## **Preparation, Characterization and Applications of Liquid Crystals: A Review**

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Abstract: During the last few decades one of the sources of vast attention for researchers has been Liquid crystals (LCs). LCs have unique properties and wide applicability. As LCs design and property vary with linking groups and terminal groups, they can be designed in a large number of ways and hence their preparation becomes important. Not all properly designed compounds show LC properties. Thus their characterization is also crucial, so as their applications. In this review article, the aim is to focus on the different preparation methods of LCs, various characterization techniques, and recent advances in the application of different types of liquid crystals.

*Keywords:*liquid crystals, nano-structures, cross-coupling, drug delivery

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#### I. Introduction

Liquid crystal was first revealed for cholesterol by F. Reinitzer in 1888. After all that years, the examination of liquid crystal materials is still of great interest. Starting from the first stages, where LCs were on focus for display applications; for recent times the focus has shifted towards even for biological processes due to their molecular organizations. Liquid crystalline environments exist in living cells [1]. In 1888, Friedrich Reinitzer was figuring out the correct formula for cholesterol when he found the fact that the matter appeared to have more than one melting point, two melting points to be exact. Solid crystal melted into a hazy liquid at 145.5°C which existed until 178.5 °C where the haziness went suddenly, and a clear transparent liquid is formed. The liquid crystals are intermediate between the liquid amorphous state and the crystalline solidstateand are called mesophase [2]. It is mostly named a mesomorphic state [3]. The liquid crystal is a substance that is thermodynamically situated in the middle of the isotropic liquid and the crystalline phase. The order of a crystalstill partly holds and they also have the flow like properties of liquids [4]. Aquarter state of matter following solid, liquid, and gas is what liquid crystal can be deliberated. Liquid-crystal phases, as their name suggests, be existent between the predictable crystal phase and the liquid phase. Typically, liquid-crystal molecules keep rod-like structures or disc-like anisotropic structures. The distinctive characteristic of liquid crystals is the propensity of the molecules to support themselves with long-range directional order [5].

The liquid crystals can flow like liquids, but the organization of the molecules is like crystals. In these two generic classes of liquid crystals: which are changes are driven by thermal processes, called thermotropic liquid crystals & lyotropic system are strongly influenced by solvents and many thermotropic liquid crystals exhibit a diversity of stages as the temperature of the system is altered. For example, as the temperature is increased a specific type of LC particle may show numerous nematic and smecticphases.

Thermotropic liquid crystal materials are composed of two parts, a specific molecular structure, which, namely referred to as side chain and the central core. Here the rigid body is the core part that carries shape anisotropy to the molecule, and mobility is provided by the side chain part in a flexible region [6].

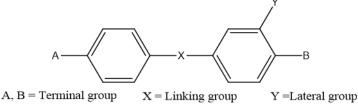


Fig. 1. The general structure of a liquid crystal.

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Therefore, they keep anisotropic physical nature for example their elastic behavior, dielectric constant, refractive index, or viscosity. But while being partly ordered, LCs also show flow properties like a liquid; they are accordingly anisotropic fluids. This anisotropic nature resemblesmagnetic and electrical properties. Thus by applying weak electric or magnetic fields, the orientation behavior of the LC molecules could be monitoredeffectively. The optical and mechanical properties of the medium can be changed by changing the orientation of LC molecules. These factors are important for applying LCs in display devices, such as calculators, digital watches, TVs, and thermometers [7-10].

#### II. Classification of Liquid Crystals

Liquid crystalline compounds are differentiated into two types: thermotropic and lyotropic [8]. The liquid crystals which change their phase upon heating or cooling are thermotropic. The mesophase is called enantiotropic when the phase is obtained upon both heating and cooling. When added in a suitable solvent lyotropic phases are formed and the appearance of the mesophase is dependent on both concentration and temperature.

#### (i) Thermotropic Liquid Crystals

Thermotropic LCs are the ones that are extensively recognized due to their application in televisions, laptops, mobile phones, and tablet displays. According to the structural features, thermotropic liquid crystals are subdivided into two main groups. The rod-like molecules are calamitic [11] and the disc-like molecules are discotic [12].

