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Annealing Effects on Dielectric Constant/Dielectric Loss Measurements in Vectra-A, Liquid Crystal Copolyester

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Abstract

The strengthening consequences for dielectric constant (ε') and dielectric loss (ε''), for Vectra-A samples, after annealing, fluctuating with temperature (25°C to 250°C) at various frequencies (1 kHz to 1 MHz), have been contemplated. In annealing phenomenon, the dielectric properties decrease due to transition of amorphous phase to crystalline phase. The dielectric constant (ε') is very small in annealed samples of Vectra-A in contrast to pristine samples. The magnitude of β-relaxation becomes small in variation of dielectric loss (ε'') with temperature because the dipolar relaxation reduces due to enhancement in crystallinity.

Keywords: *Vectra-A, liquid crystal copolyester, dielectric spectroscopy (DS) dielectric constant (ε'), dielectric loss (ε'')*

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INTRODUCTION

Polymer due to their conducting behavior with various doping level and enhanced thermal stability are used in charge storing devices like micro-electro-mechanical systems (MEMS) [1–2]. Organic polymers have applications in electrical insulating materials and power supply industries [3]. After doping with iodine vapors, Vectra-A, liquid crystal copolyester possess conducting properties [4–5]. The thermal stability of Vectra-A was also investigated by annealing it for different times [6]. Annealing of the polyesters in their mesophase region result in an enhanced structural arrangement [7]. Kaito et al. studied the effect of annealing on structure formation of a liquid crystalline copolyester and analysis of the ordered structure developed by annealing [8].

The HNA and HBA relative motions were shifted to higher temperatures while the intensity of the HNA motion was increased in annealed materials [9].

Dielectric response provides a lot of information about various molecular motions and relaxation mechanisms. An exclusive characteristic of dielectric spectroscopy (DS) is the wide-ranging frequency to which the polymer responds to an electric field [10–11].

In dielectric spectroscopy technique, the dielectric constant/dielectric loss is measured as function of frequency and temperature. The dynamic mechanisms can be recognized by their characteristics frequency of relaxation [12]. The present work shows the effect of annealing on dielectric constant and dielectric loss of samples Vectra-A by comparing them with pristine samples of Vectra-A.

MATERIAL AND METHOD

The Vectra-A, ivory in color, is procured in the form of rod having diameter 18 millimeter from Good fellow, England. The glass transition temperature of Vectra-A is around 110°C and its melting point is 280°C. It is copolyester of polyhydroxybenzoic acid, PHBA and poly(naphthoic acid), PHNA. In Vectra-A, the incorporation of 2,6-naphthalylene moieties in 4-hydroxybenzoic acid results in the structural defect and reduction in rigidity of chain. Naphthalene modifiers bring the desired characteristics to the poly(hydroxybenzoic acid) molecule that is reduction in the melting temperature and the addition of naphthalene group to hydroxybenzoic group does not change its mechanical, thermal and chemical-resistant properties. The dielectric measurements were made at frequencies ranging from 1 kHz to 1 MHz using Agilent 4284 A, a precision LCR meter. The sample placed in two-probe setup was kept in a digital temperature controlled furnace for heating in steps, i.e., at a certain temperature sample was kept for almost 10 minutes and the corresponding measurements of capacitance (C) and dissipation factor (D) were made.

RESULTS AND DISCUSSION

Effect of Annealing

The temperature dependence of dielectric constant (ε′) and dielectric loss (ε″) of pristine and annealed Vectra-A samples in frequency range (1 kHz–1 MHz) is illustrated in Figures 1–8. The samples were annealed at 100°C for 5 hours and 50 hours. The annealed samples of Vectra-A for 5 hours, shows a decrease in dielectric constant (ε′) in comparison to pristine samples below 50°C. In the annealed samples

