



Electrical Properties of Polyethylene Terephthalate under Hydrothermal Aging

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ABSTRACT

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During the working of rotating machines, dielectric materials are submitted to the action of humidity. At long term, their properties can degrade and the lifetime of devices will be reduced. This paper deals with the influence of hydrothermal aging on the electrical properties of polyethylene terephthalate. The insulation was aged in water at 80°C and 100°C. The study shows that dielectric loss factor, permittivity, volume resistivity and dielectric strength were affected by aging. The change is attributed to the fact that the aging reduces molecular bonds causing a decrease in the viscosity. Thereby, the free volume and the mean free path raise. This process leads to the raise in the mobility of charge carriers. The evolution of activation energy versus aging time exhibits minimums and maximums resulting to the plasticization and the crosslinking of the material, respectively. The TGA thermograms indicate a variation of onset temperature with aging time. It was highlighted by TGA that the decomposition occurs following one step. The FTIR analysis points out a change in the intensities of the vibrational spectra after aging. The penetration of water within the dielectric induces differential inflations. The physical chemical analysis shows a modification in the molecular conformation of the polymer. The results are analysed and discussed.

1. INTRODUCTION

It is well known that electric cables insulation contains microvoids [1] which, with time, are filled with water. When the microvoids are full of water, the insulation water content tends to saturate. The higher the temperature the more water diffuses within the dielectric material. Pelissou and Wintle [2] indicated that the water content of cable XLPE insulation, after immersion at 80°C, reached 4000 ppm. The diffusion and the absorption of water can lead to electrical failure of cables by water treeing as reported by Moreau et al. [3] and Dissado and Fothergill [4]. Several researchers reported the degradation of polymers under hydrothermal aging [5-10].

In a previous work [5], we pointed that the breakdown voltage of polyesterimide decreases with hydrothermal aging time and depends on the forms of applied electric field which are AC and DC stresses. A change in color and a crumbling of the specimens were observed.

In a recent paper [6] on hydrothermal aging of polyvinyl chloride used in medium voltage cables, we reported that dielectric loss factor, dielectric constant, volume resistivity and dielectric strength vary with aging time. A modification in elongation at break and tensile strength was highlighted. A change in the color of the samples was shown.

Djijelli et al. [7] examined the influence of hydrothermal aging on the properties of plasticized poly (vinyl chloride). The authors indicate a weight variation versus immersion time in water at 100°C. They found a reduction in elongation at rupture and tensile strength with aging time.

Roggendorf et al. [8] investigated the effect of hydrothermal aging on the electrical properties of fiber-glass reinforced with polyamide. The study shows a decrease in specific resistivity,

and a raise in dielectric constant and dielectric loss factor. The authors reported that the breakdown voltage shortens at the first stage of aging and remains practically constant for the prolonged time.

Li et al. [9] evaluated the hydrothermal degradation of epoxy at 75°C and 100% relative humidity. The study yields an increase in mass loss of the samples versus aging time. This raise reached 0.5% after 5000 h. The dielectric loss factor and the permittivity increase with aging time. While the dielectric strength and the volume resistivity decay versus aging time. The authors reported that the glass transition temperature (T_g) of the material decreases during the first period of aging and raises with more aging.

Du et al. [10] studied the hydrothermal aging of poly (ethylene terephthalate) (PET). The authors found that the presence of ester groups in the backbone of PET facilitates the attack of the material by water and causes hydrolytic decomposition. These researchers reported that hydrolytic degradation involves the transformation of ester groups to corresponding carboxyl acids and alcohols, with chain scissions and a decrease of the molecular weight.

PET is considered to be one of the most important engineering thermoplastics available [11]. This polymer has excellent physical properties [12]. Its glass transition temperature is situated around 80°C and the melting temperature is 258°C [13].

The purpose of this work is to study the effects of hydrothermal aging on the electrical properties of polyethylene terephthalate used in rotating electrical machines. The physico - chemical characterization was carried out by thermogravimetric analysis (TGA) and Fournier Transform Infrared Spectroscopy (FTIR).

2. EXPERIMENTAL TECHNIQUES

2.1 Samples

The studied polymer (PET) is formed by a film placed between two sheets. The whole is heavily gathered. The material is provided in roller shape of 0.2 mm thickness. For the tests, square samples of 7.5 cm in side were achieved.

2.2 Hydrothermal aging

Firstly, the specimens were put in a vat containing water. Then the whole was introduced in ventilated and regulated ovens. The hydrothermal aging was performed at two temperatures: 80°C and 100°C. The values of aging temperatures were chosen to be greater than the glass transition temperature T_g of PET which is about 80°C as quoted before. The aging time reached 6000 h at 80°C and 5500 h at 100°C. A sampling of samples was taken from the ovens every 500 h. The water was changed every day.

2.3 Measurements of electrical properties

The capacitance and the dielectric loss factor were evaluated with a Schering bridge (type TETEX 2830/2831 type). The sample was placed between two plane electrodes of stainless steel having 20 cm² in surface. The apparatus is outfitted with two heating plates which allow raising the temperature of the specimen up to 250°C. The device is equipped by a temperature regulator and connected to a computer. AC voltage of 2 kV, 50 Hz was applied to the sample. The insulation resistivity was measured with a picoammeter under DC voltage of 2 kV. The relative permittivity and the volume resistivity were derived.

