

STUDIES ON THE BEHAVIOUR OF LDPE FILM UNDER THE ACTION OF AN ELECTRIC FIELD

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The study aims to examine the impact of an electric field on low-density polyethylene (LDPE) samples and the subsequent evolution of their physical and chemical characteristics. The effect of an electric field on polyethylene samples has been studied using the following methods: Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), and electrical measurements. The results prove that structural and morphological changes in polyethylene also determine alterations to its electrical properties.

Keywords: Ageing, dielectric characterisation, high-intensity electric field, low-density polyethylene, structural characterisation.

1. INTRODUCTION

Polyethylene is a thermoplastic polymer obtained by ethylene polymerization. Polyethylene is the most widely used plastic material, accounting for more than 70 % of the total plastic production. Polyethylene has a low and stable dielectric constant across all frequencies and very high insulation resistance. It is successfully used in the electrical, electronics, automotive, and aviation industries, in the manufacture of electrical insulation materials, packaging materials, pipes, films, and many parts used in various technical applications.

Significant results have been achieved in the development of materials, production efficiency, and material testing in the fields of power transmission, telecommunications, and other technical applications [1], [2].

Thermoplastic polymers with superior mechanical durability and chemical resistance are used to manufacture insulating materials for underground electrical cables. These thermoplastic polymers have low production costs and are easy to process [3].

The electric field applied to the polymer

material during operation causes changes in its electrical properties. For homogeneous materials, the thermal effect of electric current can be a degrading factor. This leads to an increase in the temperature of the polymer, resulting in an acceleration of physical and chemical transformations.

For homogeneous and non-homogeneous materials, electrical degradation correlates with dielectric breakdown through the phenomenon of partial discharges (PD). Reversible and irreversible phenomena acting simultaneously can lead to a decrease in the dielectric strength of the material [4]–[6].

Various studies have been conducted on the influence of the electric field on polyethylene and cross-linked polyethylene (XLPE). Still, the physical and chemical changes induced by electrical stress on insulating materials are not fully understood. This paper studies the behaviour of LDPE samples at a relatively high electric field intensity and over a short time, compared to previous studies.

2. MATERIALS AND METHODS

2.1. Experimental Setup for Material Ageing

Commercially available low-density polyethylene (LDPE) samples with a thickness of 0.14 mm and a density of 0.912 g/cm³ at a temperature of 21 °C were used. The electrical ageing test was configured as shown in Fig. 1.

The samples were cut into 70 mm diameter discs and placed between two 50 mm diameter Rogowski electrodes at ambient temperature to create an electric field. An electric field of 45 kV/mm was then applied.

The AC voltage source was connected to the upper electrode, while the source ground was connected to the lower electrode. The applied voltage was supplied by a 110 kV transformer with a power output of 3.5 kVA at a frequency of 50 Hz. An autotransformer controls the test voltage. At certain intervals, the aged samples are removed to perform the relevant measurements for material and electrical characterisations [7], [8].

