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Analysis of Mesogenic Properties Exhibited by Linear Supramolecular Hydrogen Bonded Thermotropic Liquid Crystals

P. Rohini [a], N. Pongali Sathya Prabu [a, *]

[a] Department of Physics, Liquid Crystal Research Laboratory (LCRL), Centre for Research, Bannari Amman Institute of Technology, Sathyamangalam-638401, India

* Corresponding Author Email: pongalispn@bitsathy.ac.in

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Abstract: A supramolecular hydrogen-bonded thermotropic liquid-crystalline complex was synthesized by combining myristic acid (MC) and 4-n-pentyloxybenzoic acid (5BAO) in an equimolar ratio, and its mesogenic, optical, and thermal characteristics were systematically investigated. The formation of intermolecular hydrogen bonding between the carboxylic acid groups of the precursor molecules was confirmed by Fourier transform infrared spectroscopy, which revealed characteristic shifts in the O-H and C=O stretching vibrations after complexation. The mesomorphic behaviour of the prepared complex was examined by polarizing optical microscopy, which identified the presence of nematic and smectic C mesophases through their characteristic optical textures during the cooling cycle. Differential scanning calorimetry further established the corresponding phase-transition temperatures and enthalpy changes in both heating and cooling runs. The complex exhibited phase transitions associated with crystal, smectic C, nematic, and isotropic states, demonstrating rich phase polymorphism and good thermal response. Thermal analysis also indicated that the nematic phase possessed a wider mesophase range and greater thermal stability than the smectic C phase. In addition, specific heat and transition-order analyses supported the thermal behaviour of the system. The observed spectral response suggests that the complex may serve as a promising candidate for tunable optical filtering and thermally responsive liquid-crystalline applications.

Keywords: Linear Supramolecular Hydrogen Bonded Liquid Crystals, Phase Variance, Phase Polymorphism, Transition Temperatures, Thermal Parameters.

1. Introduction

An interesting state of matter prevailing commonly called liquid crystals is characterized by an intermediate mesophase that hold both the properties of liquids and crystalline solids [1]. The soft condensed materials possessed exciting property of long-range orientational order with varying positional order and behave as liquids, this is the validity of display applications. It's fascinating property of anisotropy provides directional properties along with physical changes of liquid crystals such as refractive index, electrical conductivity, etc., and this is suitable for various applications in flat panel field [2-3]. In the field of display technology, liquid crystals mark a major role in Liquid Crystal Displays (LCD). With the influence of orientation of molecules along with electric fields, the passive display device of LCD repels the light path which is the main character to allow the formation of high-resolution screens used in devices ranging from smart phones to flat panel televisions [4-5]. Recent investigations elaborately explained these display devices [6-8] also more suitable for optical devices, such as optical filters, optical shutters and modulators, contributing to innovations in photonics and optical engineering field [9-11]. Furthermore, more utility is also remarked in biological systems, sensors and soft matter physics research [12-13].

Various liquid crystals and its derivatives are synthesized by the researchers across the worldwide [14-16] and the self-assembling separate molecules of hydrogen bond liquid crystals (HBLC), a well-known category of thermotropic liquid crystals [17-19] leads to possess space in the present technology due to their ease formation of mesophase and the derived mesogens exposed some properties with respect to the application of external factor either in the form of optical or thermal or electrical [20-21]. These charming properties are contributed to the complimentary supramolecular assembly of hydrogen bond creation that majorly occurs between the carboxylic acid functional groups of the chemical ingredients used in the formation of HBLC. This delicate nature of hydrogen bond combines and breaks in an easier way for the formation of mesogens depends on temperature