The breakdown tests were achieved with a dielectric test apparatus (BAUR OLPRUFGERAT PGO 90 A), 90 kV, 50 Hz. The test cell contains two plane electrodes of brass. In order to avoid partial discharges, the cell was filled with insulating oil ("Borak 22"). The sample was put between the electrodes, and then AC voltage, 50 Hz, was applied to the insulation with a speed of 2 kV/s. The breakdown voltage was recorded. The dielectric strength was calculated as the quotient of the breakdown voltage and the thickness at the perforation point.

2.4 Physical chemical characterisation

The polymer was characterized before aging, and after 3000 h and 6000 h at 80°C; and after 3000 h and 5500 h at 100°C.

The thermogravimetric analysis was performed with SDT Q600 V20.9 Build -20 instrument at temperatures ranging from 30 to 800°C under a nitrogen atmosphere. The heating rate is 20°C/min. The average mass of the samples is about 5 mg.

The polymer was also analysed by Fournier Transform Infrared spectroscopy. Infrared spectra were executed on MicroLab Expert FTIR Software from 4000 to 650 cm⁻¹.

3. RESULTS AND DISCUSSION

3.1 Variation of electrical properties versus aging time

The dielectric loss factor, the permittivity and the volume

resistivity were measured at 80°C and 100°C. While the dielectric strength was evaluated at room temperature.

3.1.1 Dielectric loss factor

Figure 1 presents the dependence of the dielectric loss factor ($\text{tg}\delta$) on aging time. The evolution can be summarized as follows:

- At 80°C, $\text{tg}\delta$ increases from 0.166 to 0.183 and decreases to 0.147, then raises until 0.162 after 2000 h. Afterwards, $\text{tg}\delta$ lowers slowly to 0.147 then grows rapidly and reaches a peak of 0.214. Next it drops to 0.149 for 4500 h and remains somewhat constant until 5000 h. Then $\text{tg}\delta$ increases and attains a peak of 0.189 corresponding to 5500 h. Latter $\text{tg}\delta$ lessens to 0.172 for 6000 h. The maximum variation is 28.92%.
- At 100°C, $\text{tg}\delta$ decreases from 0.162 to 0.139 after 1500 h. Then $\text{tg}\delta$ raises quickly and reaches 2 peaks of 0.23 and 0.251 corresponding to 3000 h and 4000 h, respectively. Beyond 4000 h, $\text{tg}\delta$ falls to 0.182 for 5500 h. The maximum variation is 54.94%.

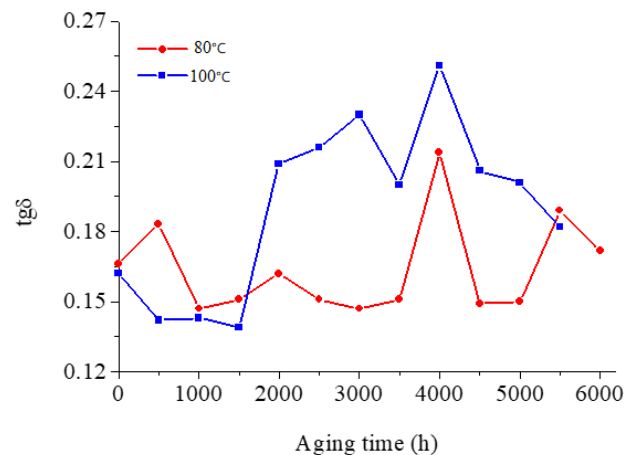


Figure 1. Variation of dielectric loss factor in function of aging time at 80 and 100°C

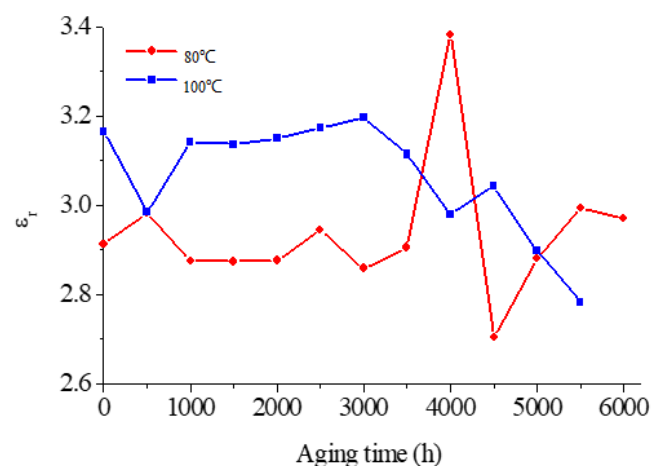


Figure 2. Variation of permittivity in function of aging time at 80 and 100°C

3.1.2 Permittivity

The evolution of the permittivity (ϵ_r) in function of aging time is exposed in Figure 2. The variation can be described as

follows:

- At 80°C, ϵ_r increases from 2.91 to 2.99, shortens slightly to 2.88 and stabilizes around this value until 3500 h. Afterwards, it raises and attains a net peak of 3.38 after 4000 h. Beyond this peak, ϵ_r falls to 2.70 and raises to 2.99 and stays somewhat invariable until 6000 h. The maximum variation is 16.15%.
- At 100°C, ϵ_r decreases from 3.17 to 2.99 and increases to 3.14 and becomes practically constant until 3000 h. Then, ϵ_r drops to 2.78 after 5500 h. The maximum variation is 12.30%.

