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Investigation of electrical ageing effects on the structural and electrical properties of polystyrene by charging and discharging current method

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Keywords: electric conduction, discharging current, anomalous current, polystyrene, electrical ageing

Abstract

This study focuses on isothermal charging and discharging currents in polystyrene (PS). It is a well known fact that the discharging current is almost the mirror symmetry of the charging current at low electric fields. However, the experimental results presented and discussed in this study have revealed the possibility of an abnormal discharging current (ADC) flowing in the same direction as the charging current. Furthermore, electrical measurements implied that the electrical ageing of PS resulted in a significant increase in the charging current, even for extended ageing periods. Moreover, the intensity and duration of the reverse discharging current have evolved with electrical ageing. This characteristic can be attributed to the presence of ionic impurities both trapped at the polymer surface and in the bulk structure. To highlight the structural changes induced by electrical ageing, detailed structural analyses were conducted using Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM) and energy-dispersive x-ray (EDX) analysis.

1. Introduction

In electrical engineering, the choice of insulating materials is of principal importance to ensure the reliability, safety, and durability of systems [1]. However, these materials, exposed to electrical, thermal, and radiation stresses, undergo ageing, which affects the lifespan of electrical components.

Insulating polymer materials are commonly used in high-voltage systems due to their outstanding physical properties and resistance to electrical stresses [2, 3]. Due to its exceptional dielectric properties, polystyrene (PS) is widely used in electrical applications, particularly in high-voltage direct current (HVDC) capacitors. Its dielectric properties, as well as its optical and electromagnetic performances, make it a promising candidate for insulation applications [4]. Naturally, PS, like all polymers, undergoes service-related stresses that impact its dielectric performance [5]. In addition to its electrical characteristics, it is essential to consider its high glass transition temperature (T_g) (~100 °C) and its thermal and mechanical properties when designing a PS component for engineering applications. PS homopolymer, a member of engineering plastics, is an amorphous (transparent), rigid, and brittle material with a high stiffness and very low impact resistance. Therefore, high impact polystyrene (HIPS) grades are generally used in flexible material. Another most commonly used form of PS is the foam structure known as expanded polystyrene (EPS) and characterized by extremely low density and thermal conductivity values. PS foams are used in thermal insulation and packaging applications. Moreover, PS block copolymers are the major group of thermoplastic elastomers (TPE) which are one of the promising and emerging materials in electrical insulating applications as highly flexible and easy processible halogen free flame

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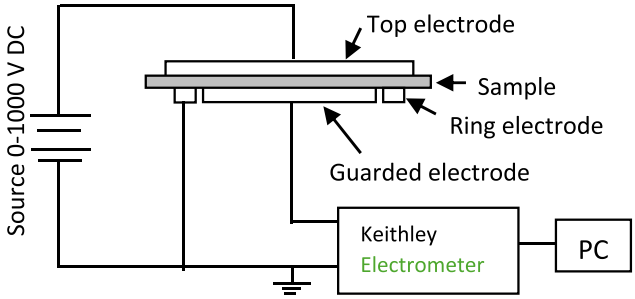
(a)



(b)

Figure 1. (a) Photo of the partial discharge. (b) test rig for the charging and discharging currents measurements.

retardant (HFFR) compounds. The most extensively used styrene-based TPE compounds are formulated with polystyrene-b-poly(ethylene-ran-butylene)-b-polystyrene (SEBS) as base material.