conditions [22-23]. Several scientists mostly investigated HBLC, because of their proton donor is existing in a carboxylic acid and proton acceptor is existing in a nitrogen containing molecule, self-molecular assembly, dynamic nature, ease of creation and the applicational utility [24]. The mesogens are harvested by the carboxylic acids due to the formation of delicate hydrogen bond. Among the liquid crystal field thermotropic hydrogen bond liquid crystals build a physico-chemical relationship between the soft condensed matter physics and supramolecular chemistry. Therefore, these thermotropic mesogens, exhibit rich phase polymorphism which is determined by the creation of hydrogen bond between its molecular assemblies. Self-assembly molecules as well as weak hydrogen bond contribution is more for this type of liquid crystals. The behavior of liquid crystal phase exposes the mesogenic phases like nematic, smectic and cholesteric phases. Mostly phase transitions are held based on the variation of temperature, which is responsible for the different structural and optical properties of the mesogens. Among the various kinds of liquid crystals, the applications of thermotropic hydrogen bond liquid crystals distinct pay attention to the area of optoelectronics, sensors and smart materials. Their tunable optical and electronic properties are accountable for organic electronic devices like OLEDs and OFETs [25-26]. These properties also responsible for tunable lenses and filters especially for adaptive eye glasses. When HBLC into smart materials, it stimulates reversible changes in their properties especially in optical, thermal and electrical, by the external factors of temperature, light and chemical ingredients. In smart window applications, it is referred as switchable privacy glass that can instantaneously change the optical properties, switching from opaque state to transparent state depends on the application electric field. These smart liquid crystal materials have applied in controlled release systems and surface engineering for better results [27-29].

Based on the above arguments and the results, an attempt is made in obtaining HBLC between two carboxylic acid group ingredients. Pentyloxy benzoic acid (5BAO), with carboxylic acid is inherently mesogenic in nature [30]. These acids are capable of forming complementary hydrogen bonds with Myristic acid (MC), a saturated fatty acid possessing a long flexible alkyl chain. Alkyloxy benzoic acid is well established in the formation of mesogenic complexes between any mesogenic or non mesogenic moieties [31–33]. Based on these observations, the present study investigates the influence of the alkyl chain length of the acid moiety particularly that of a saturated fatty acid on phase polymorphism. The analysis focuses on the mesogenic richness exhibited by the homologous complex (MC+5BAO) and examines the effect of the electronegative atom present in the rigid core on the chemical, optical, and thermal response properties of the resulting complexes. The present analysis is the combination of flexible fatty acid (MC) of long carbon chain length with a mesogenic molecule viz., pentyloxy benzoic acid (5BAO) which is of short chain length, which is quite different form the other carboxylic acids investigated and reported earlier.

2. Experimental Instrumentation

The synthesized mesogenic complex is characterized using standard experimental techniques. The purpose of chemical analysis is to establish hydrogen bonding by Fourier Transform Infrared Spectrometer (FTIR), ABB Bomem, Canada, equipped with MB 3000 series software [34–35]. The mesogenic nature of the complexes is confirmed through the observation of the mesophases and their corresponding phase transition temperatures. Both the objectives are accomplished by Polarizing Optical Microscope (POM), Nikon, Japan equipped with Nikon Imaging Software (NIS). INSTEC HCS-402 is used to give the steady temperature to the mesogens interfaced with mk 2000 temperature controller having accuracy of $\pm$ 0.1^{0}C [36–37]. The transition temperatures, their corresponding enthalpy values and other derived thermal parameters such as specific heat values, odd-even pattern are obtained by Differential Scanning Calorimetry (DSC), Shimadzu DSC-60, Japan [38–40]. Myristic acid (MC, purity $\geq$99%) and pentyloxy benzoic acid (5BAO, purity ~ 99%) were procured from Sigma-Aldrich (Germany), and all solvents used were of High-Performance Liquid Chromatography (HPLC) grade.

2.1 Synthesis of MC+5BAO complex

Equimolar quantities (1:1 molar ratio) of the precursor compounds Myristic acid (MC, 0.038061667g) and 4-n-pentyloxybenzoic acid (5BAO, 0.03471g) with respect to their molecular weight are weighed and dissolved in N, N-dimethylformamide (DMF) solvent until the compound gets completely dissolved. The resulting solution is stirred continuously for approximately 12 hours using a magnetic stirrer to ensure complete interaction between the components. The solvent should be completely evaporated by means of slow stirring and the product is then collected whose yield is approximately 95% without further purification. The dried mesogenic complex is obtained as the final product after stirring at ambient temperature. A schematic representation illustrating the formation of the MC+5BAO mesogenic complex is shown in Figure 1.

3. Results and Discussion

The stability of the synthesized mesogenic complex, in terms of their chemical and thermal properties, are verified through Polarizing Optical Microscopy (POM) and Differential Scanning Calorimetry (DSC).

