

Neutron irradiation effect on dielectric loss of bi-stretched polyethylene naphthalate

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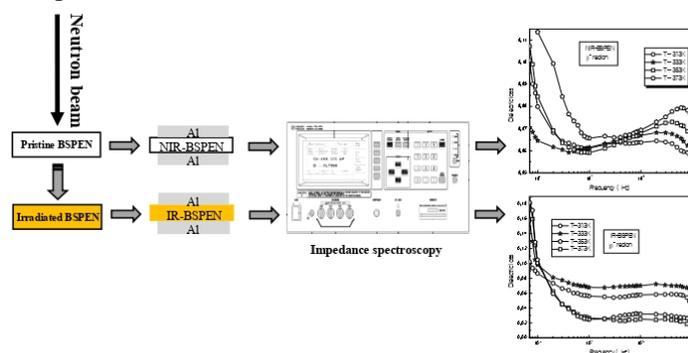
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ABSTRACT

In the present work, the study of the evolution of the β^* process in both pristine and neutron irradiated bi-stretched polyethylene naphthalate films, is realized at a constant temperature and in the frequencies domain comprised between 10^2 and 10^6 Hz by using the impedance spectroscopy technique. The results show the presence of dielectric loss peak determined in 10^5 to 10^6 Hz frequency range, where the shape, position and the intensity are affected by morphological damage due to the large quantity of neutron beam energy transferred to the polymeric molecules that were highlighted by different structural analysis such as the Fourier transform infrared spectroscopy (FTIR) and the polarized light optical microscopy (PLOM). At room temperature, the FTIR technique showed an increase in the quantity of the carbonyl and naphthalene groups responsible of the presence of the Sub-Tg dielectric relaxation in the volume of the irradiated BSPEN (IR-BSPEN) film due to the amorphization phenomena traduced, respectively, by the absence and presence of the absorption bands characteristics of crystalline and amorphous phases. The PLOM characterization, revealed change in the texture of the IR-BSPEN surface indicating by the appearance of microcracks which contribute to the oxidation of macromolecular chains. The study of the effect of temperature on the dielectric behavior of non irradiated BSPEN film, in β^* region, showed that the dielectric parameters such as the maximum of dielectric losses and its position are thermally activated. This dielectric response has not identified in the case of irradiated BSPEN film, for which the evolution of losses with temperature change above $T = 333K$.

Keywords: Bi-stretched polyethylene naphthalate; Neutron irradiation; Morphology modification; Dielectric loss.

Graphical abstract



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1. Introduction

The polyethylene naphthalate (PEN) is a linear thermoplastic polyester resulting from the assembly of elementary molecules, in chains, called monomers. The monomer unit of PEN contains an ester group, giving a polar character to the material, and two aromatic rings, naphthalene group, which give the chain thermal stability, with a melting temperature (T_m) of 538K and glass transition temperature (T_g) of 393K [1-3]. PEN finds a wide field of application in industry, it is used as a magnetic recording medium thanks to its excellent dimensional stability even in the form of a very thin film. As a diffusion barrier, food and medical packaging have benefited from its low permeability, to oxygen among others and as an organic scintillator for radiation detection [4]. In the field of electrical engineering, in the form of thin ($<1\mu\text{m}$) bi-stretched films, it occupies an important place in certain applications. It is used as a class F insulator (operational up to 428K) allowing the wrapping of conductors in electric motors. Its characteristic temperatures have also enabled the development of capacitors called Chips. These miniaturized capacitors can be surface mounted using surface mounted technology (SMT). This implies the resistance of the material to a 10-second pulse at 533K, which was unachievable with other polyesters. The current trend in the field of electronics towards the miniaturization of systems is therefore ensured by the use of this type of material [5].

However, like all polymers, PEN is exposed to different constraints (irradiation, temperature, humidity, pollution, etc.). These constraints can cause irreversible damage. The results reported in the literature have shown that ionizing radiations can cause a loss of physical, mechanical and electrical properties [6].

If an electric field is applied to a molecular system, all the dipoles taken individually tend to orient themselves in the direction of the field. However, since the molecules are in thermal interaction, the molecular agitation tends to oppose the orientation of the dipoles due to the field. The transition from the non-polarized state in the absence of the electric field to a polarized state in equilibrium with the field is not an instantaneous phenomenon. Indeed, a certain delay in the establishment of polarization occurs because of the inertia of the movement of the dipoles. this phenomenon is called dielectric relaxation [7].