It has been shown that the service conditions are the primarily factors for major faults and related structural degradation in PS insulators. These effects may mainly alter the dielectric properties of PS parts as well as their physical and mechanical characteristics. Ageing of PS have been studied in last few decades to understand the relationships between environmental conditions and physical and thermomechanical properties of PS materials. De Castro Monsores *et al* [6] showed that PS underwent structural modification affecting its mechanical performances when it is exposed to ultraviolet (UV) radiation in an oxidizing atmosphere. Tanaka *et al* [7] also demonstrated that the physical ageing changed the thermal properties of PS. Grigoriadi *et al* [8] showed that thermal ageing influenced the dielectric and chemical characteristics of PS. As the electrical ageing of PS insulators are less studied subject than their thermal and mechanical ageing so far, the underlying mechanisms are still unclear. A few studies in the relevant literature were mainly interested in the effect of electrical ageing on the structural [9], dielectric [3] and morphological [10] characteristics of PS. Therefore, understanding changes in structural and physical properties of styrene-based polymers and copolymers as a function of electrical ageing conditions, will provide very useful information for predicting long-term performances of PS-based insulating materials.

In the literature, it is well established that the charging and discharging currents can be regarded as one of the descriptive tools for assessing the electrical ageing of polymers. It has been demonstrated that the discharging current changed direction and exhibited an anomalous peak in some situations. This latter was already observed at high electric fields [11–14]. Such behaviour has not been studied yet under low dc field strength.

This study quantifies electrical ageing of PS under low dc field strength. The charging and discharging currents of circular specimens were measured. In order to highlight the physico-chemical modifications in the polymer structure, complementary analysis have also been monitored using some instrumental techniques such as Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and Energy-Dispersive x-ray (EDX) analysis.

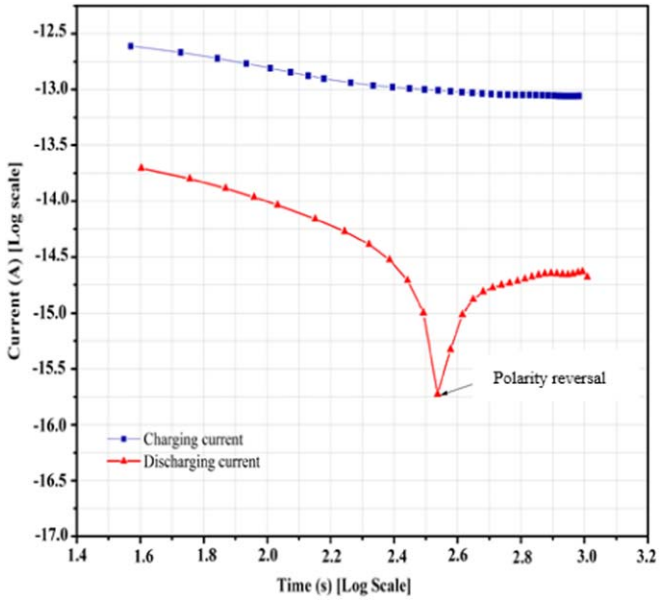


Figure 2. Time dependence of charging and discharging currents of PS under 1000 V DC-voltage.

2. Experimental

The electrical tests were performed with circular PS specimens with a thickness of 3 mm and diameter of 85 mm.

The electrical ageing was carried out with a simple Metal-Insulating-Metal (MIM) experimental arrangement. The specimen was sandwiched between two circular electrodes with a diameter of 5 mm. A 10 kV alternative voltage was applied for several periods varying from 1h to 8 h. For each ageing period, five different specimens were tested. The partial discharge is illustrated in figure 1(a). The ageing was performed under a temperature of 20 ± 2 °C and a humidity of $60 \pm 5\%$.

The charging and discharging currents are measured using a Keithley electrometer 6517 and test chamber 8009 coupled to a computer (PC). The electrode system contains a guarded electrode of 50.8 mm diameter and a guard (ring) electrode of 57.2 mm diameter. The charging current was measured by applying 1000 V DC-voltage for 1000 s. The discharging current was measured for the same period of time after removing the applied voltage and short-circuiting the sample through the electrometer. The current measurements were performed at ambient conditions. A photo of the partial discharge and the test rig used for currents measurements are respectively shown in figure 1.

Fourier Transformed Infrared Spectroscopy (FT-IR), Scanning Electron Micrographs (SEM) and Energy-Dispersive x-ray analysis (EDX) analysis were after conducted to quantify possible effects of electrical ageing on the polymer structure. In these characterizations, a Shimadzu 8400 FT-IR spectrometer and a Philips XL 30 SEM operated at 5–20 kV were used.