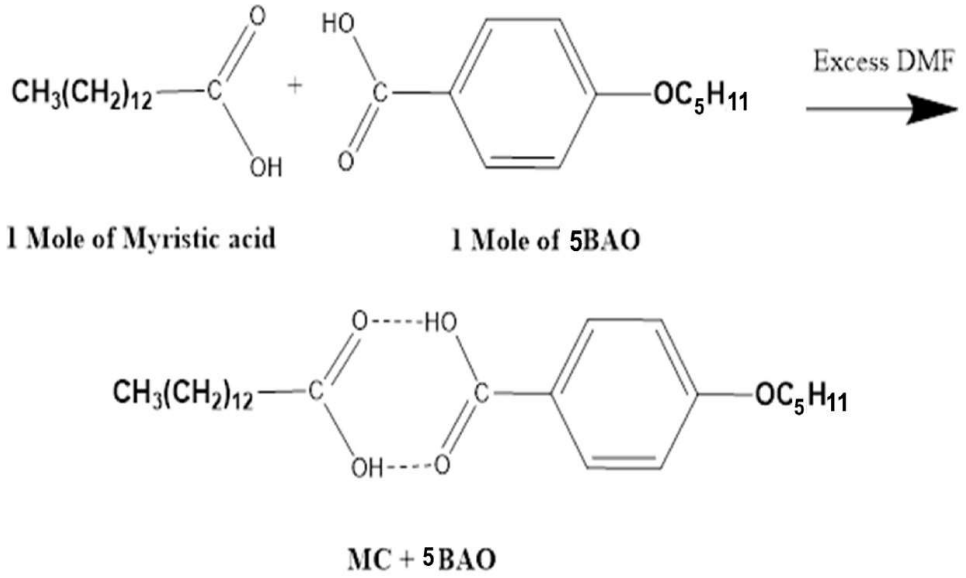

Figure 1. Schematic representation of MC+5BAO complex formation

The reproducibility of the observed mesophases and thermal transition parameters confirmed the reliability and stability of the complexes, as consistent results are obtained in successive measurements.

The delicate hydrogen bond (both intermolecular and intramolecular hydrogen bonding) is responsible for the mesomorphic behavior of this complex. The rigid and elongated hydrogen bonded dimers which are present in the carboxylic acid groups make the overall structure to exhibit mesomorphic nature. In the nematic phase, the molecules align in the same direction but not have fixed positions, but in the smectic C phase, the stronger interactions produce a layer arrangement with a slightly tilted orientation.

Hence, there is balance between rigid hydrogen bonded cores and flexible chains which leads to the perceived liquid crystalline behavior.

3.1 Phase Identification

The mesophase behavior, phase variance, and corresponding transition temperatures of the MC+5BAO complex is investigated using POM and DSC, representing the optical and thermalanalyses, respectively.

3.1.1 MC+5BAO complex

The MC+5BAO complex exhibits nematic (N) and smectic C (SmC) mesophases. These mesophases are confirmed through textural observations using POM and correlated with previously reported characteristic textures [41–43]. Figure 2 (a, b) illustrates the textures corresponding to the nematic and smectic C phases, respectively. The textures are recorded during the cooling cycle using a programmed temperature controller with a controlled cooling rate of 5°C/min. The accuracy of the measured phase transition temperatures is ±0.1°C. Specifically, plate 1 shows droplet-like textures characteristic of the nematic phase (102.8°C) and plate 2 displays a sandy layered texture corresponding to the smectic C phase (71.5°C).

The phase sequence and the nature of the transitions whether monotropic or enantiotropic for the MC+5BAO complex is represented below.

Isotropic $\longrightarrow$ N $\rightleftharpoons$ SmC $\rightleftharpoons$ Crystal (MC+5BAO)

A single-headed arrow denotes a monotropic transition, while a double-headed arrow indicates an enantiotropic transition. The nature of these transitions significantly influences the thermal properties of the mesogens in a quantitative manner.

3.2 Infrared Spectroscopy (FTIR)

The formation of hydrogen bonded liquid crystals is confirmed by the presence of intermolecular hydrogen bonding between Myristic acid (MC) and 4-n-pentyloxybenzoic acid (5BAO). For FTIR analysis, the mesogenic complex MC+5BAO is thoroughly blended with FTIR-grade potassium bromide (KBr) in an appropriate ratio and compressed using a KBr press to obtain transparent pellet The pellets are sandwiched between the IR source and detector of ABB Bomem FTIR spectrometer and the parameters such as signal intensity and gain are well chosen to achieve the FTIR spectra. The entire spectra are recorded at ambient temperature and in the mid IR range viz., 4000-400 cm^{-1}. Occurrence of a sharp and intense transmittance peak around 2885 cm^{-1} confirms the O-H bond vibration between the carboxylic acids [44-45]. The FTIR spectrum for the precursor, pentyloxy benzoic acid shows a broad peak at 2947 cm^{-1} confirming the intramolecular hydrogen bonding. A clear red shift of 62 cm^{-1} is noticed, thus confirming the intermolecular interactions between the precursor acids chosen.