The study of the dielectric properties of PEN displays that over a wide range of frequency, passes from 10^{-1} to 10^6 Hz, and temperature between 173K and 473K, the dielectric loss spectra of poly(ethylene 2,6- naphthalene dicarboxylate) (PEN) showed the apparition of three relaxation modes. In order of decreasing temperature, the relaxation processes were observed: the α -relaxation due to the micro Brownian motions of the chain segments, the β^* -process attributed to the molecular motions of aggregates of the naphthalene rings and the β -relaxation assigned to the local fluctuations of the ester groups [8-9].

Impedance spectroscopy is a technique which consists of applying a sinusoidal alternating voltage across a material in as wide a frequency range as possible and to determine the real and the imaginary part of the complex permittivity. It must be noted that, when the frequency of the field increases, we note around certain particular values of frequency the reductions in dielectric constant ϵ' , real part of the dielectric permittivity. For these frequencies there is a phenomenon of resonance, and the frequency of the field is equal to the natural frequency of vibration of the polarizable units of the type in question. This results in a maximum exchange of energy between the electric field and the medium, which has the effect of causing a significant dielectric loss. This is represented by the imaginary part of the complex permittivity, or dielectric loss ϵ'' , which presents the maximum at the frequencies considered [10].

The aim of our work is to study the effect of neutron irradiation on the dielectric properties of bi-stretched polyethylene naphthalate by using the impedance spectroscopy technique, at a constant temperature, for the frequencies ranging from 10^2 to 10^6 Hz.

2. Materials and Methods

The PEN used in this work, Teonex® Q51 type, was provided by DuPont Teijin Films (Luxembourg). It is a bi-stretched oriented PEN film (BSPEN) produced by a stenter process. It must be signaled that, the as-received BSPEN, is noted in this work, NIR-BSPEN and the irradiated film is designated by IR-BSPEN.

2.1. Irradiation process

The irradiation were performed at NUR research reactor, Nuclear Research Center of Draria, Algiers, Algeria. The samples were inserted in aluminum capsules and then placed in the irradiation position where the temperature is about 40 °C. The used reactor power is of 1 MW. In this study, the PEN films have been exposed to fast and thermal neutrons. Thermal neutrons fluence (F_{t-n}) and fast ($E_n > 1$ MeV) neutrons fluence (F_{f-n}) applied are, respectively, $F_{t-n} = 3.8 \times 10^{17}$ n/cm² and $F_{f-n} = 4 \times 10^{17}$ n/cm². The samples are also submitted simultaneously to gamma ray with a dose of about 28 MGy.

2.2. Impedance spectroscopy

In our study, An Agilent HP 4284A precision LCR meter was used, at a voltage of 1V, to determine the capacitance C and the dielectric loss in a frequency range from 10² to 10⁶ Hz. Dielectric measurements above room temperature were carried out in a temperature chamber (NUVE design. model FN 400/500)

2.3. Analysis

The infrared spectra of samples were taken using Thermo fisher FT-IR spectrometer at room temperature, without utilizing KBr, in the 400-4000 cm⁻¹ region. The observation of the non-irradiated BSPEN film surface was made by using an optical microscope with polarized light Zeiss Axio Imager A2 model's coupled to a digital camera Axiocam MRC5 and image software Axiovision 4.6 type. The surface micrograph of the irradiated BSPEN film was made by using the optical microscope with polarized light coupled to the spectrometer RENISHAW in VIA Raman.

3. Results and Discussion

The isothermal responses, at room temperature, of both dielectric constant (ϵ') and dielectric loss (ϵ'') versus the frequency, of NIR-BSPEN and IR-BSPEN films, are given in Fig. 1.

Fig. 1 (a) and (b) show the $\epsilon'(f)$ and $\epsilon''(f)$ spectra of, respectively, NIR-BSPEN and IR-BSPEN films. For both films, the function $\epsilon'(f)$ is decreasing in the increasing direction of the frequency. This can be easily explained by considering that the dielectric spectrum is a superposition of several dipole relaxations. For the analysis frequency range (10² to 10⁶Hz), only subglass relaxation processes, β^* and β , play a role, i.e. the measured dielectric characteristics are governed by the movement of short dipole groups, namely the naphthalene cycle and ester group [8, 9, 11]. From the spectra given above, the impedance spectroscopy shows a presence of relaxation peaks at 6×10^4 Hz and 3×10^5 Hz for, respectively, NIR-BSPEN and IR-BSPEN films.