In this study, it was assumed that ageing has occurred in the discoloured region associated with PD activity. Thus, the different measurement and tests were done in the region. This does not also exclude that ageing have also took place in the region located inside the discoloured region.

3. Results and discussion

3.1. Polarization (charging) current at low field

Figure 2 shows the typical behavior of charging and discharging currents of PS under a 1000 V DC voltage. Shortly after applying DC voltage, the charging current initially decreases slowly before reaching a steady state at about 800 s. The time-dependent behavior of transient current is described by the Curie-von Schweidler law, expressed here in amperes as $I(t) = 1.4 \times 10^{-12} \times t^{-0.5}$ for time periods less than 200 s. However, it is important to note that this law is not applicable for longer durations [15]. The decrease in charging current over extended time periods can be attributed to the gradual occupation of traps by charge carriers [16].

3.2. Discharging current

After removing the external field and short-circuiting the sample through the electrometer, the discharging current (also known as desorption current) was measured. Typically, this discharging current (DC) flows in the

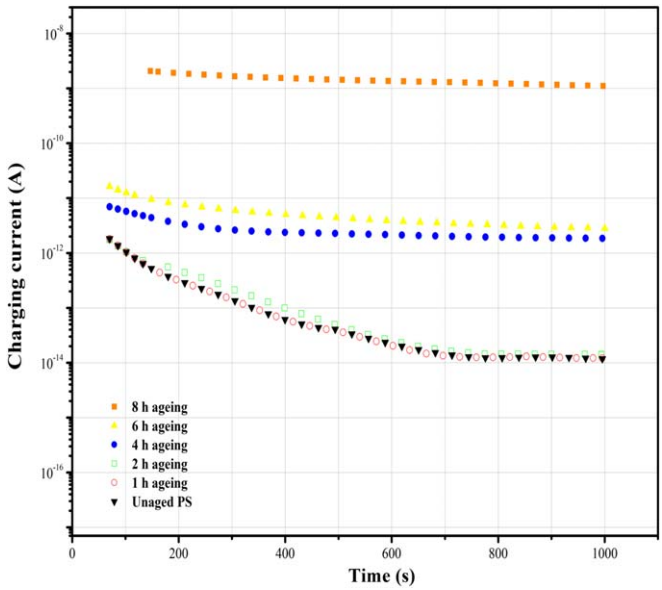


Figure 3. Effects of electrical ageing on the charging current as a function of ageing time.

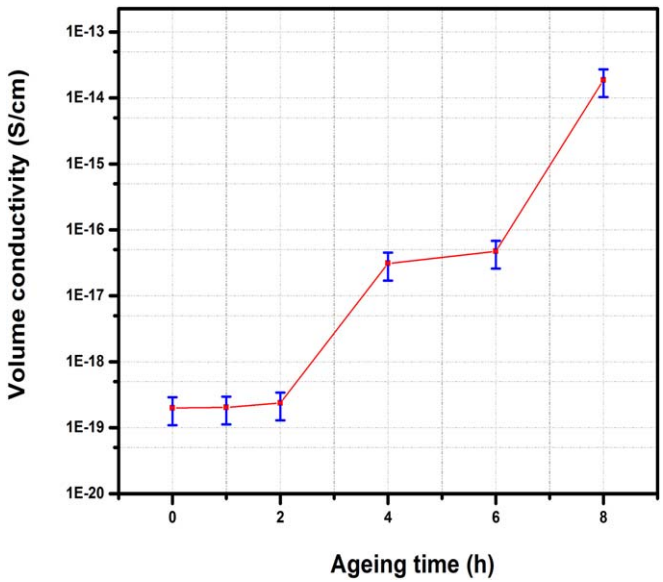


Figure 4. Evolution of volume conductivity with ageing time.

opposite direction to the charging current (CC) and represents the downward component of the charge current, especially under low electric fields and at low temperatures. The discharging current comprises at least two distinct elements: one related to depolarization current and the other to the drift of the total space charge (SC) within the polymer [17, 18].

