

Synopsis

of the thesis entitled

Old Title: Synthesis and study of heterocyclic compounds containing
Thiadiazole and Oxadiazole moiety

**New Title: Synthesis, Characterization and Applications
of Cholesterol/Heterocyclic based Mesogenic
Compounds**

Submitted to

THE MAHARAJA SAYAJIRAO UNIVERSITY OF BARODA

For the Degree of

Doctor of Philosophy

In

Applied Chemistry

By:

Bairwa Sagar Kailashchand

Under the Supervision of

Prof. R. C. Tandel



सत्यं शिवं सुन्दरम्

**Applied Chemistry Department
Faculty of Technology and Engineering
The Maharaja Sayajirao University of Baroda,
Vadodara-390001, Gujarat, INDIA**

December-2023

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Synthesis, Characterization and Applications of Cholesterol/Heterocyclic based Mesogenic Compounds

Liquid crystals have been a fascinating area of research because they combine the potentials of both, liquids and crystals to yield materials with unique characteristics and a wide range of applications. Within the numerous branches of liquid crystals studies, this study particularly focuses on the synthesis, Characterization and applications of cholesterol based heterocyclic and other liquid crystalline derivatives. Because of interesting phase transition, responsiveness to the external stimuli and ability to exhibit distinct mesophases with ordered but fluid molecular arrangement, liquid crystalline compounds have attracted a lot of interest. These compounds provide a link between the liquid states' intrinsic flexibility and the rigidity of crystalline structures, opening new opportunities for tailored material design and technological advancement.

The core of this work is to design new molecular structures and synthesis of liquid crystalline compounds. Molecular designing techniques, novel synthetic pathways and the factors influencing phase behaviour can all be investigated to gain a thorough grasp of the chemical underpinnings of liquid crystalline phenomena. Most of the study was conducted by synthesising a homologous series of the compounds by varying the terminal chain length. Synthesising such systems allows for systematic investigations of the influence of molecular size on phase transitions and liquid crystalline behaviour and also enables researchers to establish clear structure-property relationships. By exploring the synthesis, the study aims to contribute to the expanding body of knowledge in liquid crystal chemistry by providing insights on the ways in which particular liquid crystalline properties can be achieved by modifying molecular structures.

The use of Characterization techniques is essential for understanding the complex behaviours of liquid crystalline compounds. Advanced analytical methods that provide windows into the textural, photophysical, thermal and structural properties of these materials include spectroscopy, microscopy, optical and thermal analysis. In addition to confirming the synthesis's success, the Characterization procedure offers a sophisticated understanding of the dynamic behaviour of liquid crystalline phases and shed information on their potential applications.

Applications for liquid crystalline compounds are numerous and extend beyond the laboratory. Liquid crystalline materials' adaptability provides solutions to modern problems, ranging from advancements in display technologies to inventive applications in drugs delivery systems, actuators and sensors. Also, liquid crystals have application ranging from optical devices, SLMs, electro-optical devices, biomedical applications, smart windows, cosmetics, etc. The objective of this study is to investigate and explain the various practical applications that results from the unique properties of liquid crystalline materials, with a focus on how these uses may influences the development of new technology[1–5].