#### (ii) Lyotropic Liquid Crystals

Lyotropic substances are strongly birefringent. Mesophase in binary systems of amphiphiles and water may be classified as (a) the neat phase (b) the middle phase (c) the viscous isotropic phase (d) the isotropic phase and (e) the inverse phase. Lyotropic substances with other solvents are also studied. Mesophases of lyotropic liquid crystals are also affected by temperature changes. Lyotropic liquid crystals of hydrated phospholipids are studied as a model medium for studies of antimicrobial agent activity.

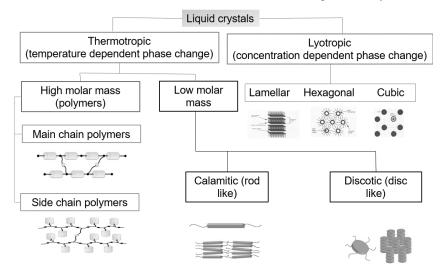


Fig. 2.Classification of liquid crystals.

#### III. Methods for Preparing Liquid Crystals

### (i) Synthetic approaches to prepare calamitic liquid crystals

Hydroxyl (-OH) and carboxylic acid (-COOH) functionalized aromatic rings can be esterified to provide ester bridged liquid crystals. Traditionally, the acid group was converted into acid chloride derivate using oxalyl chloride or thionyl chloride, followed by the reaction with hydroxyl functionalized group in presence of a base. A new simple method has been developed where the esterification reaction is carried using 4-(N,N-dimethylamino)pyridine (DMAP) base and N,N-dicyclohexylcarbodiimide (DCC) catalyst at room temperature [13].

A long, linear architecture favors liquid crystal formation and the simplest structures are 4,4'disubstituted.Multiaryls are difficult to synthesize directly. One solution for this problem is the Palladiumcatalyzed cross-couplingreactions which allow the formation of a carbon-carbon bond between, for example, twobenzene rings or a benzene ring and an acetylene unit. Their development has enabled thesynthesis of a wide range of liquid crystals that would otherwise have been extremelydifficult or even impossible to make.

The presence of pyrimidine ringsin a compound enhances liquid crystal properties and introduces in some cases nematic and smecticC phases. Coupling methodology provides an efficient route for the synthesis of phenylpyrimidine and fluorophenyl pyrimidines.Palladium-catalyzed cross-coupling reactions are also used for inserting important terminal groups required for liquid crystal structure.

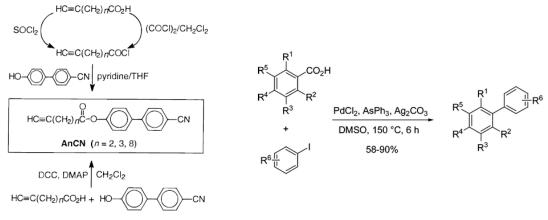


Fig. 3.Synthesis of calamitic liquid crystals by esterification and cross-coupling.

#### (ii) Synthetic approaches toprepare chiral liquid crystals

To obtain the chiral nematic  $(N^*)$  phase and the ferroelectric chiral smectic C (SmC\*) phase, chiral groups are required. Chirality is introduced into a liquid crystal system through a terminal chain in most cases. The chiral group can be introduced into a pro-mesogenic unit by using the Mitsunobu methodology (generally known as a DEAD reaction) [14]. Enzymes can be used for the synthesis of pure chiral units that can be used in the preparation of chiral liquid crystals.

$$H_{3} \xrightarrow{F} OH$$

$$H_{3} \xrightarrow{F} OH$$

$$H_{0} \xrightarrow{F} Br$$

$$H_{0} \xrightarrow{F} Br$$

$$C_{6}H_{13} \xrightarrow{F} O \xrightarrow{F} O \xrightarrow{F} Br$$

$$C_{6}H_{13} \xrightarrow{F} O \xrightarrow{F} O \xrightarrow{F} O \xrightarrow{F} Br$$

$$C_{6}H_{11} \xrightarrow{F} B(OH)_{2} \xrightarrow{F} O \xrightarrow{F}$$

Fig. 4.Synthesis of chiral liquid crystals by DEAD method and cross-coupling.