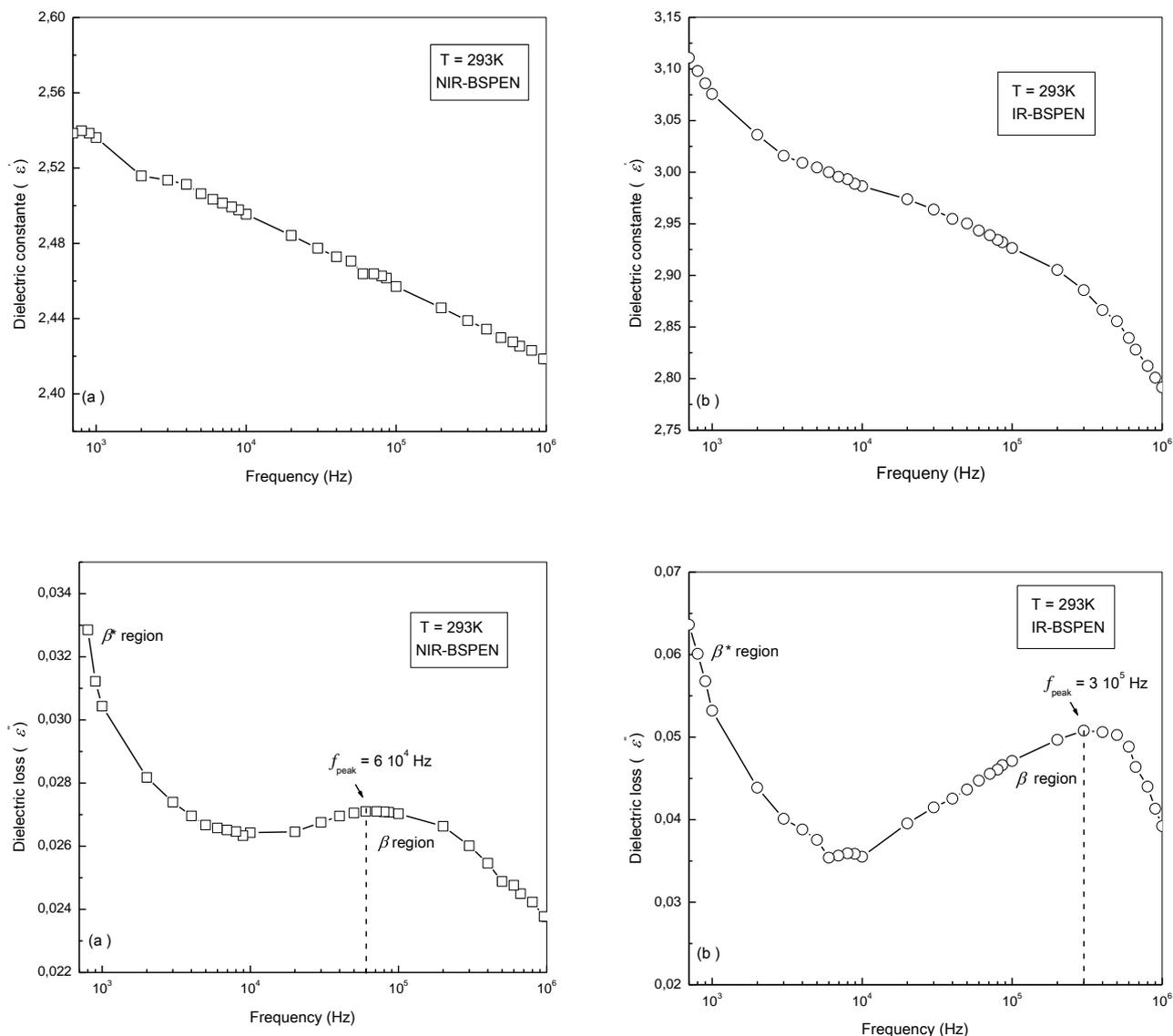


Fig 1. Dielectric constant and loss as a function of frequency in the region intermediate to the β and β^* relaxation. (a) NIR-BSPEN film; (b) IR-BSPEN film.