In figure 2, it is clearly observed that the trajectory of the discharging current follows a different path from that of the charging current. It is noted that the discharging current (DC) changes polarity after 380 s and exhibits an anomalous peak with the same polarity as the charging current. The current flowing in the direction of the charging current is referred to as 'Anomalous Discharging Current' (ADC). Negative current is represented as positive in order to utilize a logarithmic scale. The experimental discharging current data can be effectively modeled using a Curie-von Schweidler expression [19–21]:

$$I_d(t) = 7 \times 10^{-3} \times t^{-0.94} \text{ if } t < 200\,s$$

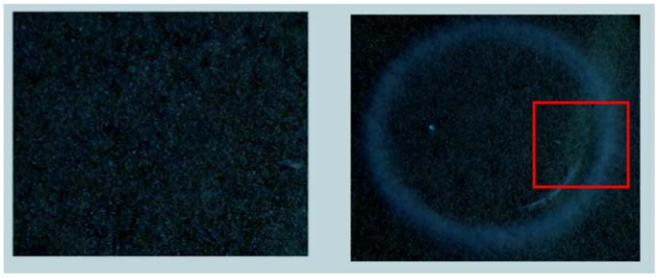


Figure 5. Effects of electrical ageing on the polymer surface (the square indicates the area where the measurements are made).

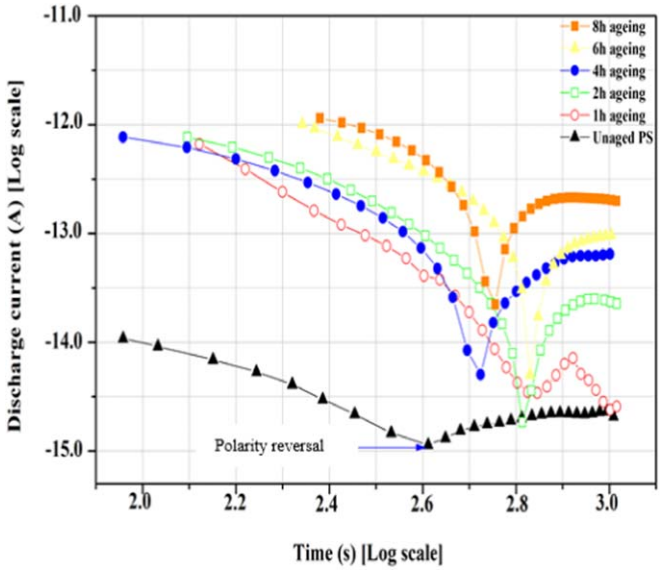


Figure 6. Effects of electrical ageing on discharging current versus time ageing.

and

$$I_d(t) = 1.3 \times 10^{-10} \times t^{-1.95} \text{ if } 200 < t < 350\,s$$

The anomalous discharge current (ADC) is observed in several polymers under high polarization and high temperature conditions [22–24], but its underlying mechanisms remain poorly understood. According to Tiwana *et al* [25], the difference between the discharge and charge current is likely due to the partial release of trapped charges, migrating towards one of the electrodes. Generally, ADC is associated with space charge injection from electrodes under high polarizing conditions and the metal-dielectric interface blocking effect [18]. However, this does not explain the present case, as PS samples are polarized by low fields at room temperature. The presence of ADC may be due to ionic charge impurities, with impurities like adsorbed or absorbed gas molecules playing a role in space charge accumulation in insulating polymers [26–28].

3.3. Effects of electrical ageing on the charging current

Figures 3 and 4 show the charging current dependency of time and the volume conductivity values of specimens as a function of ageing time, respectively.