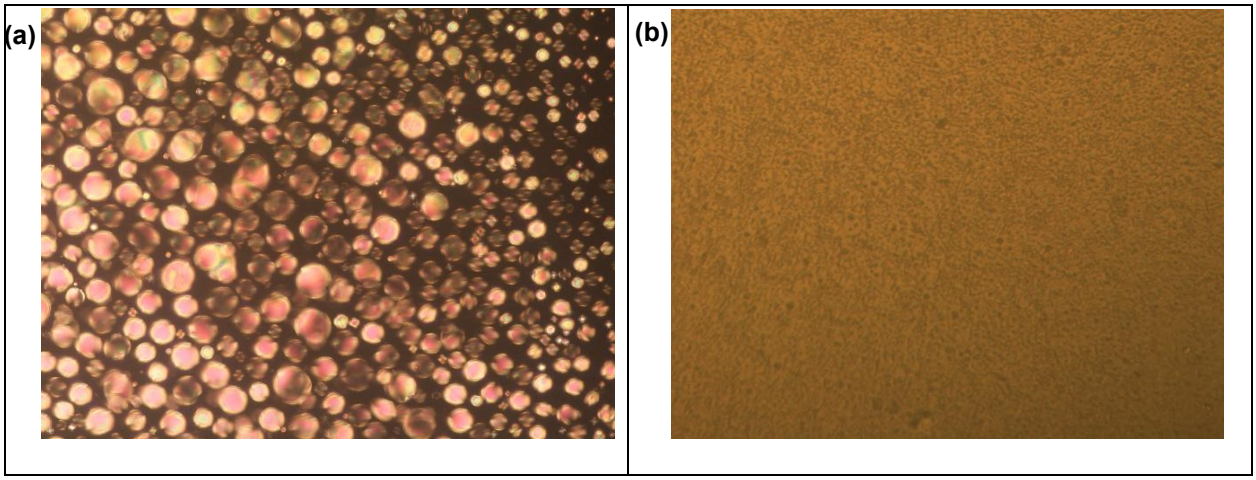

Figure 2. Plate (a) - Droplet texture of nematic phase observed in MC+5BAO complex, **Plate (b)** - Sandy texture of smectic C phase observed in MC+5BAO complex

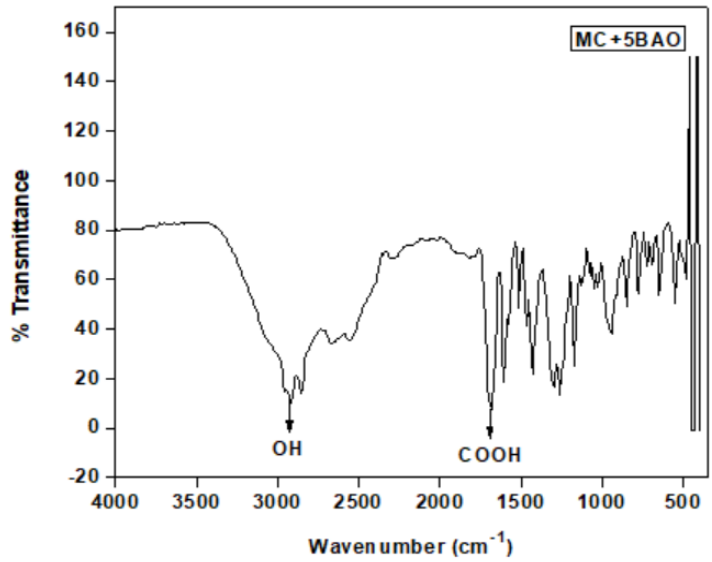

Figure 3. FTIR spectra: MC+5BAO

Again a sharp peak noticed around 1759 cm^{-1} is attributed to the C=O stretching that occurs upon complexation of the mesogens [46-47] and its corresponding precursor vibration is noticed around 1674 cm^{-1} thus confirming the complex formation. Figure 3 depicts the FTIR spectrum of MC+5BAO complex.