However, the maximum losses amplitude of IR-BSPEN, in β region, is higher than the one registered in the case of NIR-BSPEN. Also, at the corresponding frequency, the drop of the dielectric constant ϵ' is accentuated. This is due to the structural changes created in the matrix of the polymer and which is traduced by the increase of the intensity of the IR-bands attributed to the carbonyl groups and to the out-of plane movement of the naphthalene groups or its aggregates. Indeed, according to the Fig. 2, giving the absorbance bands in FTIR spectra, between 400 and 1800 cm^{-1} , we remark that the quantity of the dipolar groups responsible of the existence of the dielectric relaxation phenomena, cited above, increases in the IR-BSPEN case. As indicated in Table 1, the band at 762 cm^{-1} is attributed to the vibrations, outside the naphthalene plane, of the aromatic CHs. The band associated with the symmetrical stretching of the group (C-O) is recorded at 1080 cm^{-1} . The peaks at 1128 and 1178 cm^{-1} are assigned to the naphthalene ring vibration. At 1243 cm^{-1} appears the band due to the vibration of the naphthalene ring and to the symmetrical stretching of the C-O group. The band characteristic of carbonyl group (C=O) is registered at 1708 cm^{-1} . The assignments of all these bands are based on the works established by several researchers such as Ouchi et al., Kimura et al., Hardy et al., Zhang et al. and Lee et al. [12–16].

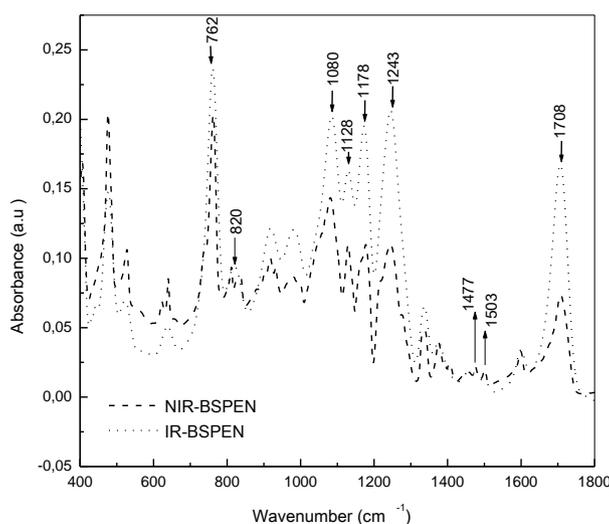
Fig 2. FTIR spectra in the range 400-1800 cm^{-1} .

Table 1. IR bands assignment of BSPEN films

Wavenumber (cm^{-1}) ^a	Band assignment ^b
762	δ (arom. CH out of plane)
932	δ (arom. CH out of plane)
1080	ν_s (C-O)
1128	Naphthalene ring vibration
1178	Naphthalene ring vibration
1243	ν (C-O) + arom ring vibration
1708	ν (C=O)

^aThe wave numbers represent the peak position in absorbance IR spectra.

^b ν : stretching, ν_s : symmetry stretching, δ : deformation.

It can be observed, from Fig. 2, the amorphization of the BSPEN due to the neutrons irradiation [17]. This result is indicated by the appearance, for instance, of the band at 820 cm^{-1} , characteristic of the amorphous phase, which is attributed to the sway of the group (CH_2) in *trans* conformation and the extinction of the IR peaks registered at 1477 cm^{-1} and 1503 cm^{-1} which are assigned, respectively, to the deformation of (CH_2) group in *trans* conformation and the vibration of naphthalene ring in the crystalline phase [5]. In addition, and from the results obtained by Hardy et al. [9], on the study of the dielectric and dynamic mechanical relaxation behavior of semicrystalline oriented PEN films, an increase of the amplitude of the relaxation is due to the release of the mobility which took place following the disorientation of the amorphous phase leading an increase of the β peak.

Fig. 3 gives surface micrographs of the NIR-BSPEN and IR-BSPEN films made under polarized light optical microscope.