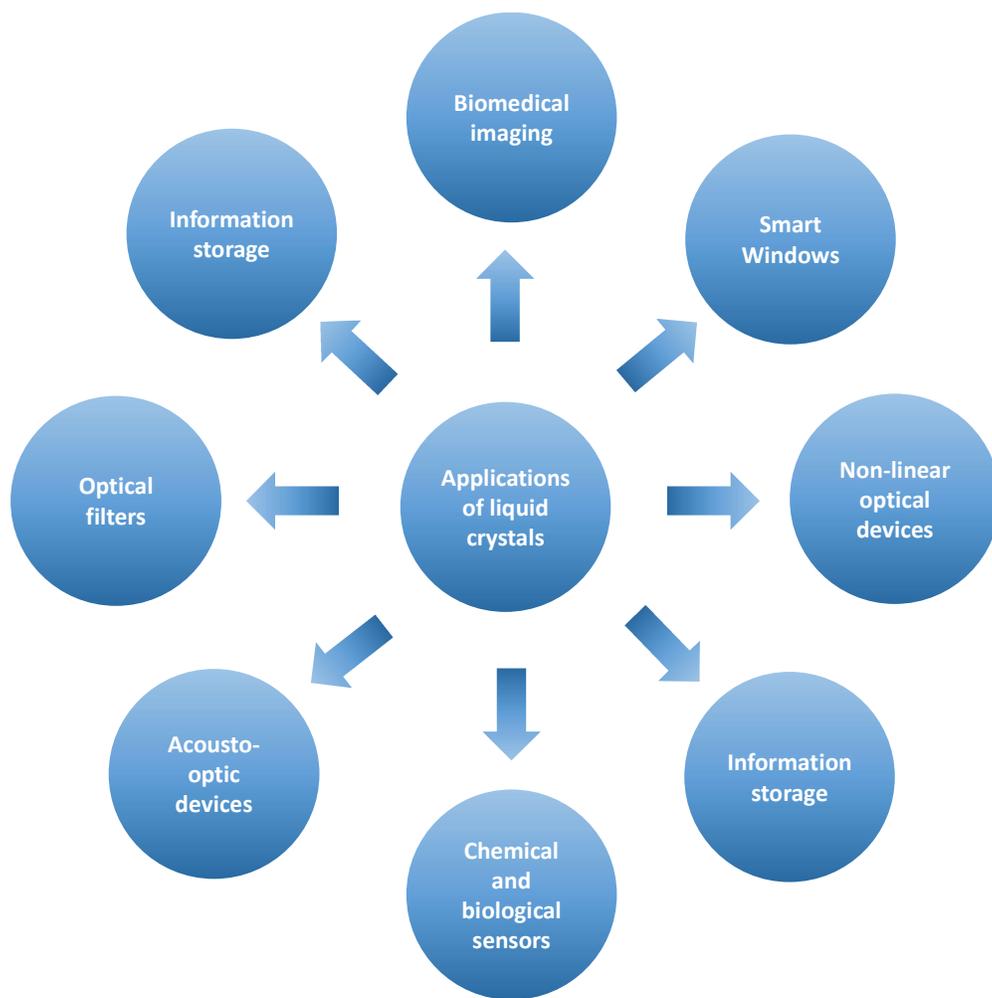


Figure 1. Some applications of liquid crystals

In conclusion, this study of liquid crystalline compounds encompasses a multifaceted examination that includes designing, synthesis, Characterization and applications. As we navigate through the details of these materials, we embark on journey that not only advances our scientific understanding but also holds the promise of contributing to technological

breakthroughs with far reaching implications. Through these studies, we aim to contribute to the growing body of knowledge surrounding liquid crystalline compounds, opening the way for more advancement and uses in this fascinating area of research.

Summary of the research work:

The thesis will be structured into seven chapters as follows:

Chapter 1	Introduction
Chapter 2	Cholesterol based mesogenic Schiff's base derivatives
Chapter 3	Cholesterol functionalized azo-based biphenyl liquid crystals
Chapter 4	Mesogenic Schiff's base derived from thiadiazole moiety
Chapter 5	1,3,4-oxadiazole based unsymmetrical liquid crystals
Chapter 6	Biologically active cholesterol-based thiazole derivatives
Chapter 7	Summary, Conclusion and future aspects

CHAPTER 1: Introduction

In this section of the thesis, an overview is presented, offering a general introduction, a concise historical context and essential aspects of liquid crystal[6]. This chapter discussed the different types of phases of liquid crystals.

Liquid crystal is a state of matter which has properties between those of anisotropic crystal and isotropic liquid. Liquid crystals were first discovered serendipitously by an Austrian botanist and chemist Friedrich Reinitzer (1857–1927) around the end of 1800s at the Karl Ferdinands Universität, while studying the cholesterol derivatives[7]. He found that the cholesteryl benzoate exhibited not one, but two different melting points. German physicist Otto Lehmann (1855–1922) at Aachen University[8] was one of the pioneers who combined a POM with a hot stage, to confirm the unusual phenomenon of two melting points. He was the first to coin the term '*flüssige krystalle*' or, '*flowing crystals*' because of the anisotropic properties of the liquid crystals. Thus, these substances are now known as '*liquid crystals*.'

Liquid crystals are generally classified as thermotropic and lyotropic (as shown in Fig. 2)[9]. In thermotropic liquid crystals, one can arrive at the liquid crystalline state by raising the temperature of solid or lowering the temperatures of liquid whereas in lyotropic liquid crystals important controllable parameter is concentration rather than temperature. Thermotropic liquid crystals are often classified based on their molecular organization and the nature of the phases they exhibit. The main types of thermotropic liquid crystals include:

Nematic Liquid Crystals: The molecules in the nematic phase exhibit long-range orientational order but no positional order. This implies that, while the molecules tend to align in one way, they are free to move in that direction as well.

Smectic Liquid Crystals: Smectic liquid crystals are ordered in both orientation and translation. The molecules in the smectic phase create layers or sheets in which they are coordinated, yet there is still freedom of movement inside each layer. Subphases of smectic phases include Smectic A, Smectic C, and others.

Cholesteric (Chiral Nematic) Liquid Crystals: Cholesteric liquid crystals are a variation on nematic liquid crystals. As one goes through the mesophase, the orientation of the molecules rotates. The presence of chiral (twisting) centres causes this twist.