Thus the intermolecular hydrogen bond between the mesogenic complex formations is well established through the chemical analysis performed for the complex MC+5BAO.

3.3 DSC Studies

DSC thermograms for the mesogenic complex MC+5BAO are obtained in both heating and cooling cycles [48]. Aluminum pan is used to fill the sample and the same is crimped and placed in the heat chamber of DSC for thermal scan (10°C / min). The thermal run reaches the isotropic state from room temperature and hold for a while to attain thermal stableness. The cooling run is then performed till the mesogens reaches the room temperature. The MC+5BAO complex undergoes the respective transition temperatures accompanied by their corresponding enthalpy values.

3.3.1 Thermal analysis: MC+5BAO complex

DSC thermogram of the mesogenic complex of MC+5BAO is depicted in figure 4. Figure 4 shows the DSC thermogram recorded during both heating (endothermic) and cooling (exothermic) cycles.

It is well noticed that the DSC cooling run exhibits three exothermic peaks at 107.8 °C (onset temperature – 117.9°C, end set temperature – 108.1°C), 79.8 °C (onset temperature – 83.3°C, end set temperature – 76.5°C), and 48.1 °C (onset temperature – 48.9°C, end set temperature – 45.8°C), with enthalpy values of 8.49 Jg^{-1}, 22.52 Jg^{-1}, and 80.19 Jg^{-1}, respectively.

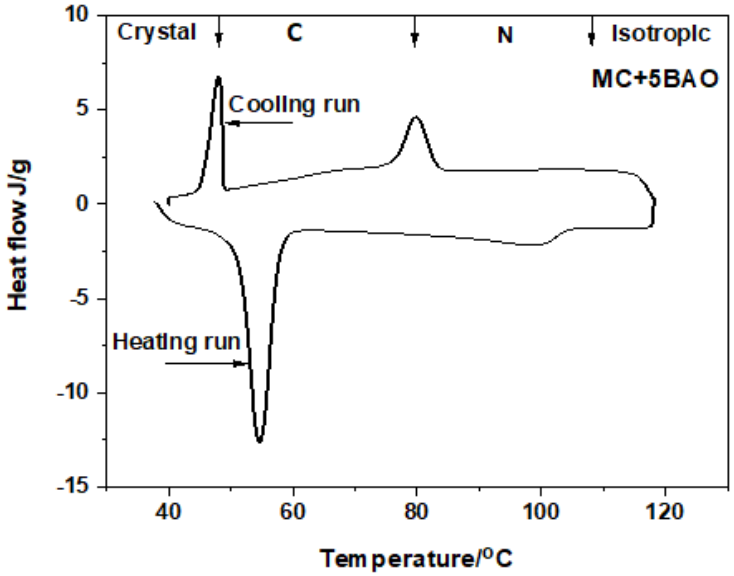

Figure 4. DSC thermogram: MC+5BAO complex

The three distinct peaks observed in the cooling run corresponds to isotropic to nematic, nematic to smectic C and smectic C to crystal phase transition respectively. In the heating run of DSC, two distinct peaks are obtained for the same complex at temperatures 54.7 °C (onset temperature – 51.5°C, end set temperature – 57.4°C), and 98.5 °C (onset temperature – 91.0°C, end set temperature – 104.1°C), possessing enthalpy values of 81.49 Jg^{-1} and 10.70 Jg^{-1} respectively. These peaks correspond to crystal to melt and melt to smectic C phase transition respectively.

The highest thermal span exhibited by the nematic phase compared to the smectic C phase, occupied 2/3 of the total mesophase range. This contribution exhibits the wider application of nematogen compared to other mesogens [49-50].

3.4 Thermal equilibrium analysis: MC+5BAO complex

Thermal properties possessed by the thermotropic liquid crystals validate the laws of thermodynamics [51]. This has been further confirmed from summating the energy given to the mesogens and energy released by the mesogens for the mesophase variation. The enthalpy values possessed by the individual mesophase transition in endothermic and exothermic cycles are cumulated and the magnitude remains almost same. A slight variation in the magnitude is attributed to the observation of monotropic transition (mesophase observed only in one thermal run) in any of the thermal runs. Hence the law has been verified. In the mesogenic complex of MC+5BAO, during heating the observed enthalpy value is 92.19 Jg^{-1} and during cooling the observed enthalpy value is 111.20 Jg^{-1}. The observed variation between the endothermic and exothermic enthalpy values reveals experimental uncertainties viz., order of observed transition as either monotropic or enantiotropic transitions. The deviations in the magnitude of the internal energy possessed is attributed to this nature of transition observed as the nematic mesophase transition is not observed in heating cycle.