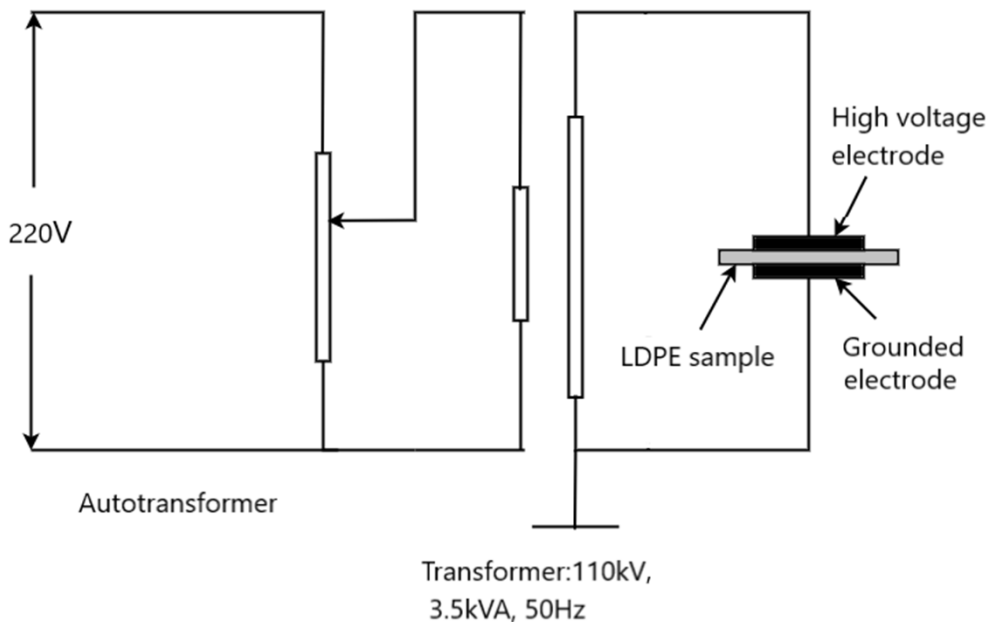


Fig. 1. Experimental setup for the electrical aging study.

2.2. Characterisation Methods

2.2.1. Material Characterisation

The Tensor 27 FTIR spectrometer from Bruker Optics was used for IR analysis to study the structure of the samples. The spectrum graph was obtained by averaging 64 scans, in the wavelength range ($4000 \div 400$) cm^{-1} , with a resolution of 4 cm^{-1} .

In the case of XRD measurements, a rotating anode SmartLab 9kW XRD diffractometer from Rigaku Corporation was used to study the diffraction spectra of the samples. The cuboid samples on which the

measurements were performed have a volume of $10 \times 10 \times 0.14 \text{ mm}^3$.

The Nova NanoSEM 630 field emission scanning electron microscope, manufactured by FEI Company, was used to study the morphological appearance of low-density polyethylene samples. A ($4 \div 5$) nm thick layer of gold was deposited on the surface of the samples analysed to eliminate the effect of the material's dielectric properties.

2.2.2. Electrical Measurements

The dielectric constant ϵ_r stands for the electric charge stored in a dielectric material positioned between two metal plates, relative to the electric charge stored between

the same metal plates in the absence of the dielectric material. The dielectric constant shows the characteristic of a dielectric material to store electrical energy.

The dissipation factor or loss tangent ($\tan \delta$) is a measure of a dielectric material that shows the energy losses of a dielectric material subjected to an alternating electric field. The dissipation factor is an indicator of the amount of electrical energy converted into heat by a dielectric material.

Dielectric spectroscopy measures the dielectric properties of materials that depend on the operating frequency. The

measurement principle is based on the interaction of the applied electric field with the electric dipole of the material. In dielectric spectroscopy, the impedance of the sample and the characteristics of electrical energy storage and dissipation are measured in a frequency range. The sample being analysed is placed between two parallel plates fixed in a device, as shown in Fig. 2a.

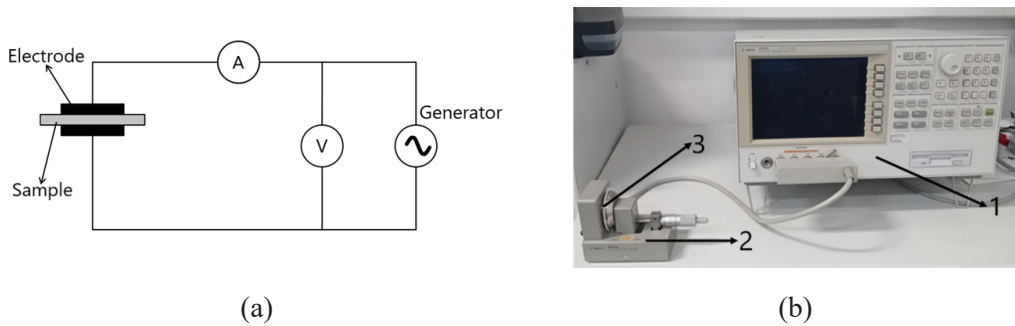
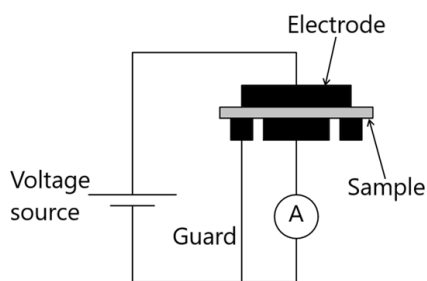


