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The Effect Of Temperature On Phase Transitions And Optical Properties In A Binary Mixture Of Cholesteryl Nonanoate And CTAB

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Abstract
The present study investigates the effects of temperature on phase transitions, optical textures, optical anisotropy, and birefringence in a binary mixture of cholesteryl nonanoate and the surfactant N-cetyl-N,N,N-trimethylammonium bromide (CTAB). A binary mixture consisting of 70% CTAB and 30% cholesteryl nonanoate exhibits sequential phase transitions of cholesteric, SmA, SmB, and SmE phases upon cooling from the isotropic state. Similar sequential phase transitions were observed for mixtures of different concentrations. Temperature variations of the refractive indices of the binary mixture indicates that during cooling, both the ordinary refractive index (n_o) and extraordinary refractive index (n_e) increase non-linearly in the temperature range of 210°C to 182°C. The value of the extraordinary refractive index (n_e) remains higher than that of the ordinary refractive index (n_o) throughout the entire temperature range, confirming the optical anisotropic nature of the binary mixture. Analysis of the birefringence (Δn) variations indicates that the birefringence remains nearly constant at approximately 0.03 in the temperature range of 182 °C to 198 °C. At 202 °C, the birefringence increases to 0.064 and decreases slightly to 0.060 at 206 °C, before reaching a maximum value of 0.078 at 210°C. The observed birefringence shows a non-linear variation with temperature, indicating temperature-dependent changes in molecular ordering within the liquid crystalline phases. The results show a non-linear variation of birefringence with temperature, indicating the temperature-dependent changes in molecular ordering within the liquid crystalline phases.

Keywords: *liquid crystal, binary mixture, texture, optical anisotropy, birefringence*

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I. Introduction

Most of the materials exhibiting liquid crystalline behaviour belong to two general classes, namely thermotropics and lyotropics. Thermotropic liquid crystals are substances that form liquid crystal phases (mesophases) with unique optical and physical properties by changes in temperature, unlike lyotropic ones that change with concentration. They typically consist of rod-like molecules with rigid cores and flexible chains, exhibiting states such as nematic, smectic, or cholesteric. They are used in displays, thermometers, and smart windows because of their responsive colour, texture, and transparency changes with heat [1–3].Transition into a mesophase obtained by a purely thermal process is called thermotropic, whereas that obtained by the influence of a solvent on a solid is called lyotropic. Thermotropic liquid crystals generally exhibit three types of phases, namely nematic, cholesteric, and smectic. Phase changes in liquid crystals (LCs) are critical for controlling their optical, electrical, and thermal properties by altering molecular ordering through temperature or concentration variations. These reversible transitions, particularly between nematic and isotropic states, allow precise and high-contrast modulation of light in display technology, switching devices, and thermal energy storage systems, utilizing latent heat without the disadvantages of traditional solid–liquid phase separation [4].Phase changes in liquid crystals are important for display technology (LCDs), thermal energy storage (TES), tunability, structural reordering, and switching mechanisms [5]. Liquid crystalline compounds exhibit optical anisotropy, which has remarkable significance [6]. Optical anisotropy in liquid crystals refers to the variation of optical properties, such as refractive index, with direction due to the partial ordering of rod-like molecules. This alignment results in two refractive indices, namely the ordinary refractive index (n_o) and the extraordinary refractive index (n_e) [7–9].Liquid crystals also exhibit birefringence, which is crucial for liquid crystal displays and optical modulators, where alignment can be controlled by electric fields. Liquid crystal birefringence (Δn) is defined as the difference between the extraordinary and ordinary refractive indices and is expressed as $\Delta n = n_e - n_o$. Birefringence is a key parameter for display technology and varies with temperature and wavelength. It is directly related to the molecular order parameter (S) [10,11]. Birefringence decreases with increasing temperature and becomes zero at the clearing temperature. In liquid crystals, the refractive index varies significantly with temperature due to changes in molecular alignment and density, generally decreasing as temperature increases. However, complex behaviour is observed for the ordinary (n_o) and extraordinary (n_e) refractive indices, including the existence of a crossover temperature at which the temperature gradient of n_o changes sign. This behaviour is important for display applications. Both n_e and n_o decrease as temperature approaches the clearing point, where the liquid crystal becomes isotropic. However, n_o may initially decrease and then increase, whereas n_e decreases continuously [12–14].