3.5 Thermal Stability Factor: MC+5BAO complex

Further thermal analysis involves determining the phase stability with respect to the thermal span of the mesophases. Thermal stability factor [52–53] is one of the crucial thermal parameter of the mesogen that needs to be determined as it describes the stableness of the mesophases thus elevating them for a particular applicational viability.

For finding the stability of nematic phase,

$S_N = T_{mid} \times \Delta T_N$

T_{mid} - Mean temperature of the nematic phase

ΔT_N - Total temperature range of the nematic phase

The thermal stability factor elaborates how broad the temperature range is in the mesogenic phase. If the range is large, the phase is considered more stable because it exhibits wide thermal span. Similarly, the thermal stableness of smectic C mesophase is determined. In the MC+5BAO complex, the thermal stability factor of the nematic is calculated as 2628 and for smectic C the value is 2025. Nematic phase has greater thermal span and hence possess higher thermal stability when compared to the other mesogens concluding the thermal viability of the nematogens.

Table 1. Order of transition: MC+5BAO complex

Mesogenic phase	Scan rate	Peak height	Cox ratio (10/5)	Transition order
Nematic	10ºC / min	0.02	2	Second order
	5ºC / min	0.01		
Smectic C	10ºC / min	2.82	1.11	First order
	5ºC / min	2.53		

3.6 Order of transition: MC+5BAO complex

Order of phase transition exhibited by a particular mesophase reflects the transitional properties in the particular thermal onset and off set temperatures. Several techniques have been implemented in executing the above argument and the technique proposed by Navard and Cox [54-55] still remains an effective one due to its efficacy in the results obtained. Weight of the mesogens are kept identical and the scan rate is being varied twice (10ºC / min) the initial one (5ºC / min) and the order of transition is elucidated from the peak heights observed (Table 1).

If the ratio of the magnitude obtained confines within 1.414, then, the transition is classified as first order and if the magnitude exceeds 1.414, then the order of transition is classified as second order transition.

For the analyzed mesogenic complex of MC+5BAO, the nematic phase transition is found to be second order, with ratio value is 2 and the smectic C phase transition is found to be first order, with ratio value is 1.11.

3.7 Specific heat value: MC+5BAO complex

The thermal energy required for each individual mesophase transition is evaluated in terms of the specific heat capacity (C_p). This thermal parameter can be determined using several methods. However, the procedure proposed by Garland [56-57] is widely recognized for its reliability. In this method, the sample, reference and standard materials are considered during analysis. Thermal scans are performed for the MC+5BAO complex, and the obtained data are substituted into the following expression:

$$C_{p(s)} = E\frac{60\times D_S}{W_S\times r} \quad (1)$$

Where:

W_s - Mass of the liquid crystalline complex
D_s - Difference in heat flow between the liquid crystalline complex and the reference specimen
r - Heating rate

The constant E is calculated using:

$$E = \frac{r\times W_{st}\times C_{p(st)}}{60\times D_{st}} \quad (2)$$

Where:

$C_{p(st)}$ - Specific heat capacity of the reference sample
W_{st} - Mass of the reference sample
D_{st} - Heat flow difference between the empty pan and the reference sample

The specific heat (C_p) values of the prepared mesogenic complex is determined from the DSC thermogram recorded at a scanning rate of 10 °C min^{-1} using the above equations. It is represented in the Table 2.

The graphs plotted based on the Garland expression for the MC+5BAO complex are presented in Figure 5. The resulting thermogram closely resembles the corresponding DSC curves, thereby validating the proposed theory.

3.8 Mechanism of Optical Filtering

Fourier Transform Infrared (FTIR) spectroscopy establishes the formation of hydrogen bonding between the precursors involved in the preparation of mesogenic complexes [58]. The spectrum denotes how material interacts with IR radiation optical source, further optical analysis is required to assess its performance in any practical filtering applications. Photons interact with the mesogenic molecules and identifies the functional groups by means of vibrations observed at a particular wavenumber. The liquid crystals shall act as a tunable optical filters based on the orientation of the light transmission. Strong intense band observed for –OH group refers to the stable mesophases which leads to a better light filtering action. Aromatic peaks lead to enhanced optical anisotropy and shifting in the peaks produces tunable optical response. Taking the above parameters in to consideration, the optical filtering analysis is performed based on the FTIR spectrum observed for the present MC+5BAO complex. When heated the liquid crystals by its mesophase temperature range, it exhibits distinct optical behavior. However, the transmitting light over a bundle of wavelengths whereas a sharp and narrow wavelength band reflecting its range [59] and this phenomenon is referred as filtering action. From the wavelength dependent transmittance characteristics, optical filters are divided as high-pass, low-pass, band-pass and notch filters. A similar trend is observed upon the careful examination of the MC+5BAO FTIR spectrum.

