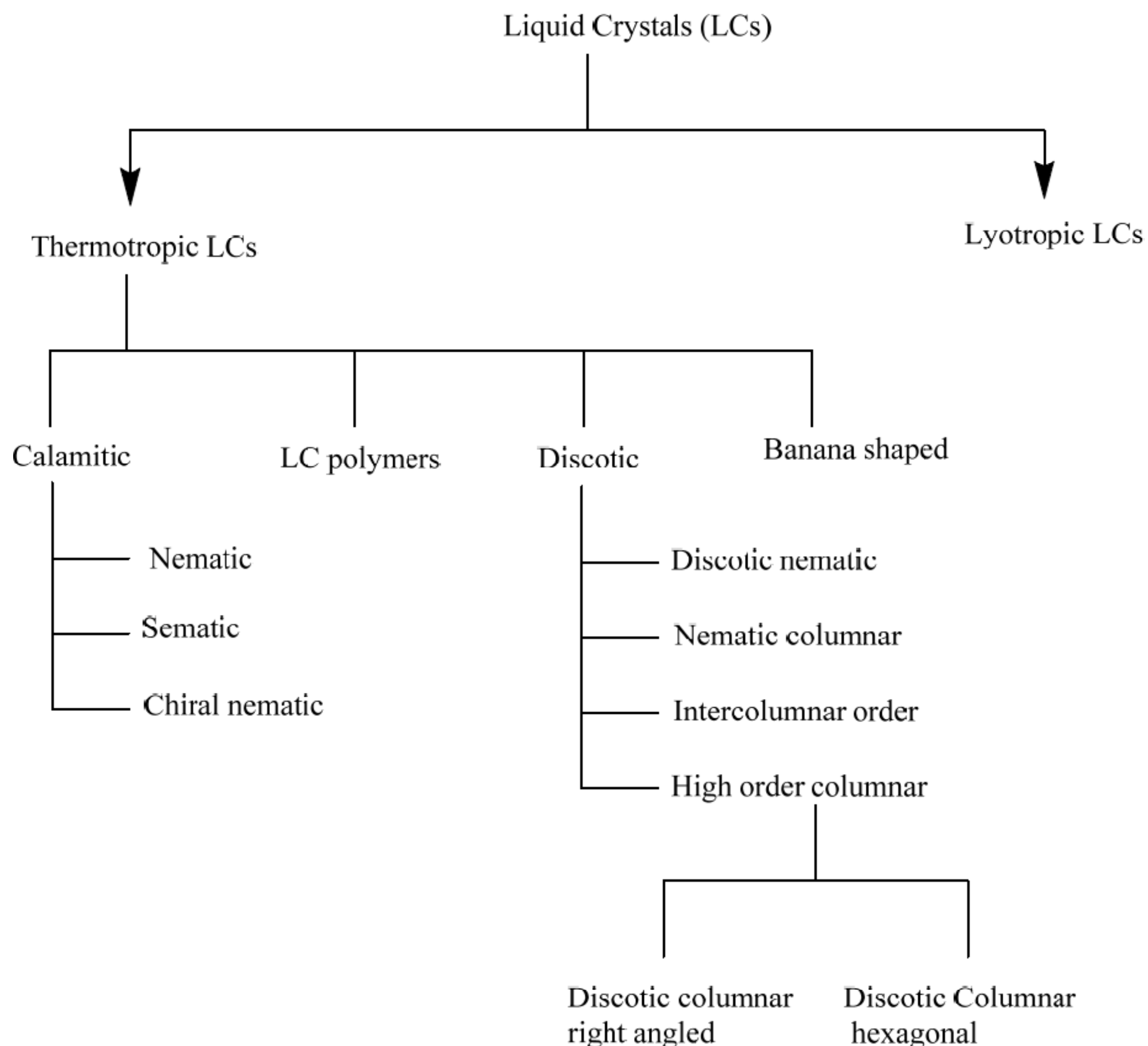
Fig. 3. Classification of LCs, modified from [13].