Fig. 2. Dielectric spectroscopy technique: (a) basic electrical circuit schematics; (b) experimental setup configuration (1 – precision impedance analyzer, 2 – dielectric test fixture, 3 – LDPE test sample).

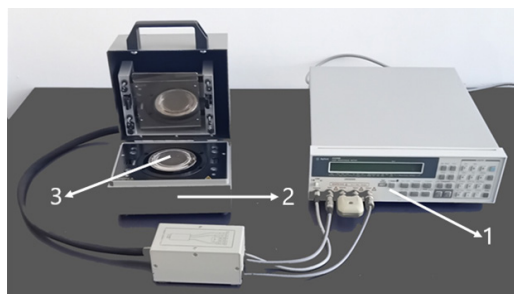
The sample impedance Z is determined at a given frequency for an applied current or voltage sinusoidal waveform. Complex impedance is defined as a vector in the complex plane, expressing the ratio between the voltage and current values, as well as the phase difference between them [9]–[13]. In this paper, the 4294A impedance analyzer, together with the 16451B measurement cell, both from Agilent Technologies, was used to measure the dielectric constant and dissipation factor of the samples (Fig. 2b). The measurements were performed according to the ASTM D150 standard.

Volume electrical resistance is the electrical resistance between two opposite sides of an insulating material in the form of a cube with a side length of 1 cm. In technical

applications requiring high insulation characteristics, the volume electrical resistance must have the highest possible values. In this case, depending on the applied electrical voltage and the measured value of the electric current passing through the sample, the electrical resistance of the material can be determined (Fig. 3a). Once the dimensions of the sample are known, its volume electrical resistivity can be determined [14], [15]. The 4339B high resistance meter and the 16008B measuring cell, from Agilent Technologies, were used to measure the electrical conductivity of the samples studied with a measurement range of up to $4 \times 10^{18} \Omega\text{cm}$, in accordance with ASTM D257 standard (Fig. 3b).



(a)



(b)

Fig. 3. Electrical resistance measurement technique in direct current conditions: (a) basic schematic diagram of the employed electrical circuit; (b) experimental setup to measure the volume electrical resistance (1 – high-resistance meter, 2 – resistivity cell, 3 – LDPE test sample).

3. RESULTS AND DISCUSSION

3.1. FTIR Characterisation

Figure 4 shows the FTIR spectra of the studied material, characterised by absorption bands that can be attributed to stretching

(2915 and 2848 cm^{-1}) or deformation (1471, 1463, 730, and 719 cm^{-1}) of the C-H bonds in the methylene group of polyethylene.

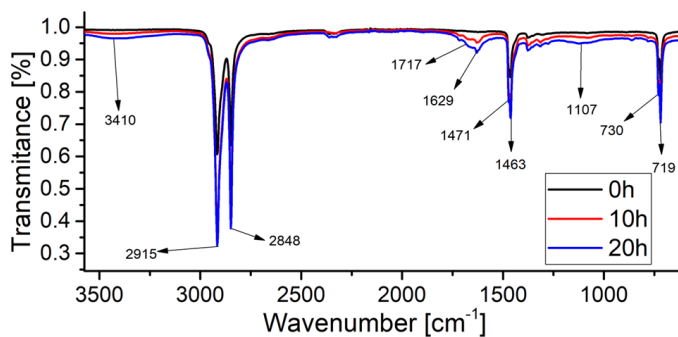


Fig. 4. FTIR spectrum of LDPE samples, pristine (0 h) and aged for 10 h and 20 h.