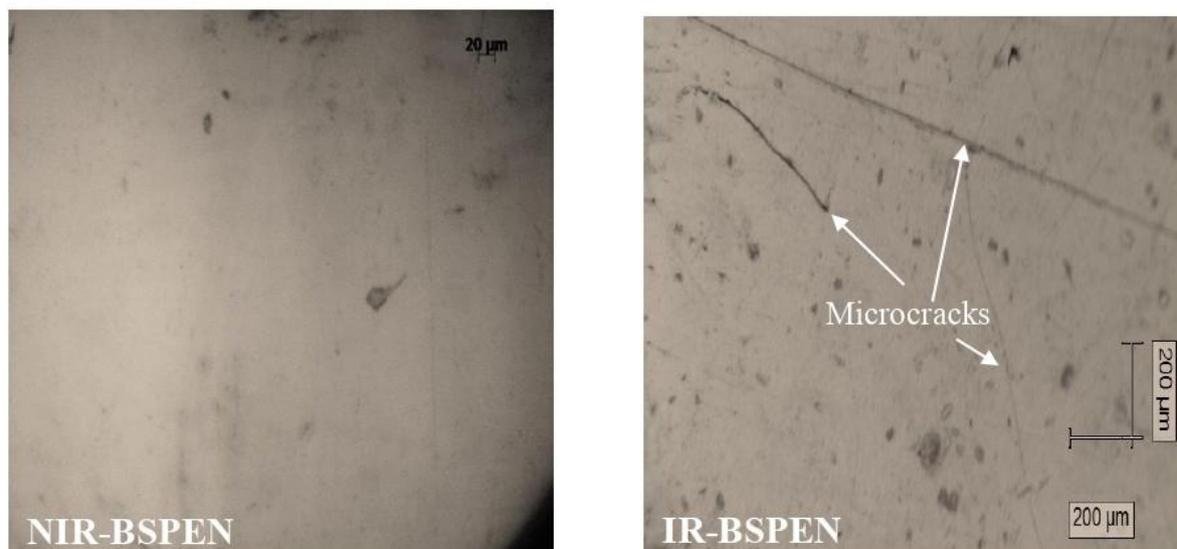


Fig 3. Optical micrographs of the BSPEN surface before and after neutron irradiation with magnification x20.

It must be signaled that this technique permits, in one hand, to provide the information about the surface modifications when the polymer film is subject to different constraints such as chemical environment, heat treatment, mechanical impact and irradiation [18-22]. Indeed, the Fig. 3 shows that the surface of IR-BSPEN film becomes rougher, in the presence of scratches which appear under polarized light optical microscopy in the form of microcracks [17]. Awaja et al. [23] and Pangs et al. [24] in their studies on the cracks and microcracks in polymer have signaled that these last constitute the principal cause of the materials failure in reason of the induced structure fragmentation which conduct to the destruction of their physical properties such as mechanical and electrical. They also showed that microcracking provided opportunities for moisture and oxygen to reach the polymer matrix. This confirm the oxidation of macromolecular chains in irradiated BSPEN films deduced above by FTIR analysis.

Moreover, different relaxation processes in PEN have been highlighted by thermomechanical stimulation and dielectric measurements [25, 26]. The first process named β has been located around 323K, it has been attributed to the non-cooperative molecular movements of some parts of the main chain [27]. The second process β^* occurs, in the case of PEN, around 338K, it has been attributed according to the work of Blundell et al. [28], on liquid crystal polyesters containing 2,6 naphthyl groups, to the cooperative movements of the naphthalene rings with the neighboring carbonyl groups. On the other hand, Menegotto et al. [29] have shown the presence of two peaks named respectively, β_1 and β_2 , and this in their studies carried out on by dielectric spectroscopy and by the technique of thermally stimulated currents. The first peak β_1 , equivalent to the β^* process, was associated with phenyl rings, while the second, corresponding to the β process, was associated with carbonyl groups. Other researches on the study of relaxation phenomena in polyethylene naphthalate with different morphologies have also identified two processes existing below the glass transition temperature, β and β^* , associated respectively with the movements of ester groups and some cooperative movements of naphthalene aggregates. The third relaxation process named α , located near 403K in PEN, constitutes the dielectric manifestation of the glass transition. It is associated with micro-Brownian movements of the chain segments due to configurationally rearrangements of the main chain in the amorphous phase [8, 30, 31].