3.1.3 Dielectric strength

Figure 3 exhibits the evolution of the dielectric strength (E_b) versus aging time. The evolution can be described as follows:

- At 80°C, at the beginning of aging, E_b is practically invariable; its value locates around 52.5 kV/mm. Latter it increases until 55.55 kV/mm and falls to 47.85 kV/mm after 2500 h. Then E_b rises to 51.19 kV/mm and decreases to 48.67 kV/mm. It remains constant until 6000 h. Its average value is 49.76 kV/mm. The maximum variation is 9.56%.
- At 100°C, at first, E_b is practically constant; its value is about 52.25 kV/mm. Then it raises and reaches a net peak of 62.57 kV/mm. Latter, the dielectric strength decays to 48.35 kV/mm and stays invariable until 2500 h. Next, E_b decreases to 45.17 kV/mm, rises to 47.06 kV/mm and stabilizes around this value until 5000 h. Afterwards, E_b increases to 49.37 kV/mm after 5500 h. The maximum variation is 18.34%.

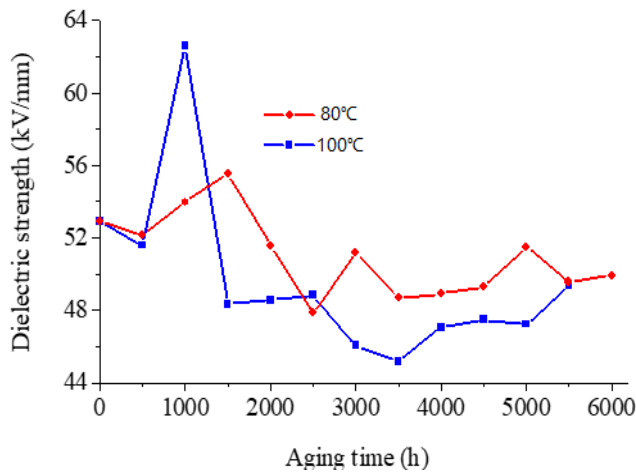


Figure 3. Variation of dielectric strength in function of aging time at 80 and 100°C

3.1.4 Volume resistivity

Figure 4 illustrates the variation of the volume resistivity (ρ) against aging time. The evolution can be summarized as follows:

- At 80°C, ρ decreases from $7.4 \times 10^{14} \Omega \cdot \text{cm}$ to $4.2 \times 10^{14} \Omega \cdot \text{cm}$ and increases until $8.8 \times 10^{14} \Omega \cdot \text{cm}$ for 1500 h. Afterwards, ρ lowers until $3.3 \times 10^{14} \Omega \cdot \text{cm}$ for 2500 h. Next, it rises to $7 \times 10^{14} \Omega \cdot \text{cm}$ and shortens to $4 \times 10^{14} \Omega \cdot \text{cm}$ for 4000 h. After this time, ρ grows to $7.8 \times 10^{14} \Omega \cdot \text{cm}$ then lessens to $5.6 \times 10^{14} \Omega \cdot \text{cm}$ and increases a little to $6.8 \times 10^{14} \Omega \cdot \text{cm}$ after 6000 h. The maximum variation is 55.41%.
- At 100°C, at first, ρ is practically invariable. Its value

locates around $1 \times 10^{14} \Omega \cdot \text{cm}$. Then it decreases to $5.8 \times 10^{13} \Omega \cdot \text{cm}$ after 1530 h. Next, ρ raises to $1.2 \times 10^{14} \Omega \cdot \text{cm}$ for 3500 h. Afterwards, it drops until $1.0 \times 10^{13} \Omega \cdot \text{cm}$ for 5500 h. The maximum variation is 89.69%.

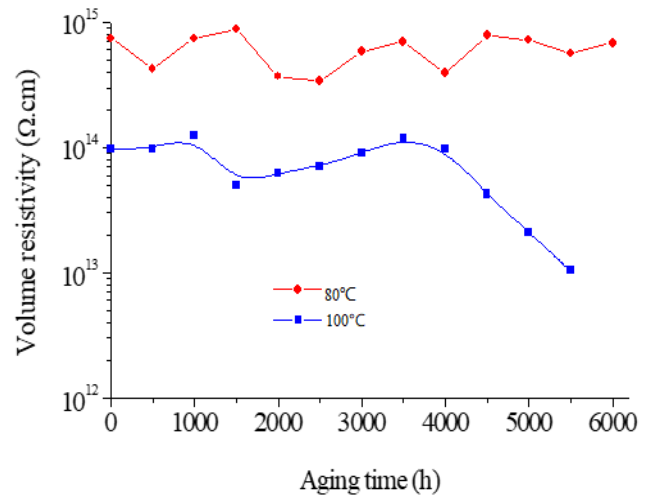


Figure 4. Variation of volume resistivity in function of aging time at 80 and 100°C

3.2 Variation of electrical properties versus temperature before and after aging

The variations of $\text{tg}\delta$, ϵ_r and ρ versus temperature were investigated before and after aging. The temperature was varied from 30°C to 150°C.