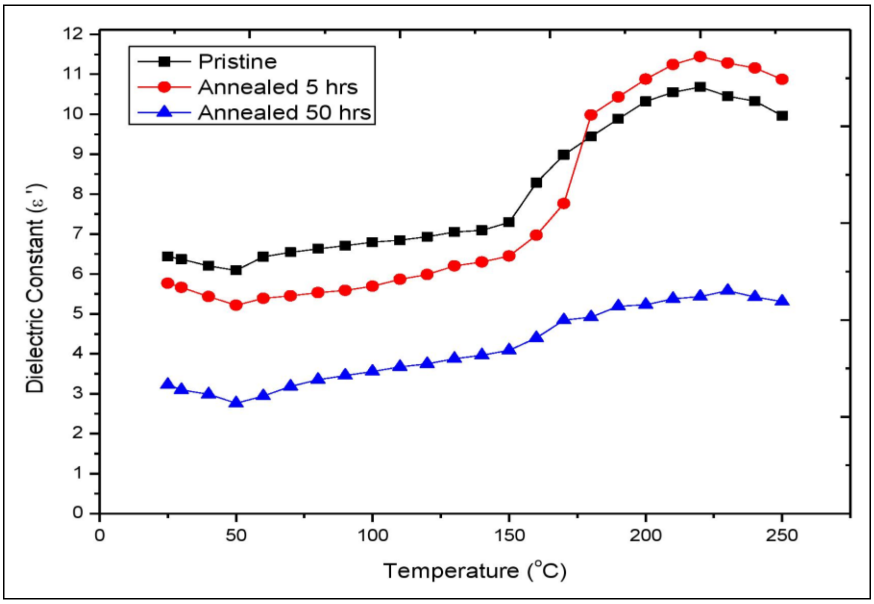

Fig. 1: *Variation of Dielectric Constant (ε') with Temperature for Pristine and Annealed Vectra-A Samples at Frequency 1 kHz (Annealed at 100°C).*

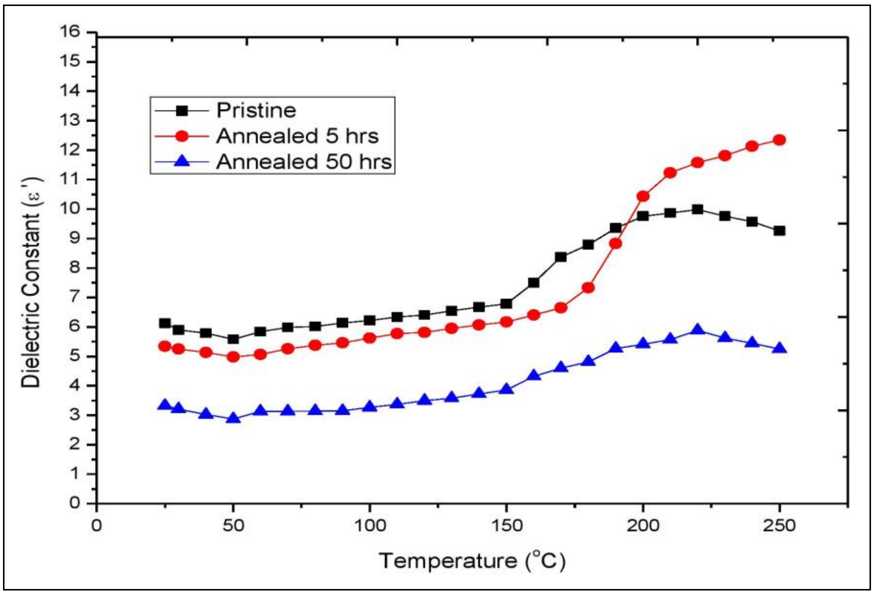

Fig. 2: *Variation of Dielectric Constant (ε') with Temperature for Pristine and Annealed Vectra-A Samples at Frequency 10 kHz (Annealed at 100°C).*

of Vectra-A (5 hours), up to 50°C, dielectric constant (ε′) decreases as compared to pristine samples. Above 50°C, dielectric constant (ε′) increases with temperature. Also, in the temperature interval (180°C–220°C), the annealed samples has greater value of dielectric constant (ε′) than the pristine samples. This transition temperature increases with the increase in frequency. As the frequency increases, the heavy dipoles are not able to respond this frequency and do not contribute to dielectric constant. Therefore, the dielectric constant decreases and results in rise in transition temperature. Further, annealing of Vectra-A samples for 50 hours, results in reduced dielectric constant (ε′). There is occurrence of multiple transition processes as a result of annealing. During annealing process, there is transition from amorphous phase to crystalline phase and hence dielectric constant (ε′) decreases [13–16].

