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Improving the Thermal Stability and Electrical Parameters of a Liquid Crystalline Material 4-n-(Nonyloxy) Benzoic Acid by Using Li Ion Beam Irradiation

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Abstract. Li ion beam irradiation studies on a liquid crystalline material 4-n-(nonyloxy) benzoic acid (NOBA) have been carried out. The material has phase sequence of I-N-SmC-Cr. Thermodynamic studies demonstrate that an irradiation fluence of 1×10^{13} ions-cm^{-2} results in the increased thermal stability of the smectic C (SmC) phase of the material. Dielectric measurements illustrate that the transverse component of the dielectric permittivity and hence the dielectric anisotropy of the material in the nematic (N) and SmC phases are increased as compared to those of the pure material due to irradiation. UV-Visible spectrum of the irradiated material shows an additional peak along with the peak of the pure material. The observed change in the thermodynamic and electrical parameters is attributed to the conversion of some of the dimers of NOBA to monomers of NOBA due to irradiation.

INTRODUCTION

High energy ion beam irradiation have been used for the modifications of various properties such as mechanical, electrical, chemical, optical, thermal and sensing of different types of materials such as long chain polymers, semiconductors, high and low temperature superconductors etc. [1-7]. The damages caused by irradiation are responsible for the changes in physiochemical properties of the irradiated material [6]. The nature of these changes depends on the type of ion, energy and fluence (ions-cm^{-2}) of irradiation. This is an area of research that needs a detailed investigation regarding the impact of high energy ion irradiation on various materials for their applications in the advance field of technologies associated with radiation environment. Liquid crystalline (LC) materials like the polymeric materials are long chain organic materials which are used in every walk of life ranging from laptop screens, dash boards of auto mobiles, displays of space shuttles to wide screen televisions. The devices made up off LC materials are used in high radiation prone environments such as nuclear installation centers, nuclear reactors and in space applications occasionally for long durations. When these devices are used in such an environment, high doses of irradiations affect their physiochemical properties. Therefore, it becomes very important to study how and upto what extent different radiations affect various properties of these materials. However, to the best of our knowledge, there are very few reports available on the effect of irradiation on the properties of LC materials [8-11]. Some of the previous irradiation studies on LC materials suggest that the transition temperatures, enthalpies and entropies are lowered; ac and dc conductivities of the materials are increased [12-13]. In the present work, we report the effect of Li ion beam irradiation (of fluence 1×10^{13} ions-cm^{-2}) on the thermodynamic and electrical properties of 4-n-(nonyloxy) benzoic acid (NOBA). NOBA is the 9th member of the alkyloxy benzoic acid homologues series and has the molecular

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formula and weight of $C_{16}H_{24}O_3$ and 264.36 respectively. The molecular structure of the studied compound is given in Figure 1.

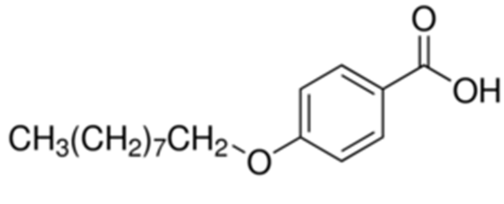


FIGURE 1. Molecular structure of 4-n-(nonyloxy) benzoic acid (NOBA).

EXPERIMENTAL TECHNIQUES

The irradiation experiments were carried out by mounting the materials on a vertical shielding ladder and irradiated in a scattering chamber of material science beam line by 50 MeV Li^{+3} ions available from the 15UD Pelletron Accelerator at Inter University Accelerator Centre (IUAC), New Delhi, India. All irradiations were performed in vacuum (of 10^6 Torr) at ambient temperature. The beam current was kept below 3 pnA. The ion beam fluence was varied in the range 5×10^{10} to 1×10^{13} ions-cm^{-2}. In order to expose the target 1.5 cm × 1.5 cm areas, the beam was scanned in the x-y plane.

Pure and irradiated materials are characterized by Differential Scanning Calorimeter (DSC) and UV-Visible spectroscopy. The thermodynamic study of the pure and irradiated materials has been carried out on a DSC of NETZSCH model DSC-200-F3-Maia. Different phases of the pure and irradiated materials have been identified from the texture study with the help of a CENSICO made Polarized Light Microscope (PLM). Optical absorption spectra were recorded at room temperature for both pure and irradiated materials using a UV-visible spectrophotometer (Ocean Optics) of model DH-2000 coupled with a detector of model HR-4000-CG-UV-NIR in the wavelength range 300-1100 nm.