3.2.1 Dielectric loss factor

Figure 5 presents the evolution of $\text{tg}\delta$ with respect to temperature before and after aging. The variation can be summarized as follows:

- Before aging and between 30 and 50°C, $\text{tg}\delta$ is practically invariable. Its average value is about 0.146. Then, $\text{tg}\delta$ grows to 0.166 for 80°C and stays constant. Next, it rises until 0.213 at 150°C.
- After 6000 h at 80°C, $\text{tg}\delta$ increases rapidly from 0.11 and attains a peak of 0.18 for 60°C and lowers to 0.137 corresponding to 150°C.

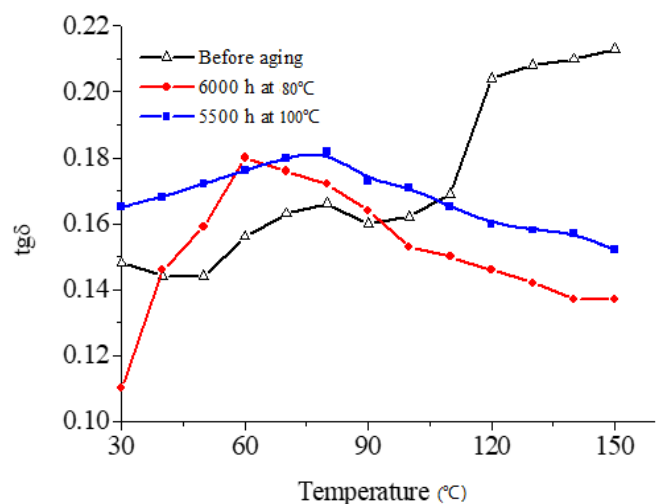


Figure 5. Variation of dielectric loss factor with temperature before and after aging

- After 5500 h at 100°C, tgδ increases slowly from 0.165 for 30°C to 0.181 at 77°C. Latter, tgδ lessens to 0.152 corresponding to 150°C.

3.2.2 Permittivity

Figure 6 reports the variation of the permittivity against the temperature before and after 6000 h at 80°C and 5500 h at 100°C. The variation can be described as follows:

- Before aging, ϵ_r increases slightly from 2.73 at 30°C up to 3.52 corresponding to 150°C.
- After 6000 h at 80°C, ϵ_r decreases from 3.87 for 30°C to 2.94 at 74.5°C and stays somewhat constant. Its value stabilizes around 2.98 until 90°C. Next, it grows slowly up to 3.61 corresponding to 150°C.
- After 5500 h at 100°C, ϵ_r raises slowly from 2.49 for 30°C until 3.35 at 150°C.
- We notice that ϵ_r at 100°C is smaller than that corresponding to before aging.

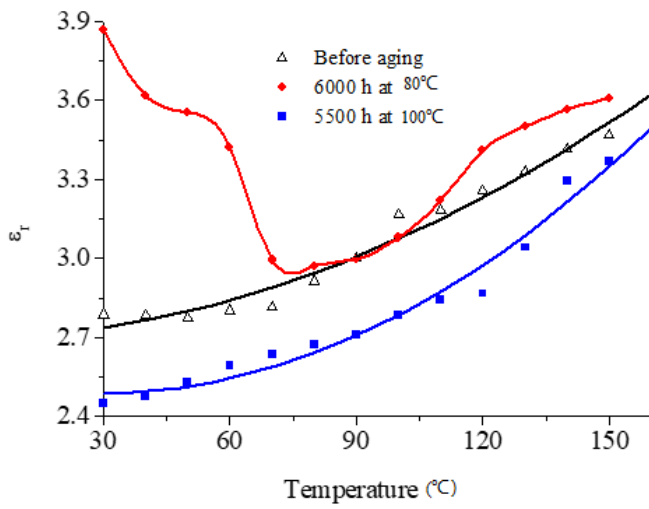


Figure 6. Variation of permittivity with temperature before and after aging

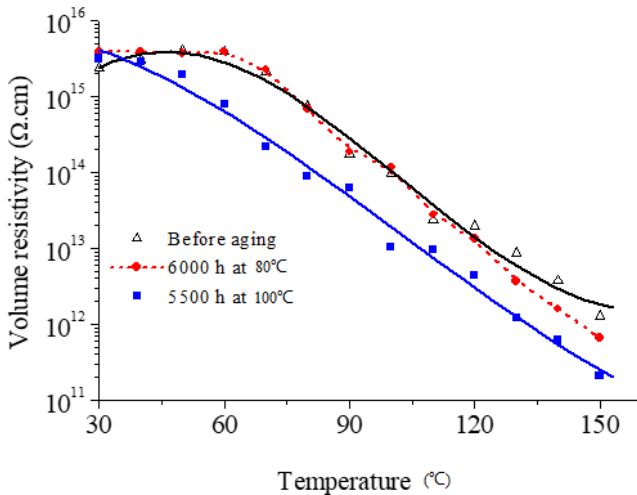


Figure 7. Variation of volume resistivity with temperature before and after aging