#### (iii) Synthetic approaches topreparediscotic liquid crystals

Discotic liquid crystals are mostly based on benzene, triphenylene truxene core structures. Discotic liquid crystals can be synthesized using Steglich esterification reaction and cross-coupling reactions using Palladium as a catalyst [15].

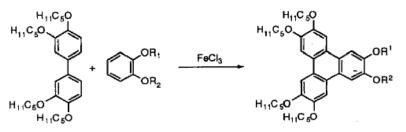


Fig. 5.Synthesis of discotic liquid crystals by coupling method.

#### (iv) Synthetic approaches to prepare polymeric liquid crystals

Kevlar is a simple nylon-type polymer that is synthesized by heating a dicarboxylic acid and a diamine. Many polymers are prepared by transesterification. The various types of poly(acrylates) are prepared by radicalpolymerization process by using an initiator (*e.g.*, AIBN or hydrogen peroxide) underthermal conditions or by photo-induction [16].

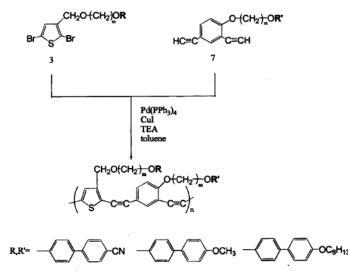


Fig. 6.Synthesis of polymeric liquid crystals.

#### (v) Synthetic approaches topreparelyotropic liquid crystals

Spray drying, probe sonication,bottom-up approach,top-down approach, heat treatment are some methods for preparing lyotropic liquid crystals and their nanoparticles[17].

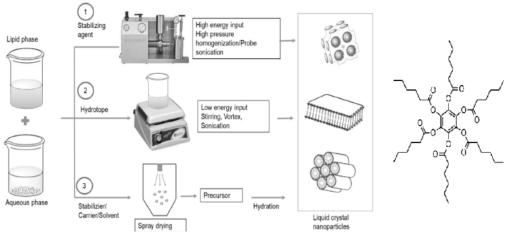


Fig. 7.Synthesis of lyotropic liquid crystals by top-down (1), bottom-up (2), and spray drying (3) techniques.

#### IV. Characterization of Liquid Crystals

To characterize liquid crystals different techniques can be used. Factors to be considered are the positional order of the molecules, as well as orientational order and also bond orientational order. By utilizing these parameters not only the proper phases but also the interactions within molecules can be identified. PolarizedOpticalMicroscopy(POM) is a useful tool for identifying phase transitions and phases of liquid crystals. DifferentialScanningCalorimetry(DSC), X-raydiffraction (XRD), and some other tools are used too for the characterization of liquid crystals.

#### (i) Polarized Optical Microscopy (POM)

In a polarized optical microscope, plane-polarized light is used. If the material under study is isotropic, i. e., the direction of the molecules are parallel or perpendicular to the director, the visual will appear black under the crossed polarizers. But liquid crystals are anisotropic, and they appear bright under crossed polarizers

due to the rotation of polarized light. Generally, a stepwise increase in temperature (typically 5°C in every step with a heating rate of  $1^{\circ}C/min$ ) is used to encourage phase transitions.

#### (ii) Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) is a complementary toolfor determining the phase transitions in liquid crystals. As a function of temperature, an increase in sample and reference temperature and subsequent heat is recorded, and then heat difference is calculated by this thermoanalytical technique. Maintaining the same temperature thoroughly for the sample and reference is required. Generally, the temperature of the sample holder as a function of time increase linearly, and DSC analysis is designed for this temperature program.

#### (iii) X-ray diffraction (XRD)

For the characterization of mesophases, X-ray diffraction (XRD) can be used that gives detailed information about the structure and long-range order in liquid crystals. Three-dimensional arrangements can be identified by Small-angle X-ray scattering (SAXD) which is used to identify different groups in the formulation liquid crystals.

#### (iv) Grazing-incidence X-ray diffraction (GIXD)

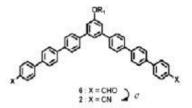
Grazing-incidence X-ray diffraction (GIXD) is used to find out the nanostructures present in the thin films.

#### (v) Transmission electron microscopy (TEM)

Transmission electron microscopy (TEM) is used to analyze the morphologies of different discotic liquid crystal phases.