Table 2. Specific heat calculation: MC+5BAO complex

Temperature (°C)	Sample ($JKg^{-1}K^{-1}$)	Empty pan ($JKg^{-1}K^{-1}$)	Indium ($JKg^{-1}K^{-1}$)	D_s ($JKg^{-1}K^{-1}$)	D_{st} ($JKg^{-1}K^{-1}$)	E value	$C_{p(s)}$ ($JKg^{-1}K^{-1}$)
107.78	1.83	0.07	1.55	1.76	1.48	0.035047	0.132178
79.75	4.64	-0.13	1.23	4.77	1.36	0.03814	0.389842
48.07	6.79	0.06	0.44	6.73	0.38	0.1365	1.968525

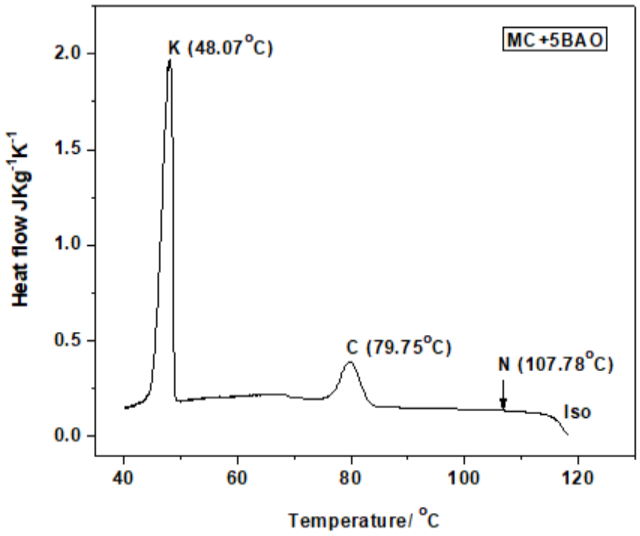

Figure 5. Specific heat curve: MC+5BAO complex

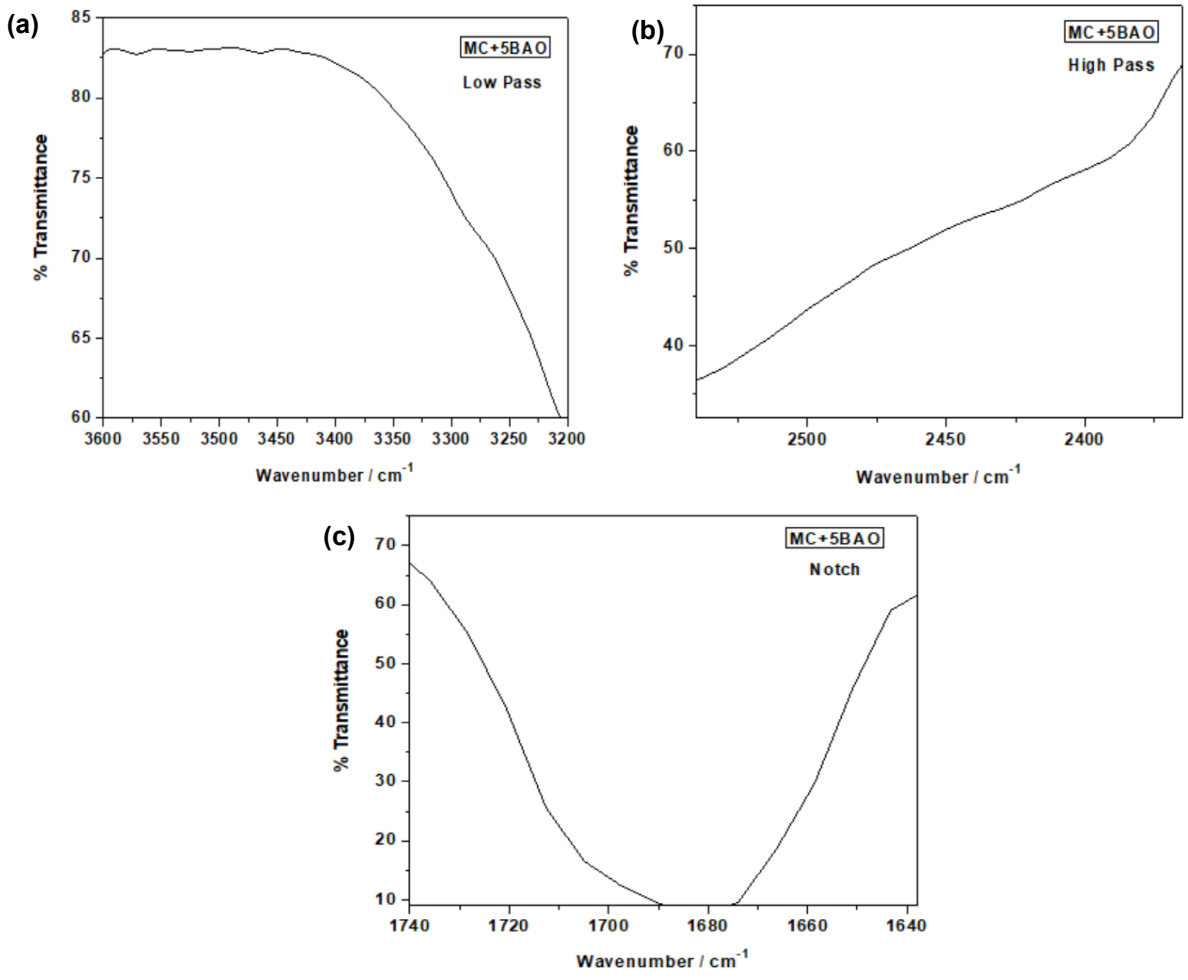

Figure 6 a-c. Optical filtering behavior: MC+5BAO complex

3.8.1 Spectral Classification of Optical Filters: MC + 5BAO complex

From the observation of filtering action, the various kinds of optical filtering behavior are perceived in the MC+5BAO complex (Figure 6a–6c) which are elaborated. The transmitted spectra deliberate the specific regions exhibiting features that corresponds to different kinds of optical filters, as proven by the response of the complex that are evinced through incident IR radiation.

The MC+5BAO complex exhibits low-pass filter behavior in the range of 3600-3200 cm^{-1} (Figure 6a) and in this range, the lower frequency components reveal the allowing transmission while the upper frequencies attenuating the signal. In contrast, within the 2550-2350 cm^{-1} region (Figure 6b), the high-pass filter behavior is identified while analyzing the spectra and allowing the upper frequency components to pass predominantly. Furthermore, the notch filtering action is observed in the range of 1740-1640 cm^{-1} (Figure 6c) through the analysis of the spectrum, where the selective narrow band of frequencies are attenuated [60]. The characteristics of distinguished wavelength transmission and attenuation are detected in the mid-IR region which specifies the self-assembly mesogenic systems be able to function as tunable optical filters.