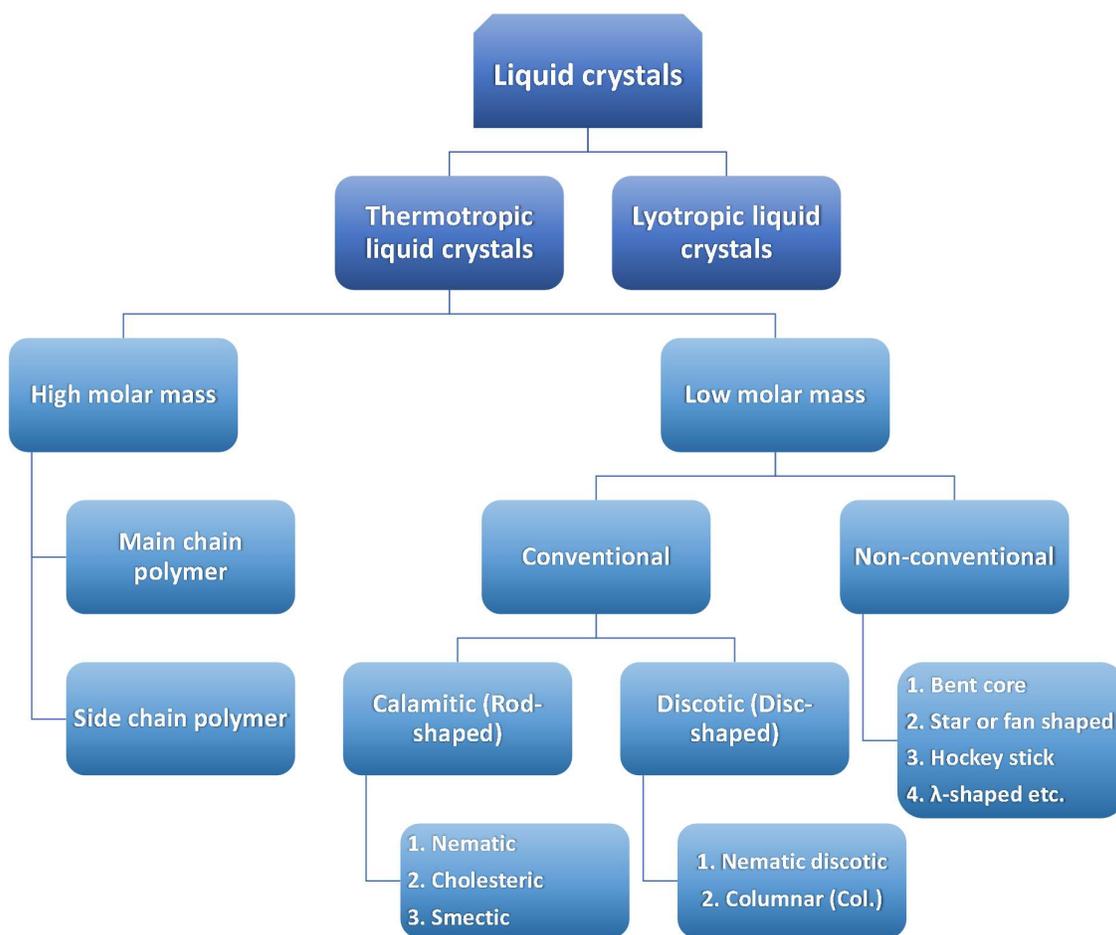


Figure 2. Classification of Liquid Crystals

CHAPTER 2: Cholesterol based mesogenic Schiff's base derivatives

The steroidal derivatives have been found to be extremely good mesogens since their origin. Due to their inherent chirality, they have the potential to induce a wide variety of liquid crystalline phases, including frustrated phases, depending on the structure of the steroidal skeleton and the substituents attached. The frustration of the phases is generally observed only in chiral systems with high enantiomeric excess and strong molecular chirality (short pitch)[10]. In this chapter, two new homologous series of Schiff's base derivatives were synthesised by condensing 4-n-alkoxy aniline with 4-formyl phenyl cholesteryl carbonate and 4-formyl-3-methoxy phenyl cholesteryl carbonate[11]. All the compounds were characterized

using elemental analysis and spectroscopic techniques such as FT-IR, $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$. In order to study the liquid crystalline behaviour of the synthesised compounds, optical texture studies were carried out using polarising optical microscope in heating and cooling cycles. The derivatives showed a variety of mesophases, including chiral nematic (N^*), twist grain boundary-A (TGB_A), smectic A (SmA) and chiral smectic C (SmC^*) phases. The methyl to n-butyl derivatives of Series I show oily streaks texture of cholesteric (chiral nematic) phase. The n-pentyl and n-hexyl derivatives show oily streaks of the N^* phase, while on cooling from isotropic melt, in addition to the cholesteric phase, they also show the SmA mesophase. The n-heptyl to n-decyl derivatives show enantiotropic $\text{SmA-N}^*\text{-Iso}$ transition. The SmA to N^* transition is accompanied by an interceding TGB_A phase. The n-dodecyl derivative shows only enantiotropic SmA phase in both heating and cooling cycles. The higher derivatives from n-tetradecyl to n-octadecyl show enantiotropic $\text{SmC}^*\text{-SmA-Iso}$ transition. Similarly, from the textural observations of series II, variety of mesophase were confirmed, including chiral nematic (N^*), twist grain boundary-A (TGB_A), smectic A (SmA) and chiral smectic C (SmC^*) phases. The mesophase behaviours of both the series were studied and compared well. The thermal behaviour was determined using a differential scanning calorimeter and thermogravimetric analysis. In the plot of transition temperature versus number of carbon atoms in the alkoxy chain in series I, the smectic A to chiral nematic curve rises to maximum, while the Smectic C^* to Smectic A shows a falling tendency. The chiral nematic/ $\text{SmA}/\text{SmC}^*\text{-isotropic}$ curve shows the usual falling tendency. The structure-property relationship of both the liquid crystalline derivatives was discussed by comparing the laterally substituted derivatives with the laterally non-substituted one to study the scope of mesogenic characteristics[12]. In such compounds the liquid crystalline behaviour is largely determined by the repulsion forces of the surrounding molecules, which press on the additional hydrocarbon chains attached to the basic core and increase the molecule's length-to-breadth ratio[13]. Also, Ahmed et.al., suggests that a lateral substituent in a nematogenic molecule can decrease the thermal stability of the mesophase due to steric hindrance which is the case[14]. All the synthesised compounds are UV-active and show photoluminescence in the blue emission region with good quantum yield, indicating that all of the materials have blue light emission properties that can be used in potential applications such as OLED materials, biotags for biological sensing applications and fluorescent probes in biological applications. Also, the radical scavenging activities of the synthesised compounds were assessed using DPPH assay which showed that some compounds exhibited better scavenging activity as compared to standard ascorbic acid.