As seen in figure 4, the volume conductivity value of polystyrene (2.0×10^{-19} S cm^{-1}) was found to be consistent with the previosuly reported values in literature [29]. However, these values are higher than ones corresponding to the specification of the test rig. The calculation of volume conductivity relies on parameters derived from measured charging currents, as well as the geometric characteristics of electrodes and samples. On the other hand, we observe that the electrical conductivity increases with the electrical ageing time. This increase is more pronounced when the ageing time is higher than 2 h. This increase in conductivity can be explained by several reasons such as (i) some molecular, chemical and/or microstructural alterations in bulk PS structure which generally occur as the formation of oxygenic species onto polymer backbone or end groups, (ii) formation

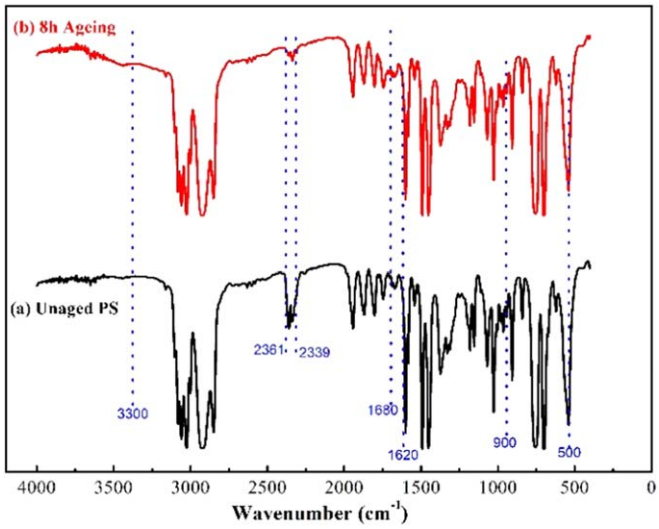


Figure 7. FT-IR spectra of PS: (a) virgin PS, (b) PS aged electrically for 8 h.

Table 1. FTIR band assignment for virgin PS.

Wavenumber (cm^{-1})	Assignment
4000–3300	OH
3159	C–H elongation
2940–3080	C–H aromatic (styrene)
2855	C–H symmetric
2731–2675	C–H elongation
2500–2632	C=O (R–C–O–O–H)
2361–2339	O–C–O stretching
2185–1950	C–O elongation
1900–1700	C=O
1670–1600	C=C stretching
1370–1028	C–O
1005–900	C–H vinyl (styrene)
921	C–C–H deformation
769–706	C–H aromatic (styrene)

of new traps, and (iii) deterioration of specimen surface notably by the formation of highly polar and hydrophilic hydroxyl groups that lead to moisture binding on the sample surface. The modification of the polymer structure may yield trap formation, which increases slowly with the ageing time. This increase is attributed to the creation of predetermined localized trap sites by the structure modification [27]. The increase in the conduction current with the ageing time is attributed to the increase in the formation of new traps [30, 31] and the acceleration of chemical and physical ageing processes induced by electrical field [32, 33]. After an electrical ageing process, an obvious degradation on the PS surface can be visually observed indeed by the formation of whitish ring erosion marks, the change in surface colour, and the existence of moisture as seen in the optical microscopy images given in figure 5. The presence of moisture reduces the dielectric characteristics of the polymer. Presence of H_2O molecules, either on the surface or in the bulk structure, can contribute to the charge mobility and promote their diffusion and dissipation. The H_2O molecules are able to dissociate to form hydronium ions (H_3O^+) and hydroxyl ions (OH^-).