Particle size distribution, entrapment efficiency is measured for the liquid crystals which are used in drug delivery. For these measurements, dynamic light scattering, Cryogenic transmission electron microscopy (Cryo-TEM), ultra-filtration techniques are utilized.

Kim et al. utilized circular dichroism(CD) and transmission electron microscopy (TEM) to investigate the self-assembly bent-shaped molecules in an aqueous solution. The observation was that the aromatic bent rod-like molecules were self-assembled at both ends in an aqueous solution while the compounds contain nitrile groups, and with no nitrile groups, there were no apparent aggregations [18]. Vega et al. found different types of smectic phases for the molecules having chiral centers. For characterization, they used polarized optical microscopy (POM), differential scanning calorimetry (DSC), and X-ray diffraction techniques. The result of XRD was further confirmed usingcircular dichroism(CD) [19].



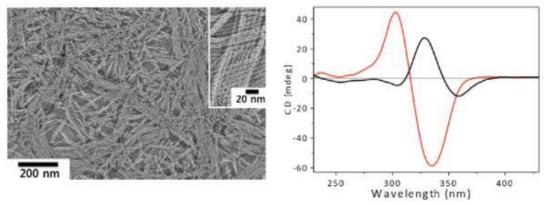


Fig. 8.TEM images and CD spectra of bent-core liquid crystals. Copyright (2009) John Wiley & Sons.

Organosiloxane mesogens were synthesized and characterized by Roberts et al. The mesogens undergo SmA-SmC phase transitions investigated byoptical tilt angle measurements which are combined with small-angle X-ray scattering (SAXS). Information about the high translational and low orientational order of the compounds were also obtained by SAXS measurements [20].

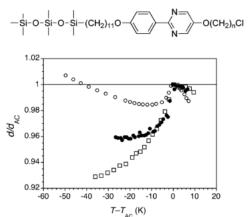


Fig. 9.Relative smectic layer spacing  $d/d_{AC}$  versus reduced temperature T-  $T_{AC}$  obtained using measured by SAXS. Copyright (2010) American Chemical Society, Journal of American Chemical Society.

Westphal et al. synthesized discotic azo compounds and characterized the phases and identified the phase transitions using POM, TEM. They also utilized XRD and photoisomerization for the characterization of columnar discotic phases [21]. Stillings et al. also used the same techniques for the identification and characterization of discotic block copolymers that show columnar phases [22].

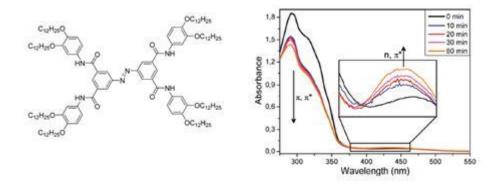
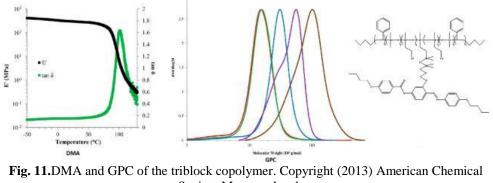
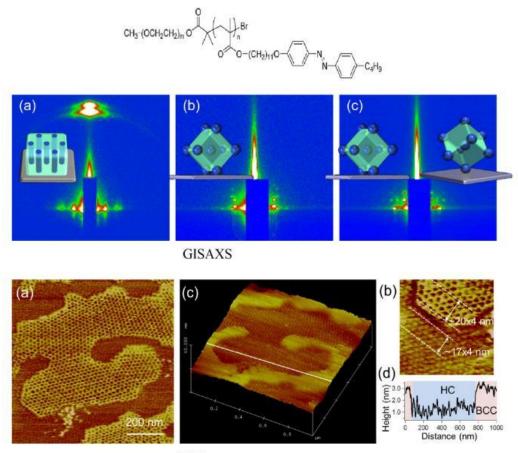


Fig. 10.Photoisomerization of the compound in chloroformsolution at room temperature. Copyright (2010) American Chemical Society,Macromolecules.