For the dielectric measurements electrical cells in the form of parallel plate capacitor were prepared using ITO and gold coated glass plates having sheet resistance less than 25 Ω, as electrodes, using 10 μm and 40 μm thick spacers. The active capacitance (C_L) was determined by using organic liquid of known dielectric permittivity, in this case cyclohexane, into the cell. Capacitance (C) and conductance (G) of the cell filled with material were determined in the frequency range 1 Hz to 35 MHz using N4L's phase sensitive multimeter model PSM-1735 coupled with impedance analysis interface model IAI-1257. A measuring electric field of 0.5 V_{rms} has been applied across the material. Acquired data of C and G were used to determine the frequency dependent dielectric permittivity ($\varepsilon'_\perp$) and dielectric loss ($\varepsilon''_\perp$) as follows:

$$\varepsilon'_\perp = \left(\frac{C_m - C_a}{C_L}\right) + 1 \tag{1}$$

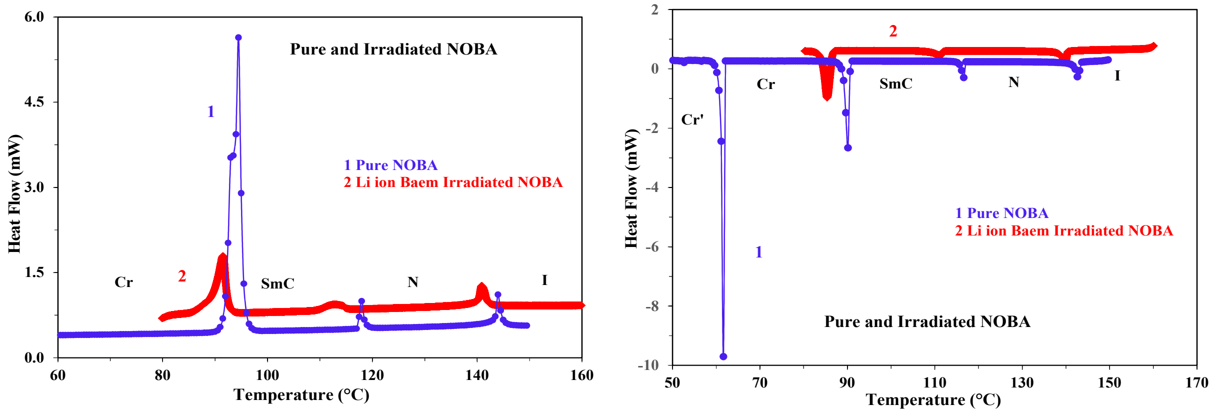
$$C_L = \left(\frac{C_l - C_a}{\varepsilon_l - 1}\right) \tag{2}$$

$$\varepsilon''_\perp = \frac{\sigma}{\varepsilon_0 \omega} = \left(\frac{G_m - G_a}{\omega C_L}\right) \tag{3}$$

where C_m and G_m are the measured capacitance and conductance of the cell filled with material, C (a) and G (a) are the measured capacitance and conductance of the cell without any material i.e. with air, C_l is the measured capacitance with standard liquid (in this case cyclohaxane) in the cell and ε_l is the dielectric permittivity of the standard liquid filled in the cell. Further details about the experiments can be found in our earlier publications [12-13]