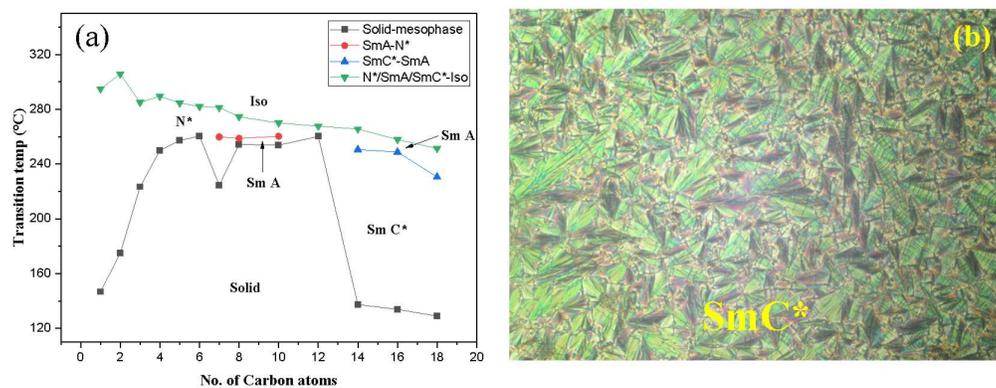


Figure 3: (a) Plot of transition temperatures ($^{\circ}\text{C}$) versus number of carbon atoms in the alkoxy chain (b) broken fan shaped texture with helix lines of SmC^* mesophase

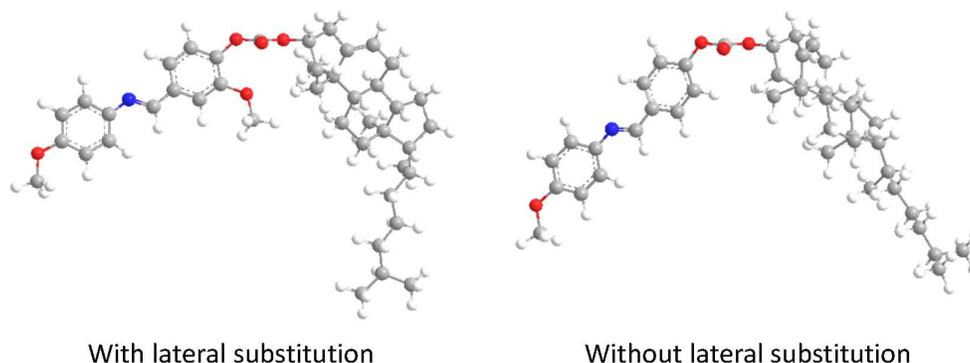


Figure 4: Molecular structure of laterally substituted and unsubstituted compounds determined by the MM2 method.

CHAPTER 3: Cholesterol functionalized azo-based biphenyl liquid crystals

In recent years, cholesterol-based liquid crystals have emerged as a fascinating class of materials, exhibiting unique mesomorphic properties and diverse applications in various fields. In this chapter, thirteen new liquid crystalline derivatives with cholesteryl group, biphenyl moiety and azo-carbonate linking group interconnected with terminal alkoxy chains were designed and synthesized. All the compounds were characterized using elemental analysis and spectroscopic techniques such as FT-IR, ^1H -NMR and ^{13}C -NMR. The mesomorphic properties of all the compounds were investigated in heating and cooling cycles using a crossed polarising optical microscopy (POM). The derivatives displayed a wide variety of mesophases, including chiral nematic (N^*), twist grain boundary-A (TGB_A) and smectic A (SmA) mesophases. The

lower homologs of the series show N* mesophase, the middle homologs shows both N* and SmA mesophase while the higher homologs show only SmA mesophases. The isotropic temperature of all the compounds within the homologous series seems to decline with the increase in carbon numbers on flexible chains. The thermal behaviour of all the synthesised compounds was checked using differential scanning calorimetry and thermogravimetric analysis. The structure-property relationship of present liquid crystalline derivatives was discussed to investigate the scope of mesogenic properties. The photoresponsive behaviour of the synthesised compounds were well examined by UV study[15]. All the synthesised compounds are UV-active and exhibit photoluminescence in the blue emission band with a notable quantum yield. The radical scavenging activity of the synthesised mesogenic derivatives were also evaluated using the DPPH assay.