II. Materials And Methods

Cholesteryl nonanoate is a thermotropic liquid crystal with the molecular formula $C_{36}H_{62}O_2$. Its liquid crystalline phases, specifically the cholesteric and smectic phases, depend on temperature changes in the pure compound rather than on concentration in a solvent. Cholesteryl nonanoate has a well-defined liquid crystalline temperature range of 77 °C to 90 °C [15–17]. The overall liquid crystalline window, spanning the crystal → SmA → cholesteric → isotropic transitions, lies approximately between 77°C and 90°C. Within this temperature range, cholesteryl nonanoate also exhibits thermodynamically stable blue phases (BPI, BPII, and BPIII) just below the isotropic phase, interposed between the cholesteric and isotropic states [18]. N-Cetyl-N,N,N-trimethylammonium bromide (CTAB) is a cationic quaternary ammonium surfactant used as an emulsifying agent because of its micelle-forming capability in aqueous solutions. CTAB has the molecular formula $C_{19}H_{42}BrN$ and a melting point in the range of 237°C to 251°C. CTAB is well known for its dispersion, suspension, solubilisation and transport properties. These characteristics make it a suitable compound for mixing or interacting with other materials to form emulsions [19,20]. Homogeneous mixtures of different concentrations of cholesteryl nonanoate and CTAB were prepared in the present study. Desiccators were used for prolonged periods to obtain samples of different concentrations. To achieve homogeneity, the samples were subjected to repeated cycles of heating, stirring, and centrifugation.The prepared samples were sandwiched between a glass slide and a cover slip and sealed properly for microscopic observation. The optical textures of these mixtures at various temperatures were examined and recorded using a Gippon polarising microscope equipped with a hot stage. The refractive indices of the corresponding mixtures were measured by the method of minimum deviation using a goniometer spectrometer. The temperature of the sample was increased by gradually increasing the voltage across the terminals of the spectrometer.

The molecular orientations of the optical textures exhibited by the samples were observed and recorded using a Gippon polarising microscope in conjunction with a hot stage. In each case, the specimen was prepared in the form of a thin film and sandwiched between a glass slide and a cover slip.

Optical Texture Studies

The molecular orientations of the optical textures exhibited by the samples were observed and recorded using a Gippon polarising microscope in conjunction with a hot stage. In each case, the specimen was prepared in the form of a thin film and sandwiched between a glass slide and a cover slip. A mixture consisting of 70% N-Cetyl-N,N,N-trimethylammonium bromide (CTAB) and 30% cholesteryl nonanoate exhibits cholesteric, SmA, SmB, and SmE phases sequentially when cooled from the isotropic phase. The phase transitions were carefully observed and recorded. When the sample was cooled from the isotropic phase, nucleation initially appeared in the form of small bubbles growing radially. These were identified as spherulitic textures characteristic of the cholesteric phase, as shown in Figure 1(a). The cholesteric phase exists in the temperature range of 210°C to 175°C.On further cooling, the texture gradually transformed into a composite structure consisting of isogyre-like patterns and SmA phase textures. This composite structure exists in the temperature range of 210°C to 196°C.With continued cooling, the texture slowly transformed into the SmB phase, in which molecules are arranged in well-defined layers, as shown in Figure 1(c). The SmB phase exists in the temperature range of 196 °C to 182°C.Further cooling caused the unstable SmB phase to transform into the SmE phase, as shown in Figure 1(d). The SmE phase exists in the temperature range of 182°C to 175°C.On further cooling, the specimen finally entered the crystalline phase.