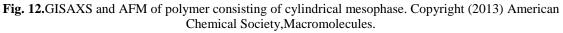
DSC, POM, and also Dynamic Mechanical Analysis(DMA), SAXS, TEM, and photocontraction tests have been carried out by Petr et al. for the synthesis of thermoplastic siloxane elastomer which can be used for functional actuators [23]. Gel Permeation Chromatography (GPC) was used to determine the molecular weight of the polymers.



Komsura et al. used *in situ* Atomic Force Microscopy (AFM) to confirm the formation of a cylindrical mesophase of a block copolymer. Along with X-Ray diffraction techniques, Grazing Incidence Small-Angle X-ray Scattering (GISAXS) analysis was used to find out order-order transition change. Self-assembled nanostructures for liquid crystalline materials can be visualized using AFM [24].

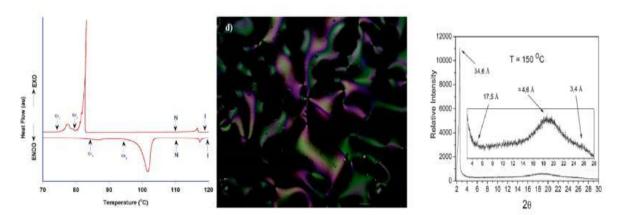


AFM



FiberWobblingMethod (FWM) is a tool that combinesbirefringencemeasurement and viscositymeasurement. This apparatus has high sensitivity to viscosity andgood sensitivity to birefringence. This tool was used by Itoh et al. to investigate the shear viscosity in nanometer-sized gap widths. These studies help to use the LCs for lubrication [25].

Wang et al. studied the nematic phase behavior of cyanobiphenyl compounds having multifluorophenyloxy terminus. DSC and POM were utilized to identify the nematic phases of the materials [26].Reddy et al. have studied three- and a four-ring core containing mesogens, thiophene having phenyl ring cores [27,28]. Structural elucidations were done byusing FourierTransform-InfraRed spectroscopy(FT-IR), XRD, and <sup>13</sup>CNMRspectroscopy.



**Fig. 13.**DSC thermogram, POM image, and XRD pattern for liquid crystalline materials. Copyright (2020) Taylor and Francis, Liquid Crystals. Copyright (2013) Taylor and Francis, Molecular Crystal Liquid Crystal. A table for all the characterization methods is summarized below for different liquid crystalline materials.

Characterization Techniques	Properties investigated	Types of LCs	
DSC	Phase transition temperatures	All thermotropic mesomorphic compounds. e.g., mesomorphic azo compounds, t-shaped polyphilic molecules, main chain, and side-chain liquid crystal polymers	
РОМ	Textures for different liquid crystal phases	All compounds that have mesophases.e.g., organosiloxane mesogens, biphenylacetylene LCs, bent rod-shapedmolecules, <i>p</i> -alkoxybenzoic acids, different polymeric and lyotropic compounds	
XRD	Orientation and structural details of molecules that constitute a mesogen. Layer by layer constitution, tilt angles can also be known.	Acetylene LCs having biphenyl cores, rod-shaped molecules having bent structure, discotic azo compounds, bolaamphiphiles having a shape like 't', diblock copolymers, polyphilic LCs, benzenammonium columnarLCs	
SAXS	Structural analysis like morphologies	LC side chain diblock copolymer,organosiloxane mesogens, liquid-crystalline brush copolymers, PEO-b-PMA(Az) block copolymer, ABA triblock B5 C in copolymers, wedge-shaped onium salts	
CD	Structural properties in solutions	Bent rod-shaped molecules, biphenylacetylene LC	
TEM	Morphologies in the fiber state	Liquid crystalline blockcopolymers which are discotic,rod-shaped molecules having bent structure, a liquid crystalline diblock copolymer having side chain and brush copolymers, ABA triblock copolymer	
WAXS	Microphase structures	Block copolymers which are triphenylene-based and are side-chainliquid crystalline, brush copolymers having liquid crystallinity, polylactide based liquid crystalline brush-like block copolymers, onium salts which are wedge-shaped	
WAXD	Molecular weight dependence and aggregation states	Poly(methacrylate) with liquid crystal side chains, ABA triblock copolymer	
SEM	Mesomorphic properties, thermodynamic properties, the photomechanical response of the nanocomposite film	polymer films of cross-linked liquid crystal, liquid crystal/graphene oxide nanocomposites dispersed in a polymer	
Cyclic voltammetry and Molecular Dynamics simulation	Twist viscosity, the alignment angle, molecular and crystal structure of liquid crystals	2,4,6-tris(thiophene-2-yl)-1,3,5-triazines based molecules, biaxial nematic liquid crystal phases	
UV/Vis	Photoresponsive behavior	Liquid crystal polymers, 2,4,6-tris(thiophene-2-yl)- 1,3,5-triazines based molecules	
DMA	Mechanical strength and photoresponsive behavior	Liquid crystal tri-block copolymer	
GISAXS	Orientation behavior in nanoconfinementshexagonal cylinder domains by faceted grain growth, thermotropic LCs	Inorganic template of discotic columnar LCs in nanopores,polyphilic molecules having 't' shape, columnar liquid crystals of benzenammonium, block copolymer filmsLCs containing azobenzene	
AFM	Liquid crystal films realignment process	Block copolymer films LCs containing azobenzene, an inorganic template of discotic columnar LCs in nanopores	
FWM	Refractive indexes and viscosities	4-Cyano-4,2 -pentylbiphenyl	