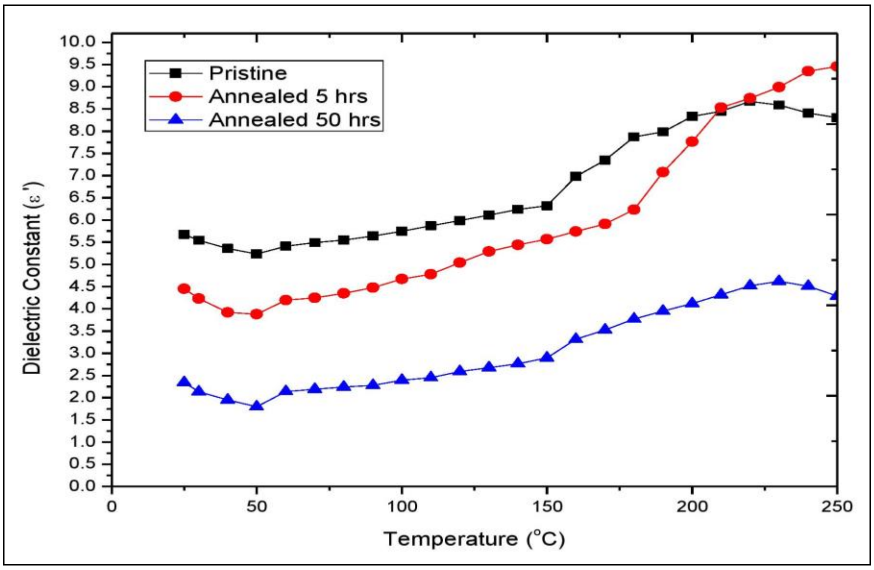

Fig. 3: *Variation of Dielectric Constant (ε') with Temperature for Pristine and Annealed Vectra-A Samples at Frequency 100 kHz (Annealed at 100°C).*

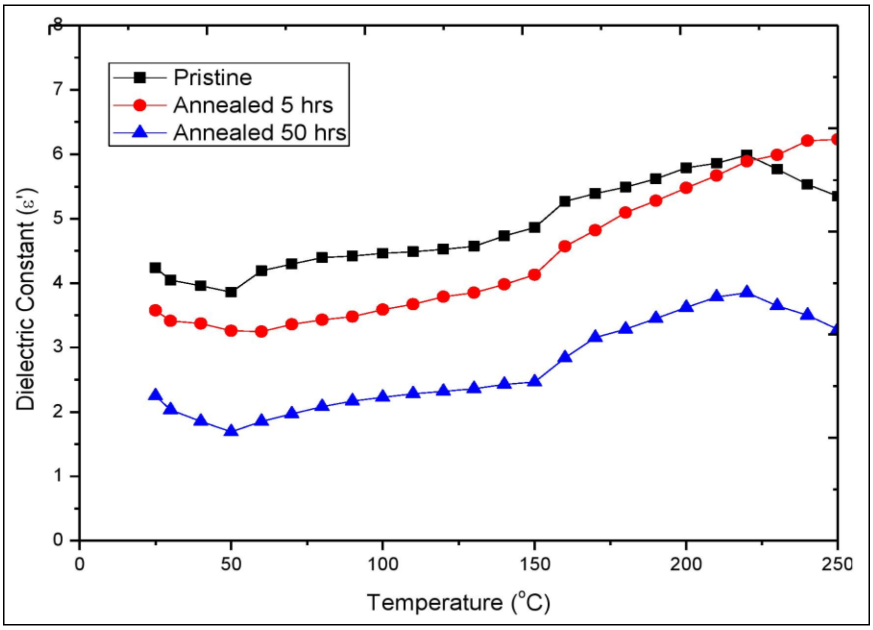

Fig. 4: *Variation of Dielectric Constant (ε') with Temperature for Pristine and Annealed Vectra-A Samples at Frequency 1 MHz (Annealed at 100°C).*