3.4. Effects of electrical ageing on the discharging current

Figure 6 illustrates the typical discharging current plots of specimens as function of the electrical ageing periods. From these plots, the anomalous current are observed for all specimens. The intensity and time of reversal of discharging current (peak current) vary with the electrical ageing time. Time of the reversal current does not show a regular trend with the ageing time, while the peak current readily increases with ageing time. This relationship indicated that the space charge density formed in the sample increases with the ageing time. Such increase may be due to the new traps created after ageing. Moreover, under short-circuit conditions the

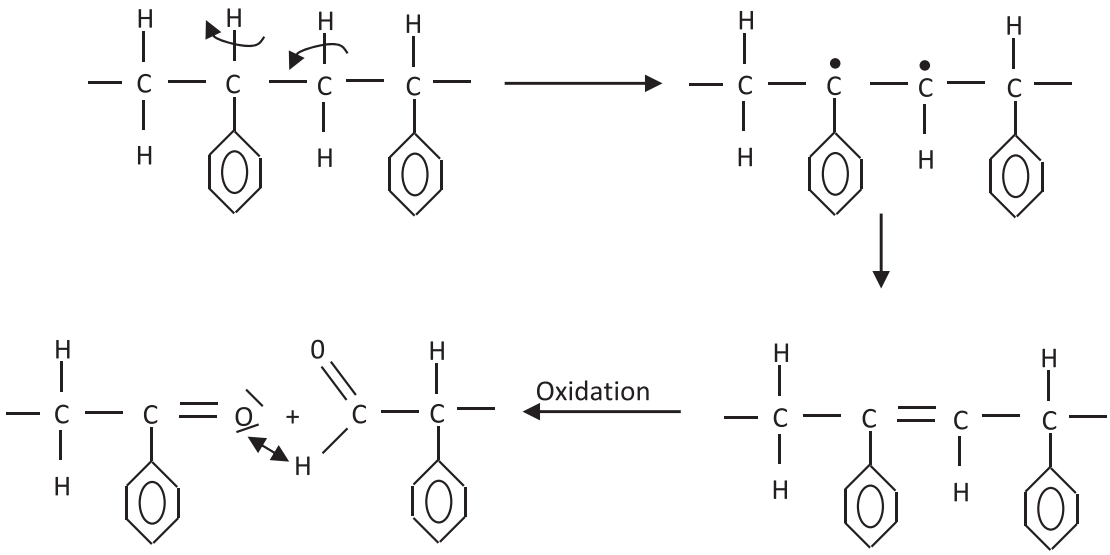
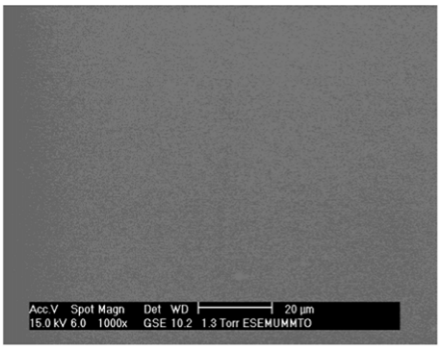


Figure 8. Chemical reaction of PS oxidation under electrical ageing.

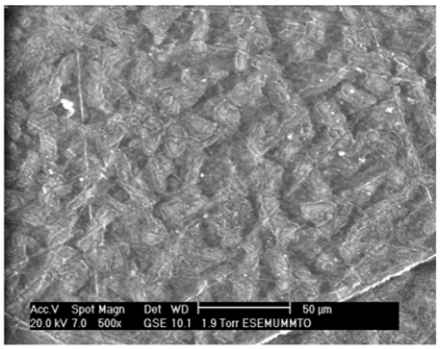
externally applied electric field no longer exists and electric field within material bulk is induced by accumulated space charges. This result was consistent with the findings reported in literature indicating that the intensity of ADC was increased with the charge density [29].