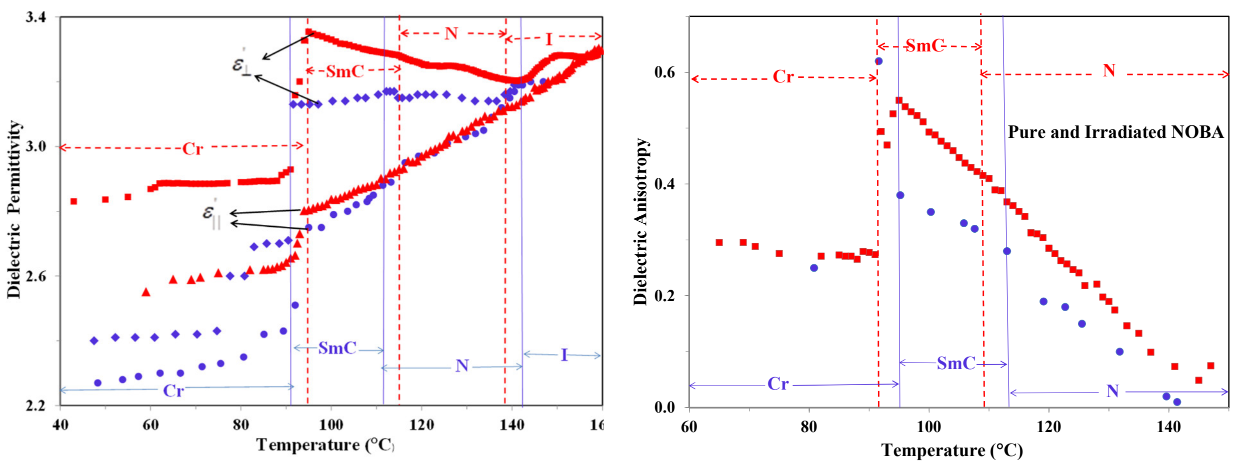
RESULTS AND DISCUSSION

Figures 2 show the variation of the heat flow (mW) with scan rate (°C/min) of the pure and 1×10^{13} ions-cm^{-2} irradiated NOBA. From the figure, it is evident that in the heating cycle of the pure and irradiated materials, Cr-SmC, SmC-N and N-I transitions are observed. In the cooling cycle, for the pure materials, I-N, N-SmC, SmC-Cr and Cr-Cr' transitions are observed while for the irradiated material I-N, N-SmC and SmC-Cr transitions are observed. From the comparisons of the thermodynamic data of the pure and irradiated materials, it has been observed that all the transition temperatures except that of the SmC-N-SmC of the irradiated material are decreased as compared to those of the pure material. It has been also observed that the range of SmC phase of the material in the heating and cooling cycles has been increased from 20.7 °C to 21.8 °C and 24.8 °C to 27.6 °C respectively. From the comparisons of the thermograms of the pure and irradiated materials, it has been also observed that all the transition processes are slowed down due to irradiation as evidenced from the peaks of different transitions of the pure and irradiated materials.



(a) **(b)**

FIGURE 2. DSC thermograms for the pure and irradiated (1×10^{13} ions-cm^{-2}) NOBA at the scan rate of 5.0 °C/min (a) Heating cycle, (b) Cooling cycle.



(a) **(b)**

FIGURE 3. (a) Variation of the longitudinal ($\varepsilon'_{\parallel}$) and transverse ($\varepsilon'_{\perp}$) components of the dielectric permittivity of the pure and 10^{13} ions-cm^{-2} ion beam irradiated NOBA in the cooling cycle. Rhombus (blue) & circles (blue) and squares (red) & triangles (red) are representing the pure and irradiated data respectively. (b) Variation of dielectric anisotropy ($\Delta\varepsilon = (\varepsilon'_{\perp} - \varepsilon'_{\parallel})$) of the pure and 10^{13} ions-cm^{-2} ion beam irradiated NOBA in the cooling cycle. Circles (blue) & squares (red) are representing the pure and irradiated data respectively. Solid and broken vertical lines and arrows are used to show the ranges of different phases of the pure and irradiated material respectively.

The dielectric permittivity of the pure and irradiated materials has been measured in two different geometry of molecules (parallel ($\varepsilon'_{\parallel}$) and perpendicular ($\varepsilon'_{\perp}$) to the direction of the measuring electric field). FIGURE 3 (a) show the variation of the longitudinal ($\varepsilon'_{\parallel}$) and transverse ($\varepsilon'_{\perp}$) components of the dielectric permittivity of the pure and 1×10^{13} ions-cm^{-2} Li ion beam irradiated NOBA in the cooling cycle. The values of $\varepsilon'_{\parallel}$ and $\varepsilon'_{\perp}$ are in good agreement with the literature data [14]. From the comparison of the dielectric data of the pure and irradiated materials, it has been observed that the transverse component of the dielectric permittivity in the nematic and SmC phase is increased as compared to that of the pure material due to irradiation. The value of the longitudinal component of the dielectric permittivity in the N and SmC phase is almost unchanged due to irradiation.

FIGURE 3 (b) show the variation of dielectric anisotropy ($\Delta\varepsilon = (\varepsilon'_{\perp} - \varepsilon'_{\parallel})$) of the pure and 1×10^{13} ions-cm^{-2} Li ion beam irradiated NOBA in the cooling cycle. It has been observed that the nature of the dielectric anisotropy of the pure material is intact due to irradiation. From the comparison, it is also evident that the dielectric anisotropy of the pure material is increased due to irradiation as compared to that of the pure material. In the UV-Visible spectra of the 1×10^{13} ions-cm^{-2} Li ion irradiated data (not reported here) an additional peak is observed with the pure material peak which suggest the formation of new species due to irradiation. The increase in the value of the dielectric permittivity and anisotropy can be explained in the following way. 4-(n-Alkyloxy) benzoic acid (nOBA) consists of molecules dimerized by hydrogen bonding [14]. Due to irradiation, hydrogen bonding of some of the dimers is broken and due to this they are converted to monomers. Petrov *et. al.* have shown that monomers have tendency to be aligned normal to the layer plane [15]. Due to this, the transverse components of the irradiated material as compared to those of the pure material are increased due to irradiation.

CONCLUSIONS

The transition temperatures of all the phase transitions except the N-SmC have been decreased due to irradiation. The peaks of all the transitions for the irradiated materials are becoming wider and shorter as compared to those of the pure material. This suggests that the transition process is becoming slower due to irradiation. The value of the transverse component of the dielectric permittivity and hence dielectric anisotropy are increasing because of the dangling of some of the hydrogen bonding of the dimers of NOBA due to irradiation.

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