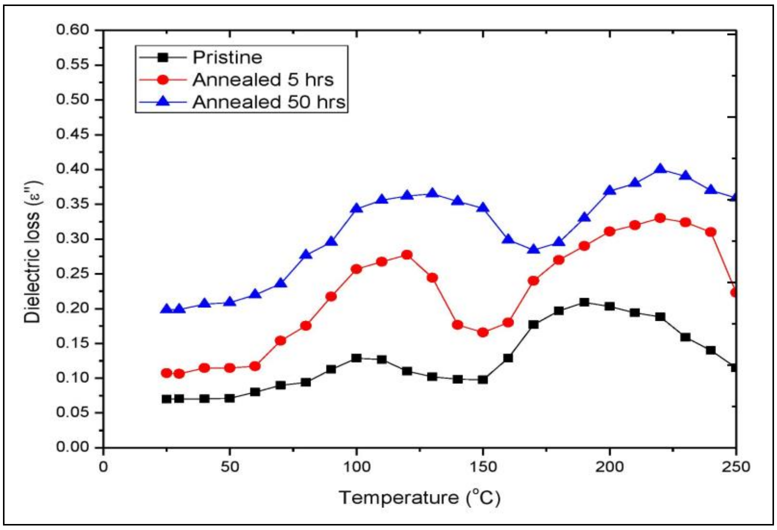

Fig. 5: *Variation of Dielectric loss (ε'') with Temperature for Pristine and Annealed Vectra-A Samples at Frequency 1 kHz (Annealed at 100°C).*

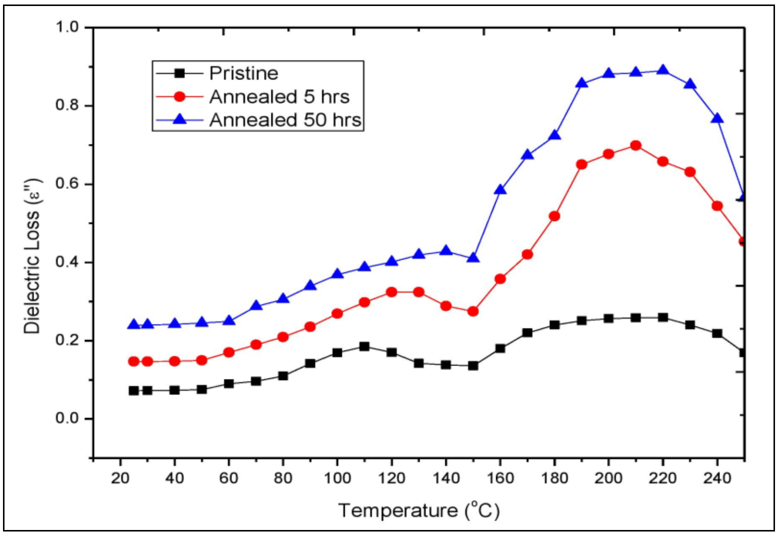

Fig. 6: *Variation of Dielectric Loss (ε'') with Temperature for Pristine and Annealed Vectra-A Samples at Frequency 10 kHz (Annealed at 100°C).*