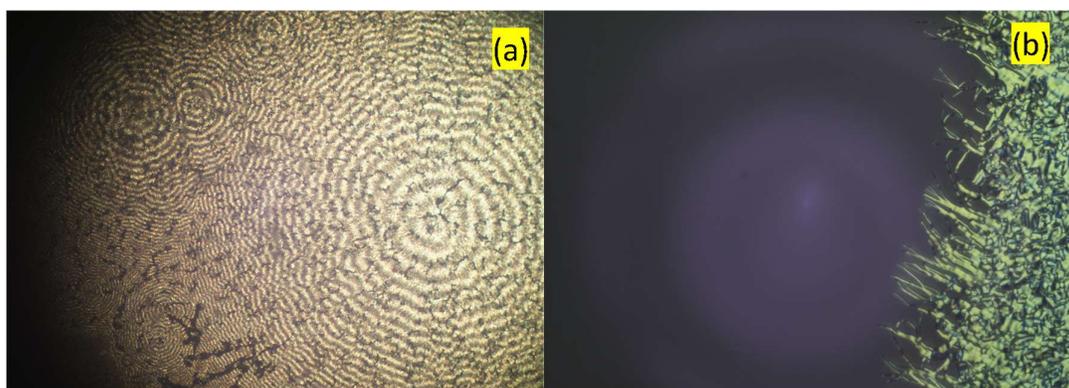


Figure 6: (a) Fingerprint texture of cholesteric phase (b) twist grain boundary phase (frustrated phase)

CHAPTER 4: Mesogenic Schiff's base derived from thiadiazole moiety

The steroidal derivatives are widely known for their ability to display a variety of mesophases, including frustrated phases, depending on the structure of the steroidal skeleton and the substituents attached. In this chapter, thirteen new homologous Schiff's base derivatives were synthesised by condensing 4-formyl phenyl cholesteryl carbonate with 5-(4'-n-alkoxy phenyl)-2-amino-1,3,4-thiadiazole. All the synthesised derivatives were characterized using elemental analysis, FT-IR, ¹H-NMR and ¹³C-NMR. Optical texture studies were carried out using a polarising optical microscope in heating and cooling cycles to examine the liquid crystalline behaviour of the synthesised compounds. The derivatives displayed a variety of mesophases, including chiral nematic (N*), twist grain boundary-A (TGB_A), smectic A (SmA) and chiral

smectic C (SmC*) phases. The methyl to n-butyl derivatives show oily streak texture of cholesteric (chiral nematic-N*) phase in both heating and cooling cycles. The n-pentyl and n-hexyl derivatives also exhibited chiral nematic mesophase. Moreover, on gradually cooling from isotropic melt an additional SmA phase was observed. The n-heptyl to n-decyl derivatives exhibited enantiotropic SmA-N*-Isotropic phase transition. The SmA-N* transition in n-pentyl to n-decyl derivatives were accompanied by an interceding frustrated mesophase (TGBA-twist grain boundary). The higher derivatives from n-dodecyl to n-octadecyl show an enantiotropic SmC*-SmA-Isotropic transition. The isotropic temperature of all the compounds within the homologous series seems to decline with the increase in carbon numbers on flexible chains. Moreover, the current series was compared with the similar achiral derivative and a chiral derivative with non-bulky group to study the structure property relationship of the compounds. The thermal behaviour of all the synthesised compounds was checked using differential scanning calorimetry and thermogravimetric analysis. All the synthesised compounds are UV-active and exhibit photoluminescence in the blue emission band with a remarkable quantum yield. As the terminal chain length increases, more pronounced emission and the higher fluorescence quantum yield was observed. The radical scavenging activity of the synthesised mesogenic derivatives were also evaluated using the DPPH assay. Some of the mesogens showed good to moderate radical scavenging activity.