3.2.3 Volume resistivity

Figure 7 shows the evolution of the volume resistivity as a function of temperature. The variation can be summarized as follows:

- Before aging and between 30 and 54°C, ρ increases slowly from $2.35 \times 10^{15} \Omega \cdot \text{cm}$ to $3.88 \times 10^{15} \Omega \cdot \text{cm}$. Then it lowers rapidly to $1.8 \times 10^{12} \Omega \cdot \text{cm}$ for 150°C.
- After 6000 h at 80°C, in the range of 30 to 60°C, ρ is practically invariable. Its value is situated around $3.8 \times 10^{15} \Omega \cdot \text{cm}$. Beyond 60°C, ρ decreases rapidly until $6.51 \times 10^{11} \Omega \cdot \text{cm}$ for 150°C.
- After 5500 h at 100°C, ρ falls from $4.16 \times 10^{15} \Omega \cdot \text{cm}$, for 30°C, to $2.44 \times 10^{11} \Omega \cdot \text{cm}$ at 150°C.
- The curves before and after 6000 h at 80°C are practically confused.

3.3 Determination of activation energy

In order to determine the activation energy of the degradation process, the volume resistivity is plotted with respect to reciprocal absolute temperature ($1/T$) before and after the different aging times. For convenience, only one example of the curve is presented, as it can be seen in Figure 8. The characteristic is a straight line and obeys to the Arrhenius' law. The volume resistivity ρ of polymers is expressed by the following formula [14]:

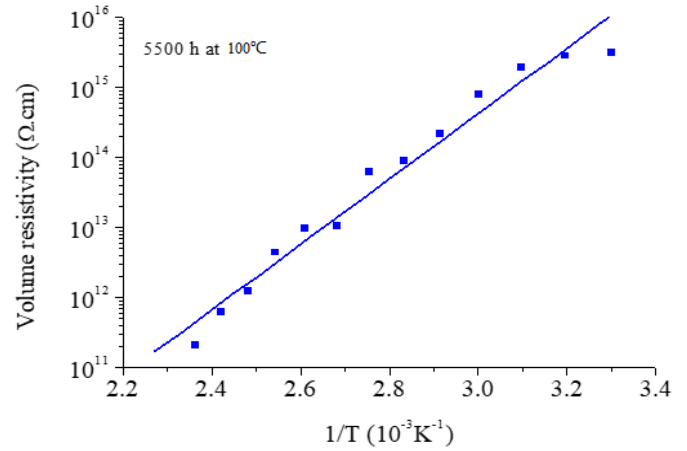


Figure 8. Variation of the volume resistivity in function of reciprocal absolute temperature after 5500 h at 100°C

$$\rho = \rho_{\infty} \exp\left(\frac{-E}{RT}\right) \quad (1)$$

with:

ρ_{∞} ($\Omega \cdot \text{cm}$): limit of the resistivity at the infinite temperature;

E (J mol^{-1}): process activation energy;

T (K): absolute temperature;

R : gas constant ($R = 8.314 \text{ J mol}^{-1} \text{ K}^{-1}$).

The activation energy was deduced. The dependence of E on aging time at 80°C and 100°C is shown in Figure 9. The variation can be described as follows:

- At 80°C, E decreases from 17.84 kcal/mol to 16.60 kcal/mol and increases to 18.14 kcal/mol after 1000 h. Then it shortens to 16.69 kcal/mol and grows to 19.89 kcal/mol after 3000 h. Next, it decays to 10.67 kcal/mol and raises until 18.63 kcal/mol corresponding to 5000 h. Afterwards, E decreases to 15.25 kcal/mol and increases to 20.09 kcal/mol after 6000 h. The maximum variation is 40.19%.
- At 100°C, at the first 1500 h of aging, E remains constant. Its value stabilizes around 17.91 kcal/mol. Then E

decreases to 14.12 kcal/mol and increases to 19.86 kcal/mol after 3000 h. Afterwards, the activation energy decreases slowly to 17.74 kcal/mol and raises to 21.48 kcal/mol for 5500 h. The maximum variation is 20.40%.

