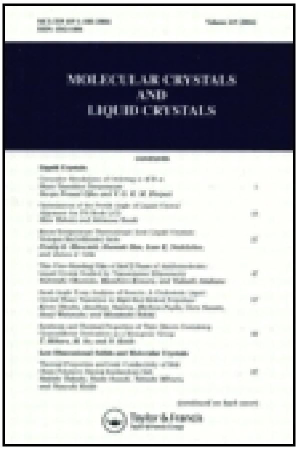

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Synthesis and Characterization of Canola-Based Cholesteryl Ester and Nematic E7 Mixture

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The fatty acid of Canola Oil, 61% of which is Oleic acid, was extracted. Fourier Transform Infrared Spectrophotometry (FTIR) confirmed the fatty acid extraction. Canola-based Cholesteryl Ester (CANCE) was obtained through esterification. FTIR scans of the CANCE and the fatty acid were compared to verify the complete esterification. The resulting crude product is a yellow wax with some crystals embedded on it. Thin layer chromatography was then performed to determine the appropriate solvent for the column chromatography to purify the crude sample. The purified product was characterized using Optical Polarizing Microscopy and Differential Scanning Calorimetry. Mixtures of the Nematic E7 and CANCE in different ratios were prepared. Transition temperatures of each mixture were determined using differential scanning calorimetry and verified using optical polarizing microscopy.

Keywords: synthesis; cholesterics; formulations

INTRODUCTION

The high price of commercially available liquid crystals serves as a barrier to its widespread use for different types of applications. For this very reason, the need to look for sources of liquid crystals that are cheap and easily available arises. The success of the previous attempt to synthesize liquid crystal from coconut oil became a motivation to look for other materials as potential sources. The research yielded a liquid crystal that was solid at room temperature and yet was able to induce chirality on nematic liquid crystals [1].

Esters of cholesterol have been by far, the most widely exploited liquid crystals. Although cholesterol in itself is not a mesomorphic compound, its fatty esters are [2]. In general, mesophase formation of fatty acid esters of cholesterol occurs above 80°C and preparing liquid crystal mixtures that are stable at room temperature from these compounds alone are difficult [3]. These compounds can be synthesized by extracting fatty acids, usually from plant oils, and reacting it with cholesterol.

Compounds that form liquid crystals are generally organic in nature and composed of molecules that are elongated and fairly rigid [4]. Canola oil is known for its high content of 18-carbon chain monounsaturated fatty acid (oleic acid). Oleic acid falls under the long-chain fatty acid group and thus it has a possibility of forming liquid crystal. The fatty acid esters present a typical thermotropic liquid crystal sequence, with chiral nematic phases predominating at short substituent chain lengths and smectic phases becoming increasingly prominent as the chain is extended [5].

METHODOLOGY

Half-liter Sunbeam Canola Oil (Sime Darby Edible Products, Ltd., Singapore) was saponified and yielded a yellow waxy paste. 120 grams of this substance was then dissolved in 100 mL of 40% H_2SO_4. An excess of 10% H_2SO_4 was also added and was left to stand overnight to ensure complete dissolution. The filtrate was washed with distilled water and placed in a separatory funnel to remove the water. $CaCl_2$ pellets were added to completely free the sample of the remaining water. The fatty acid obtained resulted in a colorless crystal and a yellow viscous liquid. FT-IR scans confirmed the liquid part as oleic acid. Reacting with cholesterol, DCC and DMAP esterified the fatty acids. Urea was filtered off and the remaining filtrate was washed alternately with 5% HCl and water thrice. It was air-dried and yielded a yellowish waxy substance with some colorless crystals embedded on it. The product formed from this reaction is the cholesteryl ester in its crude form. Optical microscopy, Differential Scanning Calorimetry, and Fourier Transform Infrared Spectroscopy were then used in its analysis.

Solvent determination was done by Thin Layer Chromatography. The proper solvent for the separation of the crude product was found out to be 2 hexanes: 5 dichloromethane, and was later used in Column Chromatography for its purification. The purified product, which is a white wax, was characterized again by Differential Scanning Calorimetry, Optical Polarizing Microscopy, and Fourier Transform Infrared Spectroscopy. Mixtures of cholesteryl ester and nematic E7 in different ratios were formulated and prepared in glass slides for characterization.