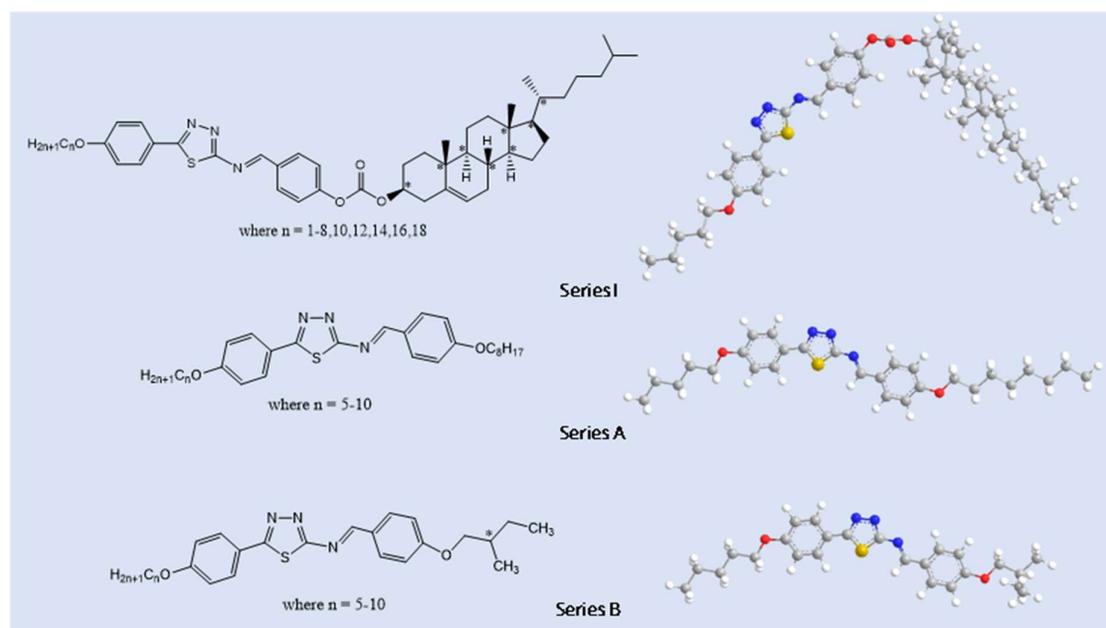


Figure 5: Comparative geometry of Series I, A and B and molecular structure of their pentyloxy derivatives determined by the MM2 method.

CHAPTER 5: 1,3,4-oxadiazole based unsymmetrical liquid crystals

A homologous series of non-symmetric 1,3,4-oxadiazole liquid crystal compounds consisting azomethine and ester linkages are reported. Thirteen new derivatives were synthesized by condensing 5-(4-n-alkoxy)-phenyl-2-amino-1,3,4-oxadiazole with 4-methoxybenzoyloxy benzaldehydes. All the synthesised compounds were confirmed by elemental analysis and characterized using various spectral techniques such as FT-IR, $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ and are in complete agreement with the structures. In order to investigate the different properties of liquid crystals the optical texture studies were carried out using crossed polarising optical microscope. The synthesised derivatives show enantiotropic mesophases including nematic (N), Smectic A (SmA) and Smectic C (SmC) phases as identified from textural observations. The lower member of the series shows nematic phase. The middle homologous shows both SmA and N phase while the higher homologs show SmC and SmA mesophases. The isotropic transition temperature of all the compounds decreases with the increasing terminal carbon chain length. The thermal behaviour was determined using a differential scanning calorimeter and thermogravimetric analysis showing that all the compounds are thermally stable up to 300-320 °C that is beyond their phase transition and isotropic temperatures. Synthesised compounds are UV-active and show photoluminescence in the blue emission region with remarkable quantum yield as compared to the standard quinine sulphate. Also, free radical scavenging activity of the synthesised compounds were assessed using DPPH assay showing good to moderate radical scavenging activity when compared to the standard ascorbic acid.