3. CHARACTERIZATION OF LIQUID CRYSTALS

Various techniques have been used to characterize liquid crystals. The main factors to be considered for describing liquid crystalline structure involve their positional, orientational and bond orientational order. By considering these parameters, one can identify the proper phase as well as the interactions between molecules of liquid crystals.

For macroscopic view, Polarized Optical Microscopy (POM) is a standard tool in the identification of liquid crystal phases and phase transitions but requires considerable experience, particularly in the study of new and less familiar materials. In this case, if the polarized light incident on any material has its polarization direction either parallel or perpendicular to the director, it will appear black under crossed polarizers. Since liquid crystals are anisotropic, they cause light polarized along the director to propagate at a different velocity than that polarized perpendicular to the director, then the LC might appear bright under crossed polarizers. The polarization of light is rotated by the LC molecules.

Along with POM, X-ray diffraction (XRD) provides a much more definitive means for the identification of mesophases. This technique not only helps to determine the structure of the LC phases but also illustrate presence of long range order.

Differential Scanning Calorimetry (DSC) is a helpful technique which complements the optical methods in determining the liquid crystal phase transitions. Grazing-incidence X-ray diffraction (GIXD) is a superior tool to analyze nanostructures in thin films.

A brief literature review has been provided below since 2010 till to date in order to understand that how these techniques have helped to characterize various aspects of LCs.

Roberts et al. have synthesized two homologous series of organo siloxane mesogens based on material design for "de Vries-like" liquid crystal materials. As the design of these new materials is based on a frustration between structural elements that promote either the formation of a SmC phase or SmA phase, measurements of these phases by small-angle X-ray scattering (SAXS) combined with optical tilt angle measurements revealed that the

mesogens were found to undergo SmA-SmC phase transitions. SAXS measurements also gave an insight about the low orientational and high translational order of the compounds, which is consistent with theoretical predictions that such materials should exhibit de Vries-like properties [14].

Vega et al. have used POM, differential scanning calorimetry, and X-ray diffraction for the characterization of LC and have found that depending on the position of the chiral center and polar nature of the target molecules, different types of smectic liquid crystals are formed. In order to further confirm the results of XRD, circular dichroism (CD) measurements have been carried out [15]. Similar characterization techniques have been used by Kim et al. in order to determine the structure of bent-shaped rigid-core molecules with flexible chiral dendrons. It has been observed that the bent aromatic rods based on hepta- and nona-phenylene with nitrile groups at both ends self-assembled into well-ordered hollow tubular structures in aqueous solution, while the bent rod based on heptaphenylene without nitrile groups showed no apparent aggregations in aqueous solution [16].

Stillings et al. have used POM and transmission electron microscopy (TEM) to analyze the morphologies of columnar discotic phases within a discotic block copolymer while using slitlike nano-confinements. It was obvious that birefringence observed was expected for the presence of some types of molecular order. TEM as well as X-ray structural analysis provided information that the columnar order existed both within the homo- and the block copolymer. DSC measurements were performed to analyze the stability of such phases and possible effects induced by confinement texture [17]. Almost same techniques were used in order to characterize and identify the phase transitions of discotic azo compounds [18], mesomorphic azo compounds [19], and other liquid crystalline structures [20-34].

Yasuda et al. have designed electro-functional octupolar columnar LCs. It has been observed that these octupolar molecules self-organize into one-dimensional columnar nanostructures and exhibit transport behavior. Their electronic structures have also been studied by cyclic voltammetry and theoretical calculations [35].

Xie et al. have synthesized new combined main-chain/side-chain LC polymer based on radical polymerization. The phase structures and transitions were investigated using DSC, POM, one- and two-dimensional (1D and 2D) wide-angle X-ray scattering (WAXS). It has been illustrated that the LC polymers can form a hierarchically ordered structure with double orderings on both the nanometer and sub-nanometer length scales. It has also been suggested that the thick main-chains of LC polymers obtained by "jacketing" the central rigid portion of terephthalate side-chain to the polyethylene backbone construct a 2D centered rectangular scaffold, which is stable until the sample becomes completely isotropic [36].