Under the action of the applied electric field during the ageing process, the phenomenon of thermolysis occurs in the studied material. The polymer chains break down, and oxidation and cross-linking occur. In the spectra of the samples subjected to degradation, bands of reduced intensity (1717 cm^{-1}) can be observed, which may be due to the vibration mode of the C=O bonds in the

carbonyl group that may occur as a result of polymer oxidation. Also, bands of lower intensity (1629 cm^{-1}) are observed that may be due to C=C bonds, which appear as a result of polymer cross-linking. In the spectra of the aged LDPE samples, bands of reduced intensity can be observed in the 4000÷3000 cm^{-1} range, which may be associated with OH bonds due to the increased

wettability of the polymer, resulting in moisture absorption.

As a result of thermolysis, in the first hours of aging, the characteristic absorption peaks of polyethylene decrease in intensity. Then, the depletion of oxygen molecules causes the cross-linking phenomenon to increase quantitatively, with the distinc-

3.2. XRD Characterisation

The XRD diffraction patterns of the LDPE samples are shown in Fig. 5. The width of the characteristic diffraction bands (21.48° and 23.77°) indicates the semi-crystalline structure of the studied material. The peaks of the diffraction bands of the electrically aged samples have not changed position; they appear at the same 2θ values. This implies that no new crystalline phase

occurs in the samples exposed to the electric field. There is a slight decrease in the crystallinity of the material under the action of the electric field. The phenomenon of thermolysis leads to the cleavage of polymer chains, and oxidation reactions occur, leading to a decrease in the crystalline domains in the material [18].

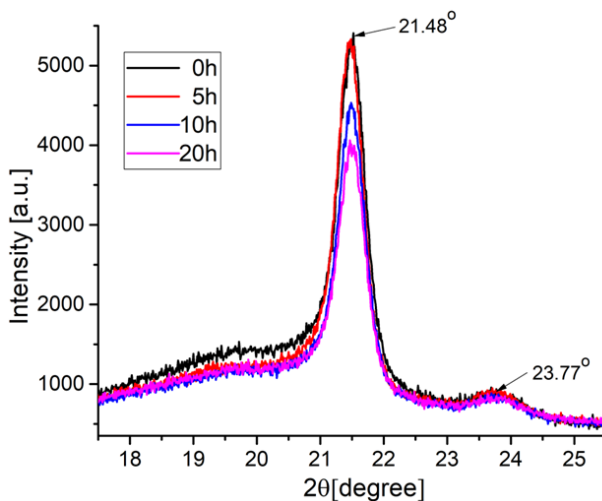


Fig. 5. XRD diffraction patterns of the LDPE samples aged under an intense electric field.

3.3. Surface Microstructural Characterisation

Figure 6 shows SEM images at a magnification of $20,000\times$ for the unaged sample and the samples subjected to electrical stress for 10 and 20 hours. The surface of the unaged sample is homogeneous and relatively smooth, exhibiting only minor

morphological changes caused by the physical handling of the LDPE film. In contrast, the surfaces of the samples exposed to the electric field display roughness and slight irregularities, along with the appearance of minor defects. This degradation can

be attributed to the evaporation of volatile polymer compounds that occur during the

breakdown process under the influence of the electric field [19], [20].