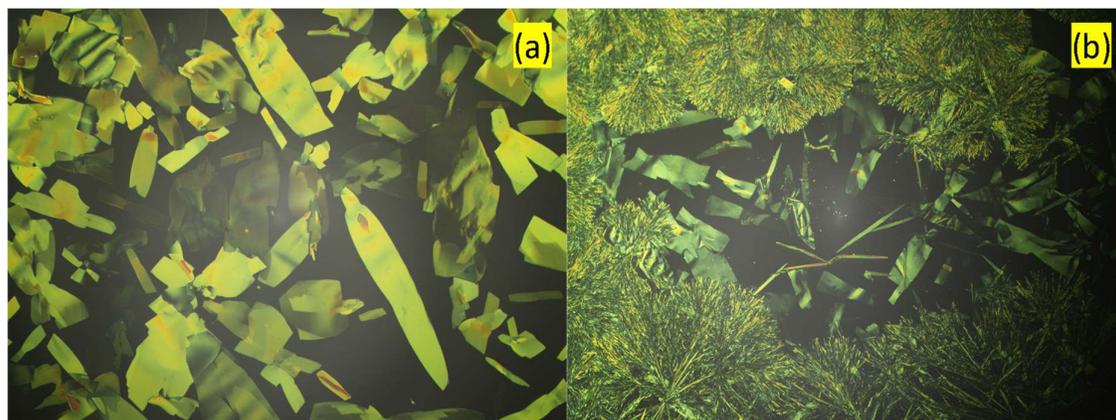


Figure 7: (a) Batonnet texture of SmA phase, (b) SmA to Cr transition

CHAPTER 6: Biologically active cholesterol-based thiazole derivatives

In the present chapter, we report the synthesis of a new series of compounds with thiazole Schiff's base linked to different substituted formyl phenyl cholesteryl carbonates. All the compounds were characterized using elemental analysis and spectroscopic techniques such as FT-IR, $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$. In order to study the liquid crystalline behaviour of the synthesized compounds, optical texture studies were carried out using optical polarising microscope in heating and cooling cycles. Among all the synthesized derivatives, only one compound exhibited liquid crystalline behaviour, showing chiral nematic (N^*) mesophase in heating as well as cooling cycle. The thermal behaviour was determined using differential scanning calorimeter and thermogravimetric analysis. All the synthesized compounds were screened for in-vitro antibacterial activity against gram-positive (*Staphylococcus aureus*) and gram-negative (*Escherichia coli*) bacteria. These compounds were also tested for their inhibitory action against *aspergillus niger* and *candida albicans* strains of fungi. Using heterocycles allows for the modulation of properties such as solubility, lipophilicity, polarity, and hydrogen bonding capacity of biologically active agent, which improves the ADME/Tox (absorption, distribution, metabolism, excretion, and toxicity) characteristics of medications or drug candidates[16]. Anti-oxidant properties of all the compounds were also evaluated using DPPH assay. While the compounds did not exhibit better radical scavenging activity in the DPPH assay as compared to standard, all the derivatives demonstrated significant to moderate levels of antibacterial and antifungal activities. All the synthesised compounds are UV-active and exhibit photoluminescence in the blue emission region. In silico predictions were made for drug likeness and bioactivity scores as well.

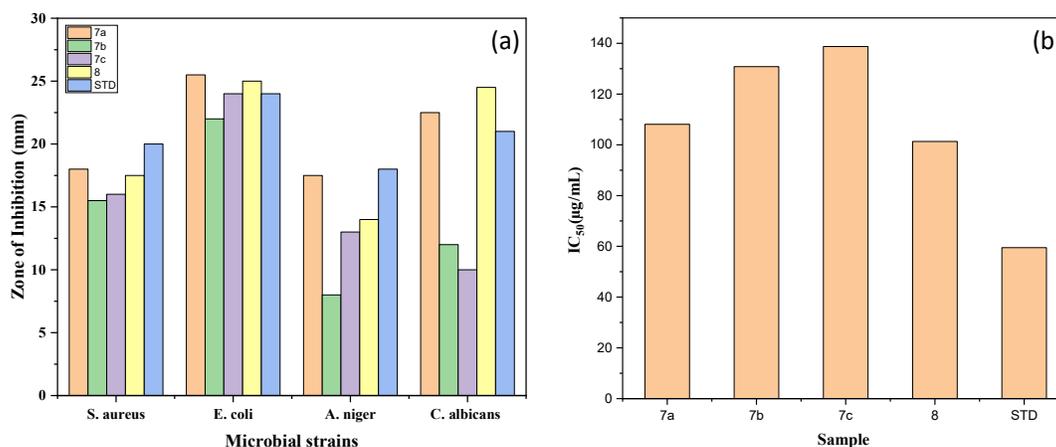


Figure 6: (a) Anti-microbial activity (b) Anti-oxidant activity of synthesised compounds

CHAPTER 7: Summary, Conclusion and Future aspects

The thesis concludes with a comprehensive summary of all the work presented and derives main conclusions from the investigations conducted. As discussed, this research probes into the fascinating area of liquid crystals, focusing on cholesterol-based derivatives and other liquid crystals. These compounds, combining liquid and crystalline properties, offer unique mesophases and applications. Emphasizing molecular design, synthesis, and characterization, the study systematically explores homologous series variations. Characterization techniques, including spectroscopy and microscopy, provide insights into properties and potential applications. Liquid crystals, with adaptability beyond laboratories, contribute to diverse fields from display technologies to drug delivery systems.

In conclusion, the study's objective is to uncover some new structural development in the field of liquid crystals, to design and synthesise new mesogens and also to study the mesomorphic properties, structural property relationships, thermal properties, optical properties and also biological properties of some mesogens which will be helpful and beneficial in the near future. This multifaceted examination aims to advance scientific understanding and contribute to technological inventions, encouraging further growth in the liquid crystal research field.

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❖ List of Publications (Related to Thesis)

- Cholesterol based mesogenic Schiff's base derivatives with carbonate linkage: Synthesis, Characterization and photoluminescence study (2023)
Sagar K. Bairwa, Srujal A. Sonera & R. C. Tandel
Liquid Crystals, DOI: [10.1080/02678292.2023.2260773](https://doi.org/10.1080/02678292.2023.2260773)
- Mesomorphic behaviour and photoluminescence study of novel homologous series of Schiff's base derived from cholesteryl carbonate and thiadiazole moiety
Sagar K. Bairwa, Srujal A. Sonera & R. C. Tandel
Liquid Crystals (Under review – Manuscript Id: [238582406](https://doi.org/10.3390/molecules24213839))
- Cholesterol based Schiff's base derivatives containing thiazole moiety: Synthesis, biological evaluation, mesomorphic behaviour and photoluminescence study (To be communicated)
- Synthesis, characterization and photophysical study of cholesterol functionalized azo-based biphenyl liquid crystals containing carbonate linkage (To be communicated)

5. Synthesis, mesomorphic and fluorescent properties of 1,3,4-oxadiazole based unsymmetrical liquid crystals (Manuscript under preparation)