A new photo-responsive side chain liquid crystalline polymer (LCP) has been synthesized. DSC showed that LCP exhibits a crystalline phase that is kinetically trapped by the nematic phase at room temperature. POM with *in situ* UV irradiation was used to characterize the room temperature photo-responsive behavior of its metastable nematic phase [37].

Bagheri et al. have investigated the physical properties of liquid crystals in Athabasca asphaltenes and maltenes identified using experimental methods as POM, DSC and mid- and near-infrared photo-acoustic spectroscopy. It has been suggested that the interdependence of transition phase properties on thermal and mechanical history of sample may help to explain the unexpected and frequently deleterious surface and interfacial phenomena arising during Athabasca bitumen production and processing [38].

Two series of wedge-shaped onium salts [39] and series of tri-block copolymer [40] have been synthesized and characterized by SAXS, WAXS, DSC, and TEM. The phase transition behavior, structural parameters and microphase behavior have been evaluated and discussed in detail in both cases.

Sarman and Laaksonen have calculated the twist viscosity and the alignment angle between the director and the stream lines in shear flow of a liquid crystal model system via using Molecular Dynamics simulation. It has been observed that the smallest twist viscosity arises by rotation around the director formed by the long axes; the second smallest one arises by rotation around the director formed by the normals of the broadsides, and the largest one by rotation around the remaining director. It has also been suggested that the relative magnitudes of components of the twist viscosities span a fairly wide interval, so this model should be useful for determining parameters for experimental data [41].

Reddy et al. have successfully synthesized rod-like mesogens with three- and four-ring core [42] phenyl ring core-based thiophene mesogens [43,44] and pyridine based mesogens [45]. Structural characterizations were done by using XRD and ^{13}C NMR spectroscopy, Fourier Transform-Infra Red spectroscopy (FT-IR) and polarized microscopy. The struc-

tural confirmation, phase transitions and role of hydrogen bonding in building supramolecular assemblies and construction of molecular, oligomeric as well as polymeric materials that show liquid crystalline properties have been investigated in detail by using these techniques.

Petr et al., have synthesized siloxane thermoplastic elastomer and their applicability as room temperature photo-actuator have been accessed by using SAXS, TEM, Dynamic Mechanical Analysis (DMA), DSC, POM, and photocontraction tests.

It has been suggested that as the approach is simple, robust, and applicable to a wide variety of functional moieties, the resulting materials are thermoplastics that can be processed to achieve preferential orientation using standard methods, thus enhancing the capability to produce and utilize functional actuators [46].

Komursa et al. have studied the formation of cylindrical microphase separated structure of a liquid crystalline block copolymer by *in situ* Atomic Force Microscopy (AFM). Grazing Incidence Small Angle X-ray Scattering (GISAXS) analysis with the simple compensation method of X-ray diffraction effect was used to confirm order-order transition change. The growth of cylindrical microphase separation grains was successfully visualized in the film surface by temperature-controlled AFM. It has been suggested that such *in situ* AFM measurements are a significantly powerful tool to afford detailed visual information on structural changes during the formation process of the self-assembled nanostructures [47].

A series of T-shaped polyphilic molecules were synthesized and their thermotropic LC phases were investigated by X-ray diffraction, calorimetry, and microscopy by Ungar et al.

A new variant of the "gyroid" phase, with axially oriented rod-like molecular cores forming the skeleton of the two infinite networks and junctions separated by exactly two molecular lengths have been observed. It has been mentioned that in this tri-continuous coreshell structure, the polar glycerol domains of appreciable size can be considered as micellar [48].