3.5. FT-IR analysis

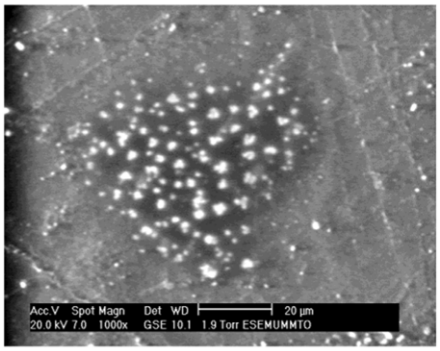
To identify the structural changes in the PS due to electrical ageing, FT-IR analyses. The specific band assignments for virgin PS are provided in table 1. Figure 7 displays the FT-IR spectra for both unaged (virgin) and electrically aged samples. The presence of C–O and C=O bands in virgin polystyrene suggests oxidation, which can be attributed to various factors, including the material's processing, manufacturing procedures, and exposure to environmental effects such as air, moisture, or other oxidizing agents. The peaks between 4000 and 3300 cm^{-1} correspond to characteristic OH streching. It is important to note that virgin PS typically does not contain OH groups. Their weak presence may attributed to oxidation processes of virgin PS in the presence of oxygen from the air or moisture. On the other hand, the intensity increase in the OH peak in aged polystyrene can result from several factors. OH groups can result from the breaking of chemical bonds or reactions with contaminants present in the material. It is important to note that the increase in OH groups can also be a sign of material oxidation, which is common in polymer ageing processes. The peaks at 2361 and 2339 cm^{-1} correspond to the characteristic vibrational frequencies of CO_2. This suggests the presence of CO_2, which could be related to oxidation in the air. For the aged sample, a reduction in these peaks is observed. Mechanisms of electrical discharge in the aged sample have led to chemical reactions that have consumed or altered the concentration of CO_2. The peaks observed between 2000 and 1500 cm^{-1} are attributed to the vibrations and deformations of the C–C and C–H bonds present in the carbon chain of polystyrene. It also exhibits peaks in the frequency range of 1500 to 1000 cm^{-1}, corresponding to the vibrations of C–C and C–H bonds. These peaks, while less informative than those associated with specific functional groups, are useful for identifying the material as polystyrene in the infrared spectrum. An increase in the intensity of the peaks associated with these functional groups is observed in the material subjected to electrical ageing, a phenomenon that can be explained by an increase in their concentration. Indeed, when polystyrene undergoes electrical ageing, it undergoes chemical reactions or structural modifications, leading to the accumulation of these functional groups present in the unaged polystyrene. For example, if chemical bonds break and give rise to functional groups, their concentration increases. We observe a peak in the frequency range of 1620 to 1680 cm^{-1} in the spectrum of unaged polystyrene, which is typically attributed to a carbon–carbon double bond (C=C). The presence of this peak could suggest the possible existence of impurities in the material. However, this peak disappears in the spectrum of electrically aged polystyrene, indicating that the ageing process has altered the chemical structure of the material, leading to the elimination or reduction of the C=C double bond. This modification may result from oxidation reactions that break the C=C double bond and result in the formation of carbonyl groups (C=O), which appear in the same region of the spectrum. Furthermore, a significant decrease in the infrared peaks is observed for the aged polystyrene in the frequency range between 900 and 500 cm^{-1}, corresponding to



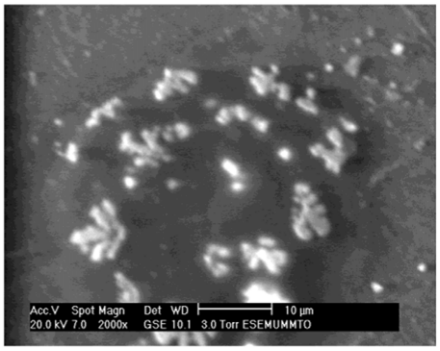
(a) Unaged PS (X1000 magnification with a scale of 20µm)



(b) PS electrically aged (X500 magnification with a scale of 50µm)



(c) PS electrically aged (X1000 magnification with a scale of 20µm)



(d) PS electrically aged (X2000 magnification with a scale of 10µm)

Figure 9. SEM micrographs of electrically aged PS (10 kV during 8 h) with different magnifications.

Table 2. EDX results.

	Atomic ratio	
	virgin PS	aged PS during 8 h
C	78.48	36.22
O	17.49	49.30
Si	2.53	1.08
Ca	1.50	2.64
Mo	—	0.54
K	—	4.45
Cl	—	5.78

C–C and C–H bonds. This indicates chain scission or degradation of the polymer chains in polystyrene due to electrical ageing.