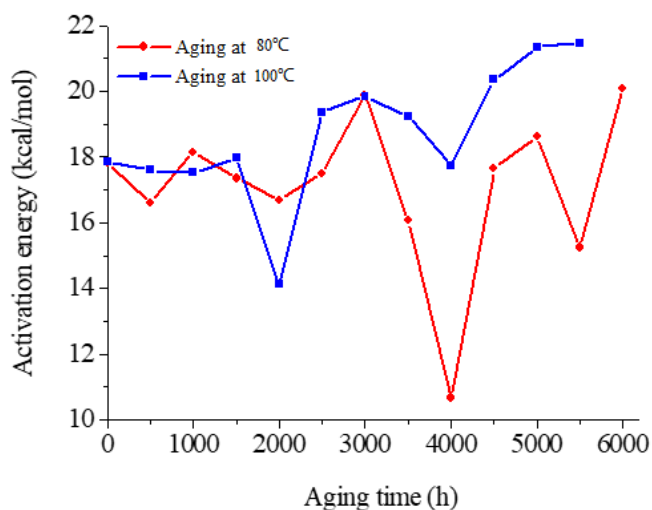


Figure 9. Changes of activation energy as a function of aging time at 80°C and 100°C

3.4. Physical-chemical analysis

3.4.1 Thermogravimetric analysis

The variation of the weight (%) of the polymer with respect to temperature is examined on aged and unaged samples. The TGA thermograms, represented in Figures 10a-10c, have the same form. A small weight loss can be noticed in the temperature range: 100°C - 250°C. This weight loss is attributed to the evaporation of water and solvents.

- Before aging, the weight loss begins at around 402°C, quickens and attains 93% at 456°C, and then it decelerates. The obtained residue is 5.5% corresponding to 796°C. The temperature of 50% mass loss is 430°C.
- After 3000 h at 80°C, the weight loss starts at about 400°C. Beyond the onset temperature, it speeds up and reaches 81.86% at 453°C. Afterwards, the weight loss slows down and it remains a residue of 8.63% at 795°C. The temperature of 50% mass loss is 434°C.