Figure (1)

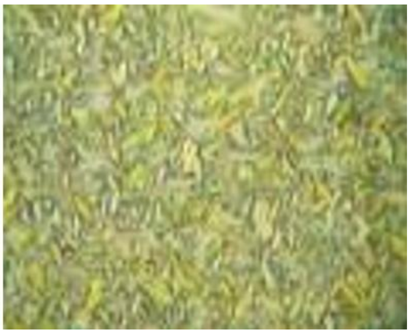


Figure (2)


Figure (3)

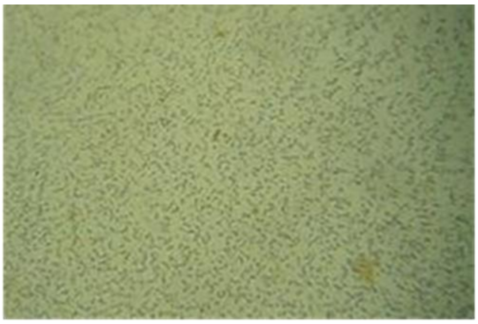


Figure (4)

Figure 1

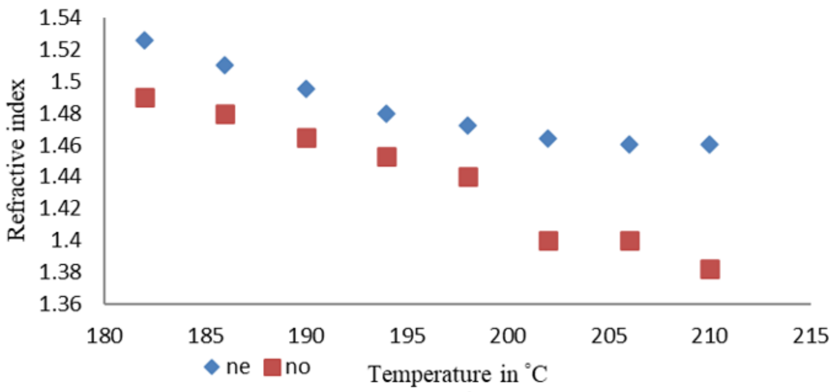
Microphotographs obtained between crossed polars: (a) Spherulitic texture of the cholesteric phase; (b) Composite texture showing isogyres and SmA phase (250×); (c) Texture of the SmB phase (250×); (d) Texture of the SmE phase (250×).

Optical Anisotropy

The temperature variation of refractive indices for the sample consisting of approximately 70% N-Cetyl-N,N,N-trimethylammonium bromide (CTAB) and 30% cholesteryl nonanoate is presented in Graph 1.From Graph 1, it is observed that upon cooling the sample from the isotropic phase, the value of the extraordinary refractive index (n_e) increases non-linearly in the temperature range of 210°C to 182°C. Similarly, the value of the ordinary refractive index (n_o) also increases non-linearly within the same temperature range during cooling.The extraordinary refractive index (n_e) is greater than the ordinary refractive index (n_o) for all temperatures in the range of 210°C to 182°C, indicating the optical anisotropic nature of the binary mixture.The temperature variations of the ordinary and extraordinary refractive indices are listed in Table 1.

Table 1. Temperature variations of ordinary (n_o) and extraordinary (n_e) refractive indices of the binary mixture.