Table 1. Characterization technic	aues of different type	of liquid orvetale
Table 1: Characterization technic	jues of afferent types	s of inquita crystals

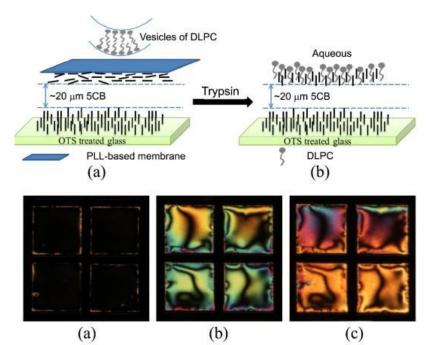
#### Applications of Liquid Crystals

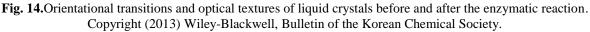
The most common applications of liquid crystals have been their uses in optical devices. Optical uses of the liquid crystals include display applications, use in thermometers, use as optical modulators, and use of ferroelectric liquid crystals in the optical data processing. Polymer dispersed liquid crystals have been used in smart windows, eye projection viewers, and so on. The recent development of liquid crystals includes their uses in therapeutics for drug delivery, introducing them in nanomaterial.

V.

#### (i) Liquid crystals in sensors

Zhang et al. introduced optical sensors that were liquid crystal based and used them for imaging trypsin activity. The activity was observed at the interfaces between aqueous and thermotropic liquid crystal phases. The progress can be understood from the change in liquid crystal texture [29].





#### (ii) Liquid crystals for organic electronics and energy conversion applications

Transistors and organic photovoltaic cells have been built efficiently by using the self-assembly feature of liquid crystals [30,31]. Bent core nematics have a high flexoelectric coefficient that can be used for mechanoelectrical energy conservation [32].

Hanna et al. functionalized some molecules with alkyl chains to give smectic phases that can be optimized for the application as a field-effect transistor. At the proper temperature where the material is in the smectic phase, it is dissolved into an appropriate solvent and achieved long-range order and high uniformly organized films [33].

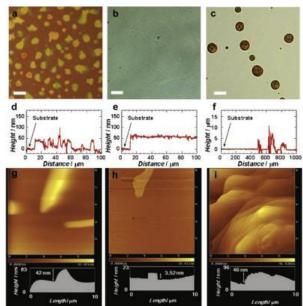


Fig. 15.Doubly alkyl terminated polycrystalline films of a terthiophenewhich is spin-coated from isotropic solutions. Copyright (2013) John Wiley & Sons, Wiley Online Library.

# (iii) Template formation of liquid crystal to create nanostructured materials (ordering nano- and microparticles)

Liquid crystals can be used as templates for nanoporous silica production that can be used in catalysis via cubic or hexagonal lyotropic phases [34]. Thermotropic liquid crystals have been used to prepare a solid by polymerizing a material inside LC or by polymerization of the LC material itself, and thus can be used as a template too [35]. Several self-organizable liquid crystals have nanoreactors like properties and can be used for nanoparticle synthesis. Semiconductor nanorods can be synthesized by materials having lyotropic columnar phases [36].Single and multiwall carbon nanotubes have been dispersed in lyotropic and thermotropic liquid crystals and thus can act as dopants [37].