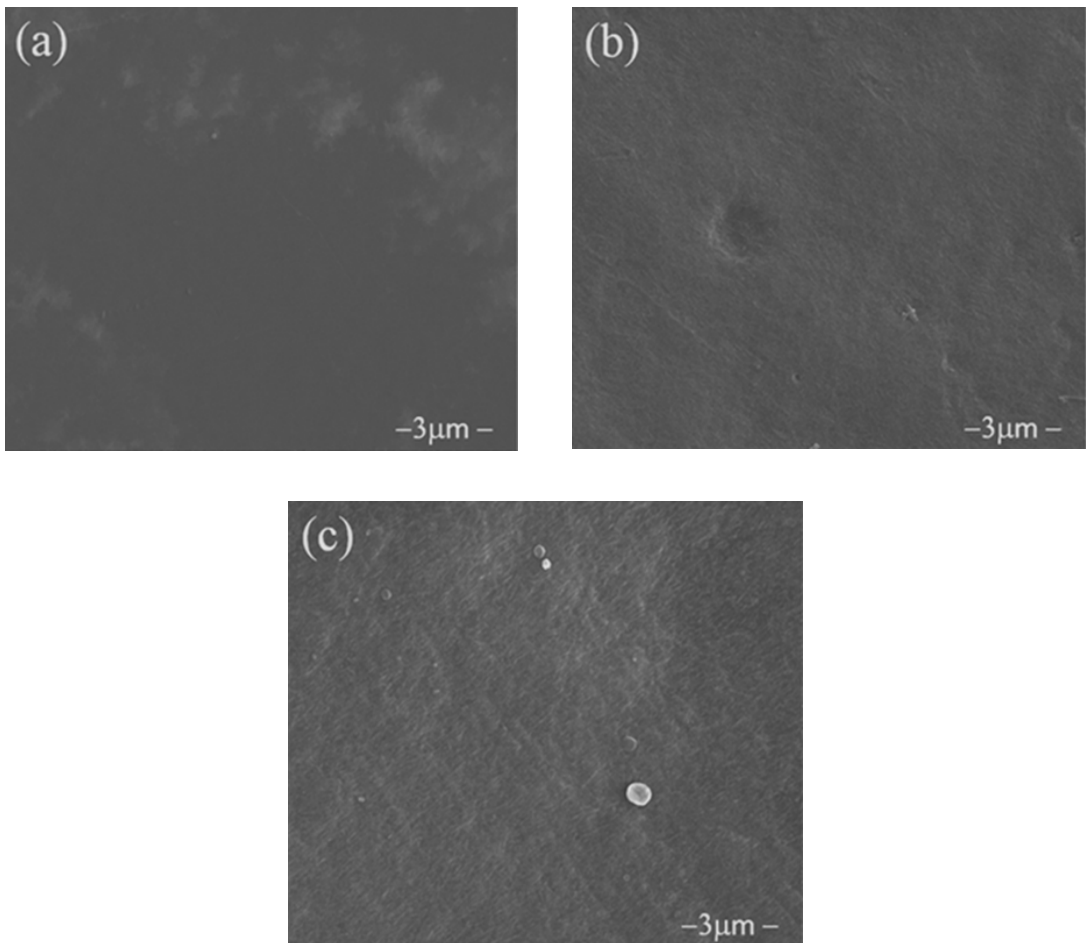


Fig. 6. SEM images of the surface of the unaged LDPE sample (a), aged for 10 hours (b), and for 20 hours (c).

3.4. Electric Parameter Determination

3.4.1. Dielectric Constant

Type of chemical bonds, degree of cross-linking, and polarizability determine the magnitude of the material's dielectric constant. Also, the dielectric constant is influ-

enced by the number of dipoles that orient themselves when an electric field is applied. Oxygen diffusion, increased oxidation rate, and the appearance of polar hydroxyl and

carbonyl bonds contribute to the total polarization of the material and, implicitly, to an increase in the dielectric constant.

Dielectric constant evolution as a function of frequency according to the exposure times of the material to the electric

field is shown in Fig. 7. The decrease in the dielectric constant can be explained by the appearance of the pronounced cross-linking phenomenon and by the rearrangement of the polymer structure from a molecular point of view [9]–[13].