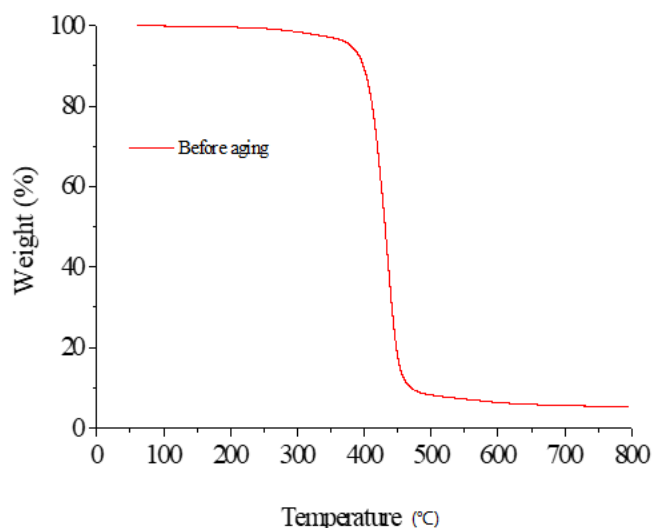


Figure 10a. Thermogravimetric analysis curve before aging

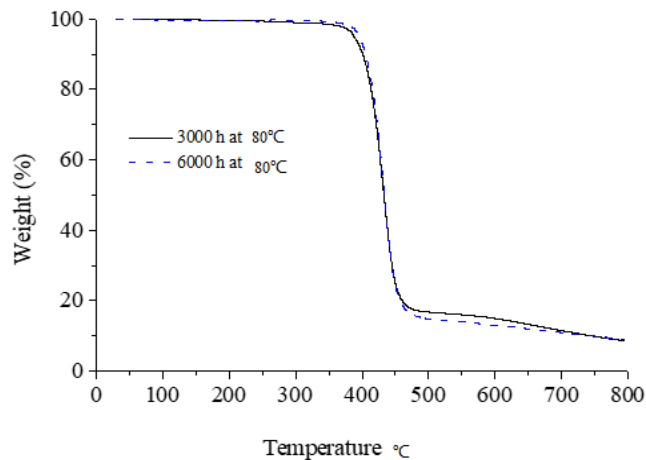


Figure 10b. Thermogravimetric analysis curves after 3000 h and 6000 h at 80°C

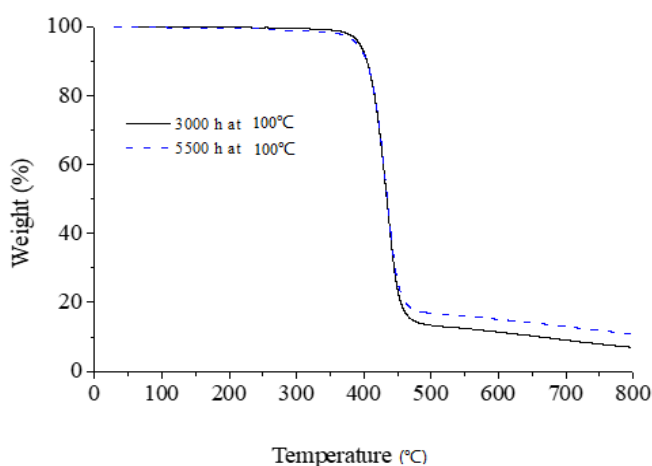


Figure 10c. Thermogravimetric analysis curves after 3000 h and 5500 h at 100°C

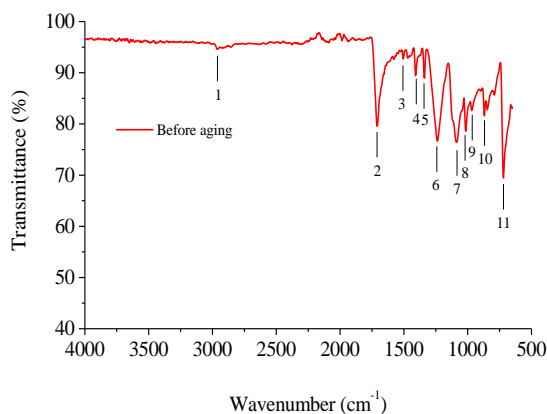
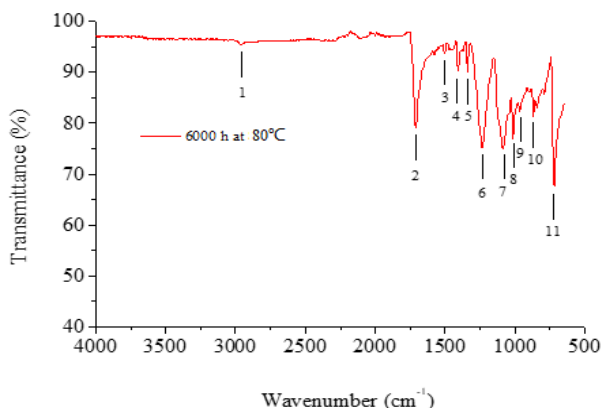
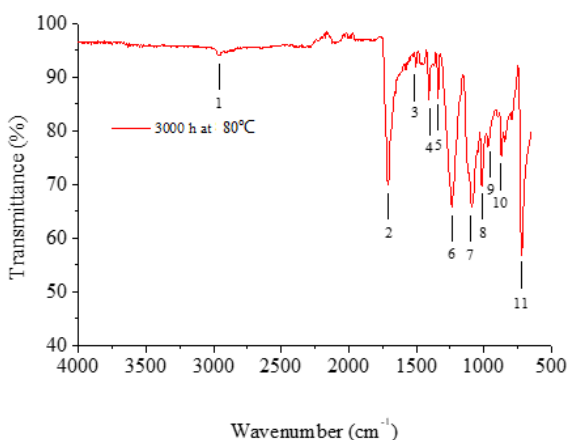
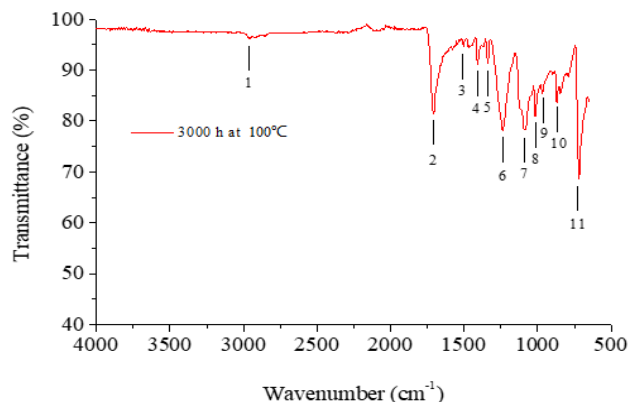
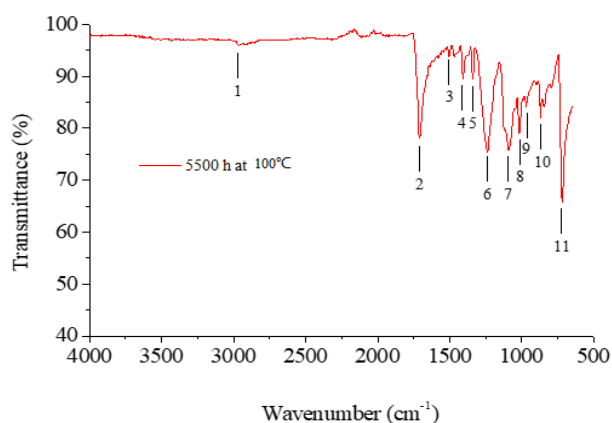
- After 6000 h at 80°C, the weight loss begins at around 406°C, accelerates until 84.82% for a temperature about 452°C. After the weight loss slackens and it stays a residue of 9.16% at 795°C. The temperature of 50% mass loss is 433°C.
- After 3000 h at 100°C, the weight loss starts at around 407°C and hastens until 85.48% for a temperature about 450°C. Then it slows down leading to a residue of 7.05% at 795°C. The temperature of 50% mass loss is 433°C.
- After 5500 h at 100°C, the weight loss begins at about 409°C. Latter, it expedites and attains 82.09% at 451°C. Beyond this point, the weight loss goes slow and it rests a residue of 11.13% at 795°C. The temperature of 50% mass loss is 435°C.
- The decomposition is governed by a first-order chemical reaction that is one stage.

3.4.2 FTIR analysis

Figures 11a-11e present FTIR spectra before and after 3000 h and 6000 h at 80°C; and after 3000 h and 5500 h at 100°C. The IR spectra of aged and unaged specimens were compared. The significant absorption bands are exposed in Table 1. These IR bands are attributed to the vibrations reported by Chércoles Asensio et al. [15].