#### (iv) Thermotropic liquid crystals in composite materials as a functional additive

Cholesteric droplets forming emulsions have been used for tunable laser applications. Cholesteric or any other chiral liquid crystals can be used for mirrorless lasing when it is tuned with a fluorescent dye matching with its reflection band and show visible reflection and it acts like a resonant cavity [38].

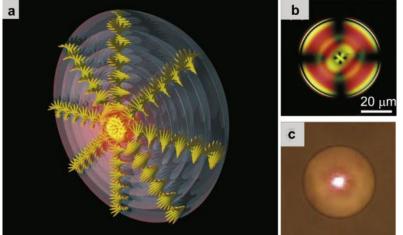


Fig. 16.Mirrorless lasing applications of cholesteric droplets.Copyright (2010) the Nature Publishing Group, Nature Photonics.

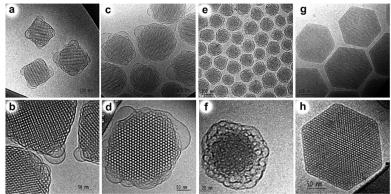
Liquid crystal droplets are utilized for colloidal preparation with low dispersity and are considered as thin shells [39]. Textile fibers can be functionalized by inserting a liquid crystal material in the core. Lagerwall et al. introduced liquid crystal core in polymer sheath fibers having small diameters[40].

#### (v) Liquid crystal elastomers in actuators

A thermotropic liquid crystal phase that has reactive mesogens with weakly cross-linking forms elastomers. These elastomers have many uses that include the use of these elastomers in artificial muscles [41], light polarization [42], heavy weight lifting capacity [43].

#### (vi) Liquid crystals in biotechnology and pharmacology

Lyotropic phases can be formed by amphiphiles inwater which is biocompatible, also biocompatible when made with oil. Ultrasonication method for preparing nanoparticles with these soft matters gives diameters in nanometer (100 nm) range. These are referred to as hexosomes and cubosomes, and can be loaded with drugs for oral delivery [44].



**Fig. 17.**Cryo-TEM images of soft nanoparticles containing lyotropic liquid crystal phases used in drug delivery. Copyright (2009) Elsevier, Advances in Colloid and Interface Science.

Cubic phases are more bioadhesive in nature so that they can suitably use in topical & mucosal depositions and delivery of different drugs by dosage forms.Controlled release of solubilized active compounds is the most widely held application pursued by cubosomes investigators. Brilliant reviews happen of attempted delivery applications for pharmaceutically active compounds that have been solubilized in bulk cubosomes and liquid crystals.Some liquid crystals have curved like the structure of lipid membranes that are used for solubilization, encapsulation, and distribution of medicines to disease zones within the body.Since the microbicidal stuff of monoglycerides can be used in intravaginal therapy of sexually transferred diseases caused due to viruses (e.g. HSV, HIV) or due to bacterias (e.g. Neisseria genorrticae). Because of the similarity between the structure of the stratum corneum and the cubic phase structure, it is judicious to suppose the construction of a mixture of stratum corneum lipids with cubosomalmonoolein.Liquid crystals have useful applications in the field of 'lipidomics' where cell signaling and trafficking are investigated [45].

#### VI. Conclusions

The field of research with liquid crystals is very broad. There are many designs for liquid crystals that make them available for many important applications. Thus, the design and synthesis of liquid crystals are of great interest. In this review, the design and synthesis of liquid crystals having different bridging groups and terminal groups have been discussed. All the methods are well known and can be implemented for synthesis. As not always the properly designed materials show LC properties, so the characterization must be done. In this article, an overview of all the characterization techniques (from mesophase structure to nanostructures) of thermotropic and lyotropic liquid crystals has been given. The interesting behaviors of different LCs make them available for many applications. The uses of LCs from optoelectronics to living cells have also been reviewed. This may provide interest to the researchers in designing new liquid crystals and applying them.

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