Temperature(°C)	182	186	190	194	198	202	206	210
n_e	1.526	1.510	1.495	1.480	1.472	1.464	1.460	1.460
n_o	1.490	1.480	1.465	1.453	1.440	1.400	1.400	1.382



Graph 1

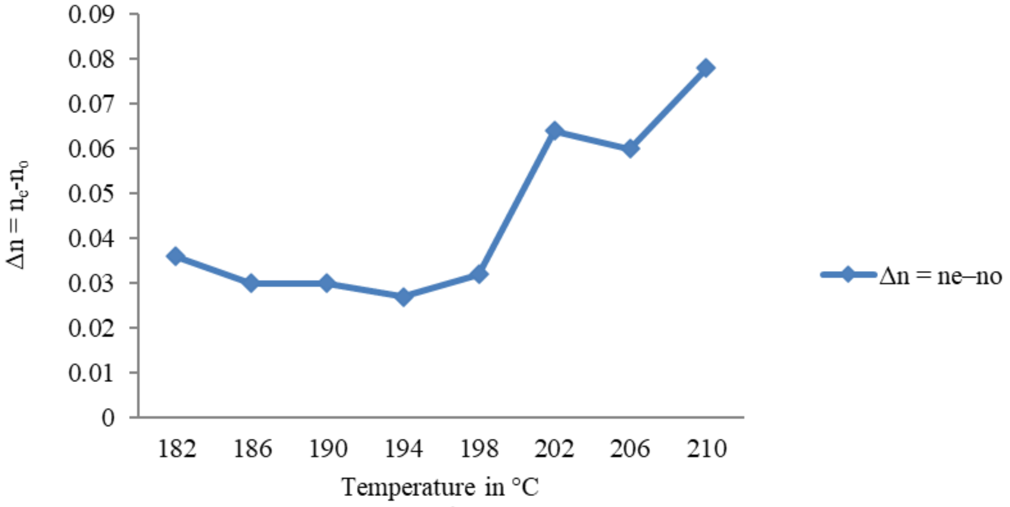
Bireferengence

The temperature variation of birefringence (Δn) for the binary liquid crystal mixture is presented in Table 2.

Table 2. Temperature variations of birefringence (Δn) of the binary liquid crystal mixture.

Temperature(°C)	182	186	190	194	198	202	206	210
$\Delta n = n_e - n_o$	0.036	0.030	0.030	0.027	0.032	0.064	0.060	0.078

The temperature variation of birefringence (Δn) of the binary mixture is shown in Graph 2. From Graph 2, it is observed that the birefringence remains nearly constant, with a value of approximately 0.03, in the temperature range of 182°C to 198°C. At 202°C, the birefringence increases to 0.064 and then decreases slightly to 0.060 at 206°C. Further, the birefringence increases to a maximum value of 0.078 at 210°C. The variation of birefringence with temperature is non-linear, indicating changes in molecular ordering within the liquid crystalline phases.



Graph 2

III. Conclusions

A binary mixture consisting of 70% N-Cetyl-N,N,N-trimethylammonium bromide (CTAB) and 30% cholesteryl nonanoate exhibits cholesteric, SmA, SmB, and SmE phases sequentially when cooled from the isotropic phase. During cooling from the isotropic phase, nucleation initially appears in the form of small bubbles growing radially, which are identified as spherulitic textures characteristic of the cholesteric phase.

The cholesteric phase exists in the temperature range of 210 °C to 175 °C, whereas the SmA, SmB, and SmE phases exist in the temperature ranges of 210 °C to 196 °C, 196 °C to 182 °C, and 182 °C to 175 °C, respectively.

The temperature variation of refractive indices for the same composition shows that both the ordinary refractive index (n_o) and extraordinary refractive index (n_e) increase non-linearly when the sample is cooled from 210 °C to 182 °C. The value of the extraordinary refractive index (n_e) remains higher than that of the ordinary refractive index (n_o) throughout the temperature range, confirming the optical anisotropic nature of the binary mixture.

The temperature variation of birefringence (Δn) of the binary mixture indicates that the birefringence remains nearly constant at approximately 0.03 in the temperature range of 182 °C to 198 °C. At 202 °C, the birefringence increases to 0.064 and then decreases slightly to 0.060 at 206 °C. The birefringence reaches a maximum value of 0.078 at 210 °C. The observed birefringence shows a non-linear variation with temperature, indicating temperature-dependent changes in molecular ordering within the liquid crystalline phases.

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