SAXS and AFM were used to study orientation patterns of two polyphilic LC confined to cylindrical pores of anodic aluminum oxide (AAO) by Ungar et al. AFM of LC-containing AAO fracture surfaces revealed that the columns of the planar anchoring LC formed concentric circles in the plane normal to the pore axis near the AAO wall. Toward the pore center, the circles become anisometric consisting of two straight segments and two semicircles. It has

been concluded that the main cause of the two new LC configurations, the "racetrack" and the "log pile", and of their difference from those of confined nematic LC, is the very high splay energy and low bend energy of columnar phases [49]. Also axial orientation of discotic columnar liquid crystals in nanopores of inorganic templates has been studied by Ungar et al. The role of the rigidity of the LC columns in achieving orientation in nanopores has been evaluated. Using 2-D X-ray diffraction, AFM, grazing incidence diffraction, and polarized microscopy, the orientation of the columns with increasing column rigidity have been observed. Furthermore, the highly detailed AFM images revealed that the columns spiral down to the very center of the disclination and that there is no amorphous or misaligned region at the core [50].

Soberats et al. demonstrated switching of ionic conductivities in wedge-shaped liquid crystalline ammonium salts. X-ray experiments revealed a highly ordered packing of the ions in the columnar phase, which prevents the ion transport [51].

Itoh et al. investigated the anisotropic shear viscosity of LCs confined in nanometersized gap widths subject to both shearing and photo alignment. The viscosity and order parameter of a LC confined and sheared in the gap range of 500 nm down to a few nm have been measured simultaneously by using Fiber Wobbling Method (FWM) apparatus that combines a highly sensitive viscosity measurement and a sensitive birefringence measurement. It has been suggested that current studies can significantly contribute to a better understanding of the confined LCs utilized for lubrication [52].

Carter et al. have reported synthetic six-tailed mimics of the bacterial glycolipid Lipid A that trigger changes in the internal ordering of water-dispersed LC microdroplets at ultralow concentrations. Surface-pressure measurements and SAXS experiments revealed that the synthetic amphiphiles mimic key aspects of the self-assembly of Lipid A at aqueous interfaces and in solution. These and other results suggested that these amphiphiles trigger orientational transitions at ultralow concentrations through a unique mechanism that is similar to that of Lipid A and involves formation of inverted self-associated nanostructures at topological defects in the LC droplets [53].

Sano et al. have studies the realignment process of azobenzene-containing LC block copolymer films in detail by time-resolved GI-X-ray scattering measurements using a synchrotron beam. Decisive evidence was obtained to support the subdomain rotation mechanism in the realignment

process [54]. Table 1 presents an overview of the literature being studied.

4. CONCLUSIONS

LCs had been proven to be the most intriguing and interesting candidates for their application in certain fields such as liquid crystal displays (LCDs), LC Thermometers, hyperspectral imaging and various other industrial applications. As research into this field develops and as new applications are discovered, LCs will play an important role in modern technology. Most LC compounds show polymorphism and hence the detection of their precise phases is very vital for deciding their fate in different applications. Various characterization techniques e.g. XRD, DSC and POM have proven quite promising in determination of liquid crystalline properties of numerous compounds. Many new techniques have been also introduced to analyze different aspects of LCs and hence as new applications of these compounds would be considered, different new characterizing techniques will come to light in future.

Table 1. Characterizing techniques used for LCs.

Type of liquid crystal	Properties being studied	Characterization technique	Ref. #
Organosiloxane mesogens	Phase behavior, morphologies	POM, DSC, SAXS	14
Biphenylacetylene LC	Phase behavior, morphologies	POM, DSC, XRD, CD	15
Bent rod shaped molecules	Structural properties in solution, solid and fiber state	CD, DSC, XRD, TEM	16
Discotic liquid crystalline block copolymers	Morphlogy, molecular structure, thermal analysis	POM, TEM, XRD, DSC	17
Discotic azo compounds	Thermal and liquid crystalline properties photoisomerization capacity	POM, DSC, XRD, ^{1}H NMR	18
Mesomorphic azo compounds	Phase transitions transition temperatures	POM, DSC	19
p-alkoxybenzoic acids	Phase behavior	Melting point techniques, DSC, POM	20
D-(+)-Glucose	Liquid crystal formation	DSC, TGA, POM	21
Bolaamphiphiles with swallow tail lateral chains	Self-assembly in thermotropic liquid crystalline phases	DSC, POM, XRD	22
T-shaped bolaamphiphiles	Influence of spacer length and position of the spacer on the self-assembly in liquidcrystalline phases	POM, DSC, XRD	23
LC side chain diblock copolymer	Structural analysis	SAXS, TEM	24
Triphenylene-based side chain liquid crystalline block copolymers	Microphase structures	DSC, POM, Variable SAXS/WAXS	25
Liquid-crystalline brush copolymers	Phase behavior	SAXS, DSC, WAXS, TEM	26
Diblock copolymers	Strutural features and hierarchical segment selfassembly	DSC, XRD, POM	27
PEO-b-PMA(Az) block copolymer	Phase behavior	SAXS, TEM	28
poly(methacrylate) withliquid crystal side chains	Aggregation states	DSC, POM, WAXD	29
LC-based sensor	Birefringent properties	POM, SEM	30
Cross-linked liquid crystal polymer films	Thermodynamic properties, mesomorphic properties	SEM, DSC, POM	31
Polymer-dispersed liquid crystal/graphene oxide nanocomposites	Photomechanical response of the nanocomposite film	POM, SEM	32