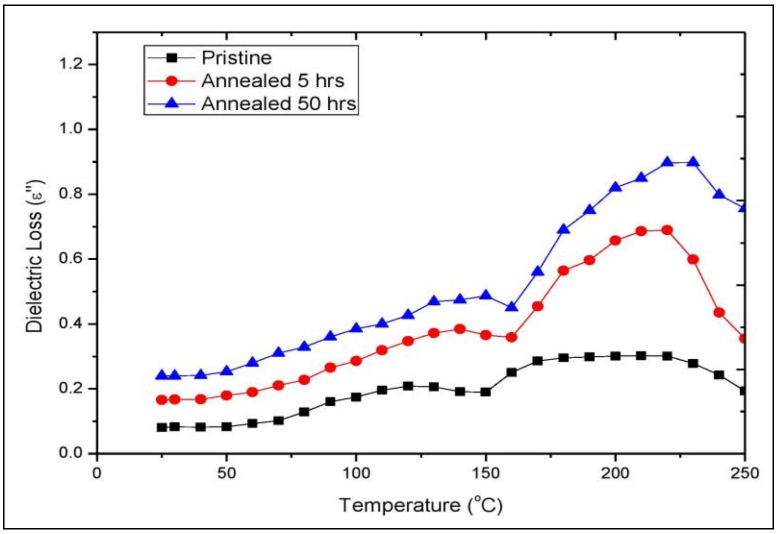

Fig. 7: *Variation of Dielectric loss (ε'') with Temperature for Pristine and Annealed Vectra-A Samples at Frequency 100 kHz (Annealed at 100°C).*

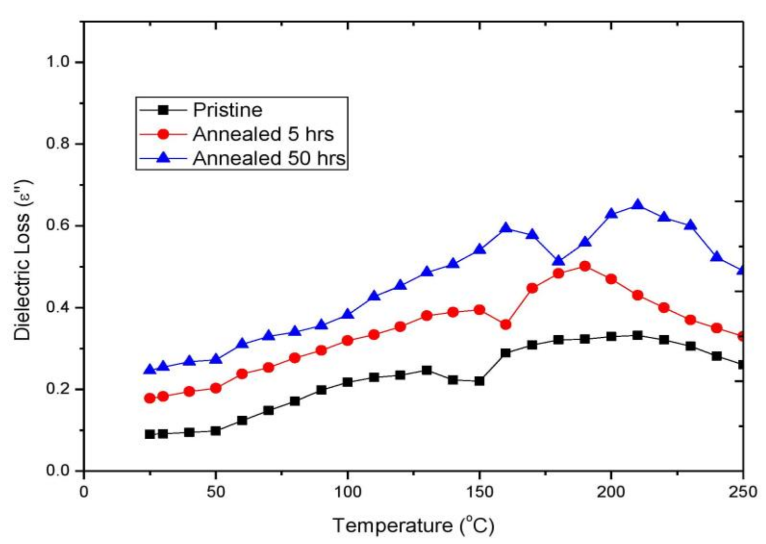

***Fig. 8:** Variation of Dielectric loss (ε′′) with Temperature for Pristine and Annealed Vectra-A Samples at Frequency 1 MHz (Annealed at 100°C).*

In the variation of dielectric loss (ε′′) with temperature for pristine and annealed samples of Vectra-A at frequency range (1 kHz–1 MHz), the β-peak is suppressed because enhancement in crystallinity reduces the dipolar relaxation. The dipolar relaxation due to main chain orientation is observed near 110°C and the position of peak shifts towards higher temperature with increase in frequency and annealing time because of cooperative motion. As the frequency is increased there is decrease in dielectric loss as happens in the variation of dielectric constant with frequency because of low response of dipole to high frequency and also due to dielectric dispersion [17]. Above 150°C space charge peak is observed that is followed by melt region of Vectra-A. The distributive relaxation behavior of Vectra-A is also confirmed from spectra of dielectric loss with temperature as is observed in case of variation of dielectric constant (ε′) with temperature at variable frequency.

CONCLUSION

The impact of annealing on dielectric behavior of annealed samples of Vectra-A with temperature at various frequencies, have been examined. In the annealed samples of Vectra-A, the dielectric constant (ε') is very small in contrast to pristine samples. In annealing phenomenon, the dielectric properties decrease due to transition of amorphous phase to crystalline phase. The β-relaxation suppresses in variation of dielectric loss (ε′′) with temperature because the dipolar relaxation reduces due to enhancement in crystallinity.

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