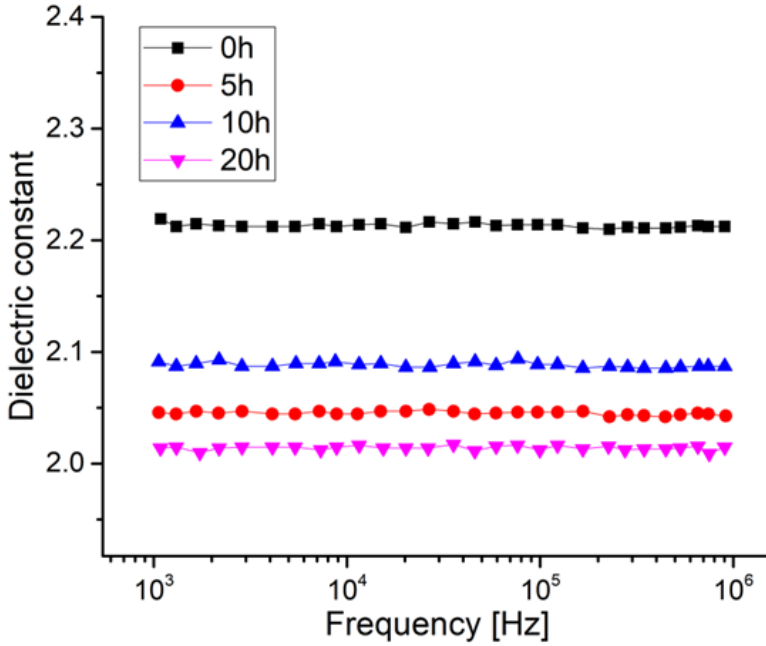


Fig. 7. Dielectric constant as a function of frequency for the LDPE samples studied.

3.4.2. Dissipation Factor

In the case of polyethylene, the dissipation factor is influenced by the occurrence of oxidation, so the appearance of carbonyl compounds that have electrical asymmetry leads to an increase in the dissipation factor, meaning that these compounds play a very important role in increasing losses and disrupting the dielectric properties of a material [10]–[13].

According to Fig. 8, electrical aging causes the dissipation factor to decrease

after 5 hours of electrical aging, followed by a slight increase in its value. The overall significant decrease in the dissipation factor value may be due to the pronounced cross-linking of the material. The increase in the dissipation factor may be due to water absorption, thereby increasing the number of polar groups and thus increasing the energy dissipated in the form of heat.

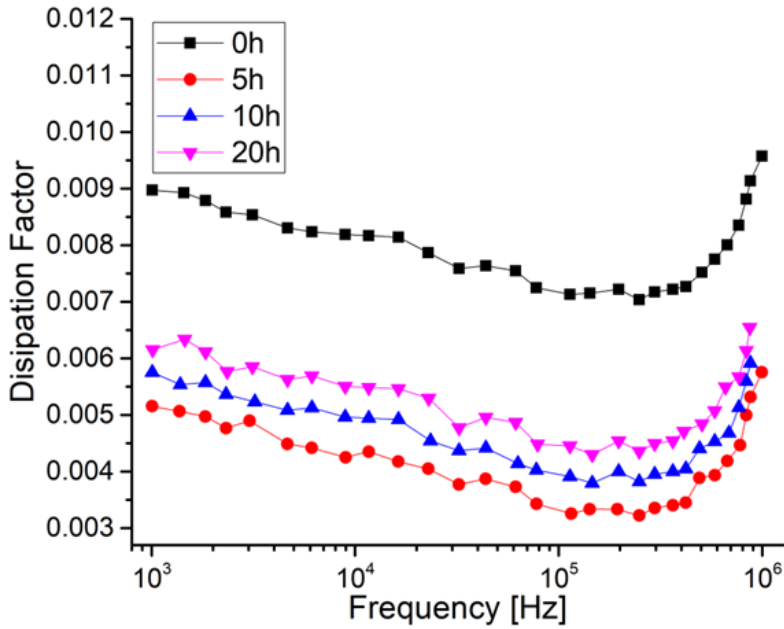


Fig. 8. Dissipation factor as a function of frequency for the LDPE samples studied.