Because of the complexity of the dielectric relaxation spectrum, we have chosen in the following to focus on the β^* process. In this case, we limit the study of the frequential evolution of the dielectric constant and dielectric loss to the temperature region associated to the β^* relaxation. i.e. $313\text{K} \leq T \leq 373\text{K}$.

The Fig. 4, gives the evolution of the dielectric properties (ϵ' and ϵ'') versus the frequency in the β^* region, registered for NIR-BSPEN films.

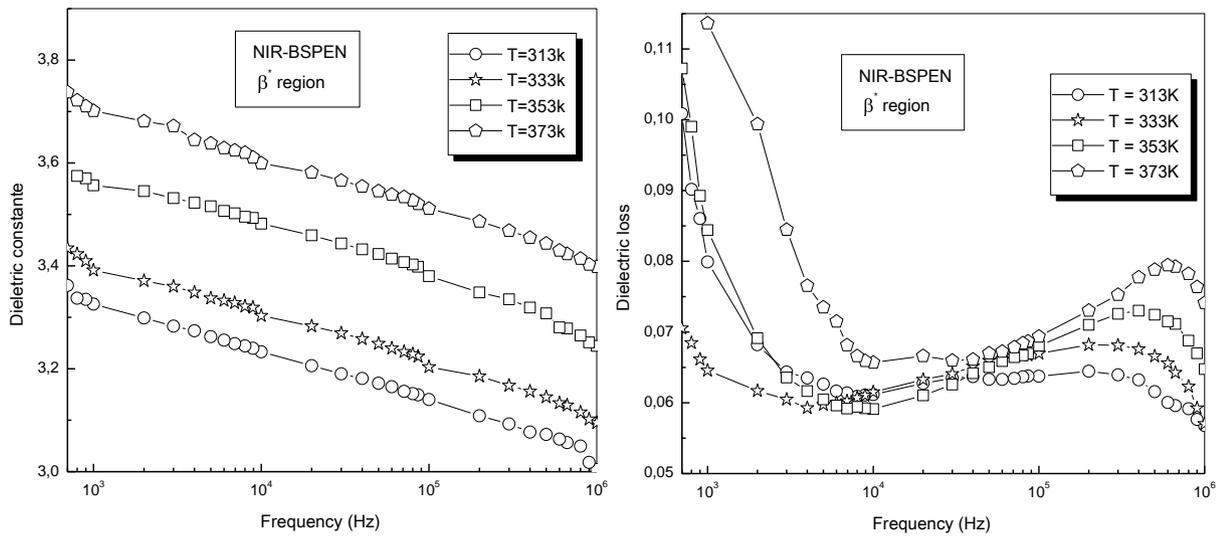


Fig 4. Dielectric constant and dielectric loss versus frequency in the β^* region of NIR-BSPEN film.

It can be seen from Fig. 4, in one hand that, the dielectric constant increases with temperature. In other hand, the magnitude and the frequency of the losses peak increase as the temperature increases.

The plot of the curve representing the variation of the maximum losses as a function of the inverse of the temperature is indicated in Fig. 5.

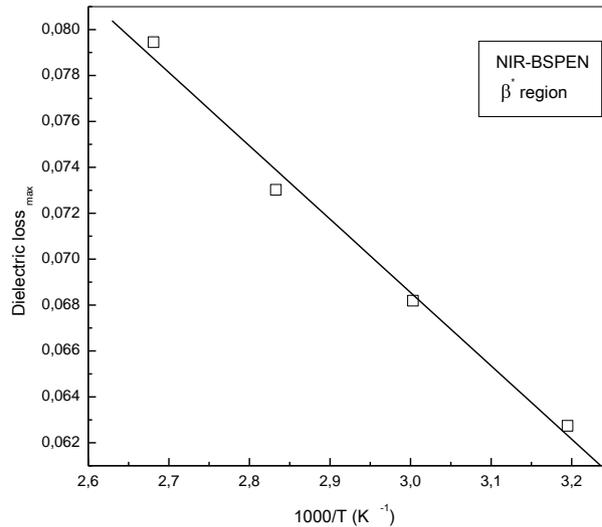


Fig 5. Variation of maximum losses with inverse temperature.