Polylactide-based liquid crystalline brush-like block copolymers	Morphlogy and phase behavior	DSC, TEM, SAXS, WAXS	33
ABA triblock B5 Cin copolymers	Morphology and photo-crosslinking, thermal properties	^{1}H NMR, FT IR, DSC, SAXS, WAXS, TEM	34
2,4,6-tris(thiophene-2-yl)-1,3,5-triazines based molecules	Crystal and molecular structure liquid-crystalline properties	X-ray, Molecular Dynamics simulation, Cyclic voltammetry, UV/Vis, POM	35
Poly(2,5-bis{[6-(4-methoxy-40-oxyazobenzene)hexyl]-oxycarbonyl}styrene) based LC polymer	Phase structures and transitions	DSC,POM, WAXS	3
LCP	Photoresponsive behavior, thermal behavior	POM with *in situ* UV irradiation, DSC, UV-/Vis	3
Naturally occurring amphotropic liquidcrystals	Physical properties	POM,DSC, Mid- and near-infrared photoacoustic spectroscopy	38
Wedge-shaped onium salts	Mesomorphic properties	SAXS, WAXS, DSC	39
ABA triblock copolymer	Phase transition, density, molecular weight dependence, structural parameters, microphase behavior	WAXD, SAXS, TEM	40
Biaxial nematic liquid crystal phases	Twist viscosity and the alignment angle	Molecular Dynamics simulation	41
Rod-like mesogens with three- and four-ring core	Structural characterizations	XRD and ^{13}C NMR	42
Phenyl ring core-based thiophene mesogens	Mesophase transitions molecular shape mesophase characteristics	HOPM, DSC, ^{13}C NMR	43
Thiophene LCs	Structural aspect and phase transition behavior	FT-IR, 2D NMR, POM, DSC	44
Pyridine based mesogens	mesophase characterization	Solid state NMR, FTIR, POM, 2D-SLF	45
Liquid crystal tri-block copolymer	Structural properties, mechanical strength and photoredsponsive behavior	SAXS, TEM, DMA, DSC, POM, photocontraction tests	46
Liquid crystalline block copolymer matrix	Faceted grain growth of hexagonal cylinder domains	AFM, GISAXS, DSC	47
T-shaped polyphilic molecules	Thermotropic LC phases	POM, DSC, XRD, GISAXS	48
Polyphilic LCs	Composite mesostructures	SAXS, AFM, XRD	49
Discotic columnar liquid crystals in nanopores of inorganic template	Orientation behavior in nanoconfiments	SAXS, AFM, GISAXS	50
Benzenammonium columnar LCs	Phase transition	XRD, GISAXS, GIWAXS	51
4-Cyano-42 -pentylbiphenyl	Viscosities and refractive indexes	FWM	52
Water-dispersed LC microdroplets	Optical transitions	POM, Forward light scattering (FSC), SAXS	53
Azobenzene-containing liquid crystalline block copolymer films	Realignment process of LC films	GI-SAXS, AFM, Polarized UV-Vis	54

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