4. Conclusions

The present investigation confirms that the equimolar combination of myristic acid and 4-n-pentyloxybenzoic acid produces a stable supramolecular hydrogen-bonded liquid-crystalline complex with significant mesogenic and thermal characteristics. FTIR analysis verified the formation of intermolecular hydrogen bonding through the characteristic shifting of the vibrational bands associated with the carboxylic acid groups. Polarizing optical microscopy revealed the occurrence of nematic and smectic C mesophases, while differential scanning calorimetry provided clear evidence for the associated phase transitions and enthalpy variations during heating and cooling cycles. Among the observed mesophases, the nematic phase displayed a broader thermal range and a higher thermal stability factor than the smectic C phase, indicating its superior thermal robustness. The order-of-transition analysis showed that the nematic transition is second order, whereas the smectic C transition is first order, further highlighting the distinct thermal behaviour of the complex. The specific heat evaluation also supported the DSC findings and confirmed the consistency of the thermal response. Overall, the MC+5BAO complex exhibits favourable mesomorphic, thermal, and spectral characteristics, making it a promising material for future exploration in tunable optical filters, thermally responsive systems, and advanced functional liquid-crystal devices.

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Data Availability

The data supporting the findings of this study can be obtained from the corresponding author upon reasonable request.

Has this article screened for similarity?

Yes