Considering the results of FT-IR analysis and those breaking the C–H bond, reducing the number of C–H and C–C bands, polymer degradation can occur as follows: The break comes at a side C–H bond which leads to the formation of a double C=C bond. Since this bond is unstable, hydrogen bonding appears after oxidation. This polymer degradation mechanism is called 'chain radical'. Transformations can occur according to the following reaction (figure 8). It can be concluded that the electrical ageing induced significant structural change in PS based on the aforementioned FTIR analysis. This effect is generally ignored in industrial applications because such structural changes do not dramatically affect the bulk mechanical properties of thermoplastic parts. In this study, it has been clearly shown that effects of electrical ageing on the long term physical properties of PS parts should be taken into account in industrial applications.

3.6. SEM and EDX analysis

Figure 9 displays SEM micrographs of both virgin and electrically aged PS at magnifications of 500, 1000, and 2000. The virgin samples exhibit a highly smooth and uniform surface, as depicted in figure 9(a). In contrast, the electrically aged sample displays a rough and crinkled surface topography, visible in figures 9(b), (c), and (d). The surface damage may result from both electrical and chemical erosion during the ageing process. It is understood that electrical stress causes discharges and enlarges void sizes when it surpasses the partial discharge inception voltage [3]. Typically, these processes are irreversible, and their effects are cumulative, leading to progressive degradation throughout the material. The craters and damages on the PS surface represent a potential source of modification in trap characteristics. This alteration of trap energy, along with changes in their density and distribution, plays a crucial role in the variations observed in dielectric properties.

To quantify the elemental composition, specifically the ratios of O and C, before and after ageing, and to identify heteroatoms originating from impurities in the polymer, EDX analysis was employed. A noticeable distinction between the aged and unaged samples is evident, as illustrated in table 2. The atomic ratio of carbon decreases, while that of oxygen increases with ageing. Additionally, several inorganic elements such as Si, Ca, Mo, K, Cl, etc, are detected in the PS both before and after ageing.

These observations can be interpreted in relation to the previously described Infrared (IR) spectroscopy results. The increased presence of oxygen measured by EDX can be explained by changes in the chemical structure of the aged polystyrene (PS) surface, consistent with IR results where characteristic bands related to oxygenated groups (C=O) were observed, indicative of oxidation. Furthermore, the decrease in the amount of carbon in the surface region may be associated with the degradation of polystyrene. The results of infrared spectroscopy also revealed signs of chemical bond breakages.

Finally, the EDX results validate that the asymmetry observed in the DC and CC plots, as well as the emergence of ADC, is attributed to impurities within the sample. This implies that the spatial distribution of charge concentrations primarily occurs in regions containing morphological defects, inclusions, and impurities. The concentration of traps is likely high around various impurities present in the sample, thereby inducing the formation of space charges

4. Conclusion

This study investigates the time evolution of charging and discharging currents in a low-field polystyrene specimen. The steady-state current is achieved approximately 800 s after the application of a DC voltage. Anomalous discharging current, flowing in the direction opposite to the charging current, is also observed

around 350 s after the removal of the external field. This discharging current does not exhibit downward symmetry in comparison to the transient current.

The appearance of the anomalous current indicates the presence of space charge generated during the charging period, which becomes trapped in the material during the manufacturing and storage processes. Furthermore, experimental results suggest that electrical conductivity is a highly sensitive parameter to electrical ageing and emerges as a valuable diagnostic parameter for assessing the degradation of thermoplastics. This measurement can also be used as a fast and easy monitoring tool for the level of degradation as non-destructive testing method and provides valuable information.

Ageing induces variations in the intensity of discharging current and the time for reversal current. These phenomena can be explained in terms of chemical and structural modifications resulting from electrical ageing. Degradation issues in the polymer due to electrical ageing are confirmed through Fourier-transform infrared (FTIR), Energy-Dispersive x-ray (EDX) and scanning electron microscopy (SEM) analyses.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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