Table 1. Absorption bands

Absorption band number	Wavenumber (cm ⁻¹)
1	2959
2	1710
3	1508
4	1408
5	1337
6	1238
7	1087
8	1013
9	964
10	870
11	718

**Figure 11a.** FTIR spectrum before aging**Figure 11b.** FTIR spectrum after 3000 h at 80°C**Figure 11c.** FTIR spectrum after 6000 h at 80°C**Figure 11d.** FTIR spectrum after 3000 h at 100°C**Figure 11e.** FTIR spectrum after 5500 h at 100°C

1. It appears absorption band with narrow intensity at 2,959 cm⁻¹. It is assigned to asymmetric and symmetric stretching vibration of -CH₂- (backbone polymer).
2. The medium absorption band, observed at 1710 cm⁻¹, is attributed to stretching vibration of C=O, characteristic of ester.
3. The observed absorption band at 1,508 cm⁻¹, with a weak intensity, is ascribed to the stretching vibration of C=C.
4. The small absorption bands at 1,408 cm⁻¹ and 1,337 cm⁻¹ correspond to symmetric and asymmetric bending vibration in-plane C-H and rocking bending of C-H -CH₂-.
5. The observed absorption band at 1,238 cm⁻¹, with medium intensity, is allotted to the stretching vibration of C-C (O)-O.
6. The medium absorption band, arising at 1,087 cm⁻¹, is due to the stretching vibration of -O-C-.
7. The weak absorption bands at 1,013 cm⁻¹ and 964 cm⁻¹ are allocated to the bending vibration in-plane of =C-H.
8. The small absorption band, observed at 870 cm⁻¹, is ascribed to the bending vibration out-of-plane of =C-H.
9. The medium absorption band, appearing at 718 cm⁻¹, corresponds to the wagging bending vibration of =C-H.

After aging, we note that there is no disappearance of the absorption bands. Also, new absorption bands do not arise. After 3000 h at 80°C, the intensities of peaks increase. The rise is more significant for the peaks at 1710 cm⁻¹, 1238 cm⁻¹, 1087 cm⁻¹ and 718 cm⁻¹. After 6000 h at 80°C, a small change of the spectra intensities was noticed. After aging at 100°C, all the peaks appear with a short change in the intensities.

3.5 Discussion

1. The raise of $\text{tg}\delta$ with time can be explained by the fact that hydrothermal aging weakens the molecular bonds leading to a reduction in the viscosity and an increase of the free volume. Thence the mobility of the charge carriers and the conductivity rise. Whereas the decrease of $\text{tg}\delta$ is assigned to the arrangement of the molecular structure which causes the opposite phenomenon. The same interpretation can explain the variation of ρ and ϵ_r versus aging time. The minima and the peaks of $\text{tg}\delta$ and ϵ_r versus aging time are allotted to the relaxation phenomena due the macromolecular movements of the polymer chains. Peaks have been highlighted in a previous work [16]. The diminution of ρ is due the increase in the electrical conductivity. While its increase may be due to the arrangement in the molecular structure as described before. It is obvious that the volume resistivity of the samples aged at 80°C is greater than that corresponding to 100°C.
2. The absorbed water makes the samples more polar as reported by Van Krevelen [17]. This phenomenon explains also the raise of $\text{tg}\delta$ and ϵ_r after aging.
3. The curve giving the dielectric strength against aging time shows two peaks attributed to the crosslinking of the insulating material. However, the minima are allotted to the plasticization caused by the absorbed water within the insulation. The crosslinking shortens the mean free path leading to an increase in dielectric strength. The plasticization induces the opposite phenomenon than the crosslinking. The variation of the dielectric strength is also allotted to the effect of space charge. The presence of the space charge has a great impact on dielectric breakdown of the material. This phenomenon has been reported by Zhou and Chen [18] and Xing et al. [19].
4. The increase of the activation energy confirms the crosslinking of the polymer. While its decrease affirms its plasticization. The raise of the activation energy after aging has been reported elsewhere [6].
5. A swelling of the samples was observed resulting to the penetration of water during the aging. The swelling will induce pressure and strain. Thence the mechanical properties of the polymer will be affected.
6. The thermogravimetric analysis allows concluding that onset temperature increases after aging at 80 and 100°C.
7. The modification in the magnitudes of the absorption peaks observed in IR spectra was associated to the variation in the dipolar moment of molecules. The results of FTIR enable to conclude that there is no degradation but a modification in molecular conformation of the material.

4. CONCLUSION

This work exhibits that the electrical properties of polyethylene terephthalate are influenced by hydrothermal aging. Thus, the dielectric loss factor, the permittivity, the volume resistivity and the dielectric strength change during the aging. The activation energy as a function of aging time shows a great variation resulting to the plasticization and the crosslinking of the polymer. A swelling of the specimens was observed. The results of TGA indicate a changing in onset temperature after aging. The results of FTIR exhibit variations in intensities of the absorption bands. The physical chemical

analysis points a change in the polymer conformation. The obtained results are in agreement with those reported in the literature [6, 16].

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