❖ **List of Publications (Non-related to Thesis)**

6. Monoazo reactive dyes: Synthesis and application on cotton, silk and wool fibers
MJ Patel, SA Sonera, **SK Bairwa**, RC Tandel
Brazilian Journal of Science, 3 (1), 175-182, 2024
DOI: [10.14295/bjs.v3i1.479](https://doi.org/10.14295/bjs.v3i1.479)
7. An efficient synthesis of some novel oxazolone derivatives showing cytotoxic activity (2023)
Srujal Sonera, **Sagar Bairwa** & R. C. Tandel
Journal of the Iranian Chemical Society
DOI: <https://doi.org/10.1007/s13738-023-02918-3>
8. Trends in the synthesis and application of some reactive dyes: A review
Patel M. J., Tandel R. C., Sonera S. A., & **Bairwa S. K.**
Brazilian Journal of Science, 2(7), 14–29
DOI: <https://doi.org/10.14295/bjs.v2i7.350>
9. BI-FUNCTIONAL REACTIVE DYES: A STUDY OF THEIR DYEING PROPERTIES ON COTTON FABRIC
M.J. Patel, R.C. Tandel, S.A. Sonera & **S.K. Bairwa**
European Chemical Bulletin, 2023,12(4), 1528-1553
DOI: [10.31838/ecb/2023.12.4.117](https://doi.org/10.31838/ecb/2023.12.4.117)
10. Nematogenic Homologous Series of Coumarin derivatives Containing Azomethine-Ester Linkages: Synthesis, Characterization, Photophysical and Mesomorphic Properties
Srujal A. Sonera, **Sagar K Bairwa** & R. C. Tandel
Liquid Crystals (Under review – Manuscript Id: [231739537](#))
11. Symmetrical homologous series of thiadiazole derivatives containing azomethine linkages: Synthesis, characterization and mesomorphic properties
Srujal A. Sonera, **Sagar K Bairwa** & R. C. Tandel
Molecular Crystals and Liquid Crystals (Under review – Manuscript Id: [238700911](#))

❖ Work presented in Conferences/ Seminars/ Workshops

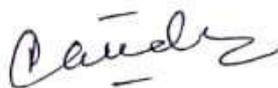
1. **Poster presentation/ Third prize**: “INTERNATIONAL CONFERENCE ON RECENT PROGRESS IN CHEMICAL SCIENCES” (RPCS-2022)
Organized by: Chemistry Department & IQAC, M. N. College, Visnagar, Gujarat, India from 22-23rd November, 2023.
Synthesis and study of oxadiazole derivatives containing central amide linkage
Sagar Bairwa, Srujal Sonera and R.C. Tandel
2. **Poster presentation/ Third prize**: “NATIONAL CONFERENCE ON EMERGING TECHNOLOGIES IN CHEMISTRY & THEIR APPLICATIONS” (NCETCA-2023)
Organized by: P.G. Department of chemistry, Government college, Sirohi, Rajasthan, India from 19-20th October, 2023.
Novel mesogenic 1,3,4-thiadiazole derivatives with azomethine and ester linkage: synthesis, characterization and photoluminescence study
Sagar K. Bairwa & R.C. Tandel
3. **Poster presentation**: “30th NATIONAL CONFERENCE ON LIQUID CRYSTALS” (NCLC-2023)
Organized by: Department of Physics, Andhra University- Vishakhapatnam in association with *Indian Liquid Crystal Society* from 02-04th November, 2023.
Synthesis, characterization and photoluminescence study of thiadiazole based liquid crystalline derivatives
Sagar K. Bairwa & R.C. Tandel
4. **Conference Attended**: International Conference on “9th Asian Network for Natural & Unnatural Materials” (ANNUM-9)
Organized by: Department of Chemistry, University of Delhi, Delhi-India on 8th April, 2022.
5. **Conference Attended**: “International Conference on Advanced Materials and Applications” (ISAMA-2022),
Organized by: Applied Physics Department & Applied Chemistry Department on 18th July 2022.
6. **Webinar**: National seminar on “Spectroscopic Techniques: A Tool for Structure Elucidation”
Organized by: Department of Chemistry, Sophia Girls’ College (Autonomous), Ajmer from 21-22nd October, 2021.

7. E-Workshop: on "Advanced Spectroscopy for Emerging Materials"
Organized online by: CSIR-National Physical Laboratory, New Delhi from 22-23rd December, 2021.
8. E-workshop: e-START Program "Chiral Sciences"
Organized by: Hiroshima University, Orenburg State University, The Maharaja Sayajirao University of Baroda, University of Colorado at Boulder, University of Glasgow, University of Lyon 1, University of Wroclaw, and Zaragoza University dated 31st January, 2022.
9. Workshop: One-week Synergistic Training program Utilizing the Scientific and Technological Infrastructure (STUTI) on "Spectrometric analysis and imaging of biological samples"
Organized by: Sophisticated Analytical and Technical Help Institute (SATHI), Banaras Hindu University and National Institute of Technology, Warangal from 25th April- 1st May, 2023.
10. Achievement: Liquid Crystal image accepted for the month July-2023 and published by the *International Liquid Crystal Society* as the featured liquid crystal artist of the month. (<https://www.ilcsoc.org/art-contest/gallery/page-193/styled-11/page-197/>)



Signature of Candidate
(Bairwa Sagar K.)

Endorsement of Supervisor;
Synopsis is approved by me



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Guide


Head

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