3.4.3. Electrical Conductivity Results

The predominant cross-linking phenomenon causes the number of free radicals to decrease, implicitly leading to a decrease in electrical conductivity of the LDPE [14],

[15]. Table 1 lists the values of both the resistivity and volume conductivity of the samples.

Table 1. Measured Resistivity and Volume Conductivity of the Samples

Time [h]	Resistivity [Ωm]	Conductivity [S/m]
0	0.3408×10^{14}	2.9342×10^{-14}
5	2.7979×10^{14}	0.3574×10^{-14}
10	0.8767×10^{14}	1.1406×10^{-14}
20	5.0556×10^{14}	0.1978×10^{-14}

During the aging process of LDPE samples, a decrease in volume electrical conductivity is observed, as shown in Fig. 9. This can be associated with the loss of vola-

tile compounds from the polymer during degradation. On the other hand, moisture absorption in the material causes a slight increase in electrical conductivity.

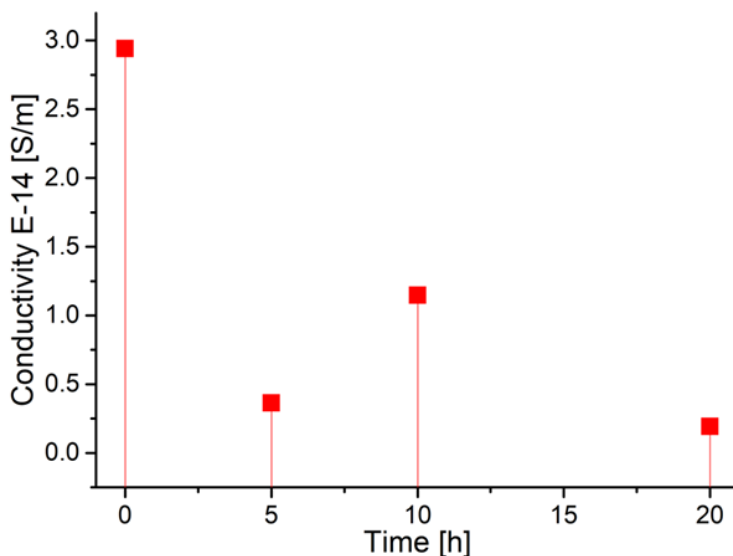


Fig. 9. Volume electrical conductivity of the LDPE samples studied.

4. CONCLUSIONS

The paper has studied how the high-intensity electric field affects LDPE film samples over a period of up to 20 hours. The samples have been subjected to an alternating electric field of 45 kV/mm.

Measurements have revealed structural and morphological changes in the polymer, as well as changes in its electrical properties. FTIR measurements have indicated the presence of oxidation and cross-linking phenomena, as well as hydroxyl groups. XRD analysis has revealed a slight decrease in crystallinity due to the breaking of polymer

molecular chains and oxidation. SEM analysis has shown morphological changes on the surface of the electrically aged samples. During the initial stage of electrical ageing, the dielectric constant and dissipation factor have decreased due to cross-linking. Additionally, there is a slight tendency for electrical conductivity to decrease.

The results of this research will enable the evolution of LDPE film properties to be studied over more extended periods, to develop new materials with improved performance.

ACKNOWLEDGEMENT

The research has been funded from the project “National Platform for Semiconductor Technologies”, contract no. G 2024-85828/390008/27.11.2024, SMIS

code 304244, co-funded by the European Regional Development Fund under the Programme for Intelligent Growth, Digitization, and Financial Instruments.

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