It shows that dielectric losses are thermally activated and the temperature dependence is of the Arrhenius type of the form

$$\epsilon''_{max} = \epsilon''_{\infty} \exp \left[-\frac{E_a}{kT} \right] \quad (1)$$

Where E_a is the activation energy, ε''_∞ is the pre-exponential factor, k is the Boltzmann constant and T is the temperature in Kelvin. The estimated activation energy E_a is equal to 2.87eV. It is worth noting that several studies conducted so far on the characterization of the different properties of polyethylene naphthalate have highlighted dielectric properties as a function of frequency and temperature. Bellomo et al. [32], by studying the effect of temperature on molecular mobility in PEN through the variation of the dissipation factor, have shown that the amplitude of the loss index becomes more important when the temperature is between 303K and 373K. Such a result was observed, at 60Hz, by Yasufuku [32, 33] for a temperature ranging from 233K to 473K. In addition, it can be seen that the peak shifts towards the higher frequency which suggests the reduction of relaxation time [34]. Fig. 7 shows the Arrhenius plot using the maximum frequency as a parameter. The evolution of this experimental curve highlights once again a phenomenon of thermal activation. This defines an average apparent activation energy E_a equal to 0.25eV. Mackersie et al.[35] have studied the low frequency response of PEN in the temperature range 403-423K, the activation energy value was 2.31 eV.

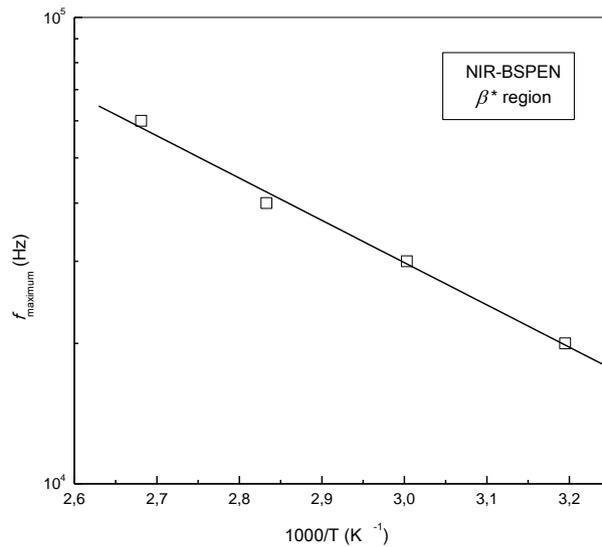


Fig 7. Variation of the frequency of maximum losses with inverse temperature.

The resonance condition given by

$$\omega_{peak} \cdot \tau_a = 1 \quad (2)$$

where τ_a is the average relaxation time and ω is such that

$$\omega_{peak} = 2 \cdot \pi \cdot f_{max} \quad (3)$$

allows to determine the average relaxation time τ_a of the corresponding dipolar relaxation mode, i.e. β^* mode. The Table 2 gives the average relaxation time in the temperature study's range.

Table 2. The values of the average relaxation time

T (K)	313	333	353	373
$\tau_a \times 10^{-6}$ (s)	7.96	5.30	3.98	2.65

The Fig. 8, gives the evolution of the dielectric properties (ϵ' and ϵ'') versus the frequency in the β^* region, registered for IR-BSPEN films.