RESULTS AND DISCUSSION

IR analysis was performed on the fatty acid that was extracted. The liquid part was confirmed to be oleic acid. It was also done on the synthesized cholesteryl ester and confirmed its purity.

The purified cholesteryl ester was mixed with a commercially available nematic liquid crystal E7 (MERCK). Sieve-dried dichloromethane was used to dissolve and mix the two components. Among the mixtures prepared the 75CANCE: 25E7 macroscopically exhibits a bright display of colors at room temperature, as shown in FIGURE 1 .

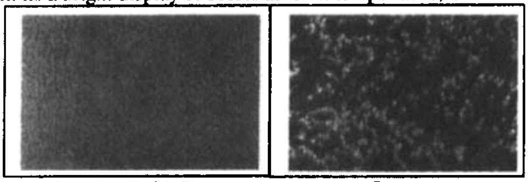


FIGURE 1 (left) Fingerprint texture of 85E7:15CANCE at room temperature (right)

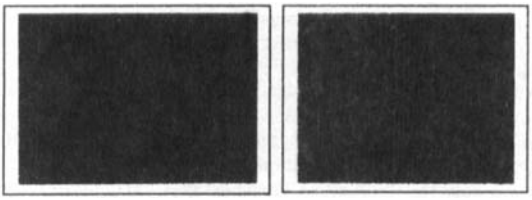


FIGURE 2: (left) grandjean texture of 90CANCE:10E7 at T=37.3°C (right) purified CANCE at T=38.5°C

Thermal analysis of the CANCE using DSC revealed two distinct peaks, one at 38.86°C and the other at 44.47°C. This was verified by observing the ester under a polarizing microscope with an attached heating stage while undergoing heating and cooling (refer to FIGURE 4 and FIGURE 2, right). The figure below is the DSC thermogram of the purified CANCE.

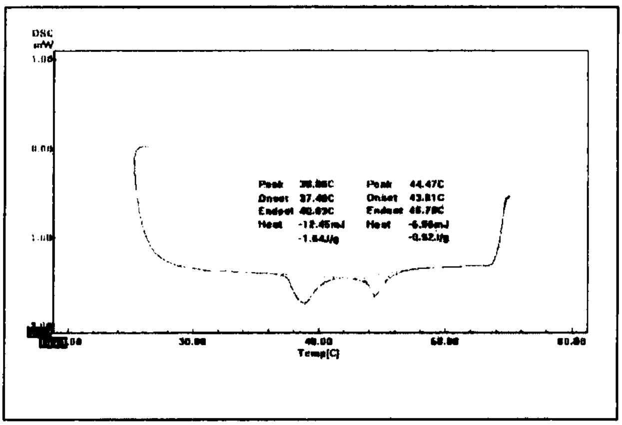
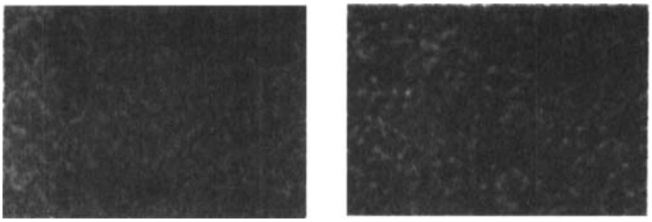


FIGURE 3: Thermogram of the purified CANCE.



a b

FIGURE 4:(a) Purified Canola-based cholesteryl ester at T = 38.5°C (b) Purified Canola-based

The effect of the cholesteryl ester on the nematic liquid crystal was studied by mixing it with a nematic liquid crystal E7. The cholesteryl ester was able to induce chirality on the nematic E7 in some formulations. This was evident in the fingerprint texture exhibited by the 85E7: 15CANCE mixture, the focal conic texture in the 75CANCE:25E7 and the grandjean texture in the 90CANCE:10E7 mixture (FIGURE 1 & FIGURE 2). The clearing temperature, as determined by the DSC thermogram, was decreased, as the mixture becomes less nematic when the ester is added.

SUMMARY

The cholesteryl ester derived from Canola oil was determined to be mesomorphic as evidenced by the two peaks obtained in the DSC thermal analysis. Observation using the polarizing microscope further confirmed the liquid crystal to isotropic transition.

RECOMMENDATION

It is recommended that other verification tests for the synthesized ester be performed. XRD analysis should be done to determine the crystals. It is also suggested that better formulation techniques suitable for electro-optic testing be employed to the samples.

ACKNOWLEDGMENT

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