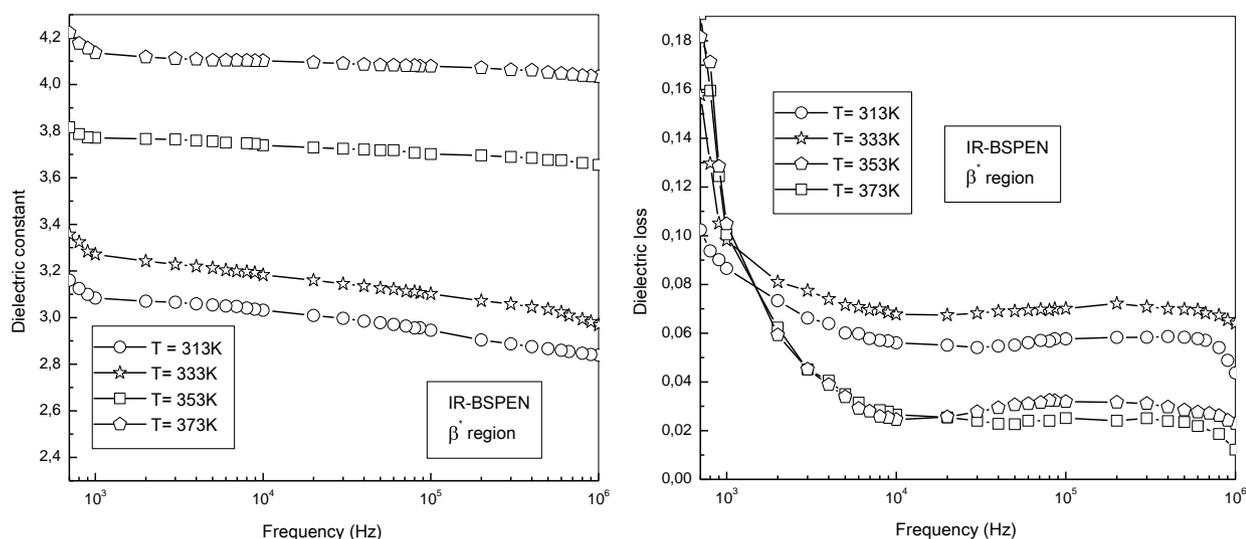


Fig 8. Dielectric constant and dielectric loss versus the frequency in the β^* region of IR-BSPEN film.

The Fig. 8 shows that, the values of the dielectric constant deduced from the dielectric response of IR-BSPEN are higher than those measured through NIR-BSPEN film. For instance at the frequency of 1kHz, the dielectric constant ϵ' , determined for the non irradiated BSPEN film, passes from 3.32 at 313K to 3.70 at 373K while, for the irradiated BSPEN film, the ϵ' constant takes the values of 3.08 and 4.13 at 313 K and 373K, respectively. Regarding the dielectric losses, the values of this parameter at 313K and 333K are important in the case of the IR-BSPEN than those measured through the NIR-BSPEN film but the amplitudes of the dielectric loss peaks created in NIR-BSPEN films, at these temperatures, become more important than one observed in irradiated BSPEN films where we remark the presence of a large peak on all range of the studied frequency. The decrease of the dielectric losses may be attributed to the crosslinking effect of the macromolecular chains, due to the irradiation [37]. This phenomena causes the decrease of the cooperative molecular motion of the naphthalene groups and thus lower the maximum loss frequencies. Furthermore, for $T > 333k$, the Fig.8 shows a modification on the dielectric behavior of IR-BSPEN film. Effectively, in this temperature region, the values of the ϵ'' become lower than ones registered at $T \leq 333k$, instead of increasing like NIR-BSPEN case. The decrease of the dielectric losses, in this case, can be supported by the decrease of the molecular motions for the β^* -relaxation with the heating during which the aggregation of the naphthalene rings decreases and, therefore, less energy is dissipated in the bulk of the IR-BSPEN film. Consequently, it can be deduced that, at $T > 333k$ The IR-BSPEN can be considered like dielectric of capacitors.

4. Conclusion

In this study, the effect of the neutron irradiation on the dielectric properties of bi-stretched polyethylene naphthalate (BSPEN) films was highlighted. In effect, the study undertaken in this work was shown, on one hand, the impact of the neutrons on the dielectric relaxation phenomena, in particular, on the subglass β and β^* relaxations, registered at room temperature and which was translated by an increasing of the values of the constant and the loss dielectric when the frequency increases. This result has been explained by the presence in the irradiated films (IR-BSPEN) of an important quantity of carbonyl and naphthalene ring groups responsible for the appearance of this relaxation type, respectively. This behavior appears when a dielectric films are subject to a sinusoidal alternating voltage. This argument was confirmed by different technique of structural characterization such as the Fourier transform infrared spectroscopy and the polarized light optical microscopy.

In other hand, the dielectric measurement carried out in the β^* region, at different values of the temperature, indicated that the relaxation phenomena are thermally activated, result determined for the NIR-BSPEN sample. The variation of the dielectric parameters versus the frequency, in the IR-BSPEN films, show a decrease of the dielectric loss and an increase of the dielectric constant values for the temperatures above 333K. This result can serve as a reference for further research wishing to use the IR-BSPEN film as a dielectric of capacitors or for a given application in the field of electronics and electrical engineering.

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Ethical Statement

This study does not contain any studies with human or animal subjects performed by any of the authors.

Conflict of Interest

The authors declare that they have no conflict of interest.

Data Availability Statement

Not applicable.

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