



ALMA MATER STUDIORUM  
UNIVERSITÀ DI BOLOGNA

ARCHIVIO ISTITUZIONALE  
DELLA RICERCA

## Alma Mater Studiorum Università di Bologna Archivio istituzionale della ricerca

Breakdown at Medium Frequencies of Thermally-Aged Polyester Films

This is the final peer-reviewed author's accepted manuscript (postprint) of the following publication:

*Published Version:*

Breakdown at Medium Frequencies of Thermally-Aged Polyester Films / Villibor J.; Zheng C.; Bak C.L.; Suraci S.V.; Rumi A.; Cavallini A.. - ELETTRONICO. - (2023), pp. 1-4. (Intervento presentato al convegno 2023 IEEE Electrical Insulation Conference, EIC 2023 tenutosi a Quebec City, QC, Canada nel 2023) [10.1109/EIC55835.2023.10177223].

*Availability:*

This version is available at: <https://hdl.handle.net/11585/939200> since: 2023-08-21

*Published:*

DOI: <http://doi.org/10.1109/EIC55835.2023.10177223>

*Terms of use:*

Some rights reserved. The terms and conditions for the reuse of this version of the manuscript are specified in the publishing policy. For all terms of use and more information see the publisher's website.

This item was downloaded from IRIS Università di Bologna (<https://cris.unibo.it/>).  
When citing, please refer to the published version.

(Article begins on next page)

This is the final peer-reviewed accepted manuscript of:

**J. Villibor, C. Zheng, C. L. Bak, S. V. Suraci, A. Rumi and A. Cavallini, "Breakdown at Medium Frequencies of Thermally-Aged Polyester Films," 2023 IEEE Electrical Insulation Conference (EIC), Quebec City, QC, Canada, 2023, pp. 1-4.**

The final published version is available online at:

<https://doi.org/10.1109/EIC55835.2023.10177223>

Terms of use:

Some rights reserved. The terms and conditions for the reuse of this version of the manuscript are specified in the publishing policy. For all terms of use and more information see the publisher's website.

*This item was downloaded from IRIS Università di Bologna (<https://cris.unibo.it/>)*

***When citing, please refer to the published version.***

# Breakdown at medium frequencies of thermally-aged polyester films

J. Villibor  
Universidade Federal de Itajubá  
Brazil  
jansen@lat-efei.org.br

C. Zheng  
University of Aalborg  
Aalborg, , Denmark  
czhe@energy.aau.dk

C. Leth Bak  
University of Aalborg  
Aalborg, , Denmark  
clb@energy.aau.dk

S.V. Suraci  
LIMES – Department of Electrical  
Engineering, University of Bologna  
Bologna (BO), Italy  
simone.suraci@unibo.it

A. Rumi  
LIMES – Department of Electrical  
Engineering, University of Bologna  
Bologna (BO), Italy  
alberto.rumi@unibo.it

A.Cavallini  
LIMES – Department of Electrical  
Engineering, University of Bologna  
Bologna (BO), Italy  
andrea.cavallini@unibo.it

**Abstract**—This article investigates the evolution of electrical properties of polyethylene terephthalate (PET) with thermal aging. Electrical tests are performed within a wide range of frequencies in order to simulate the application conditions of insulating materials in inverter-fed machines. In particular, for the aging conditions considered, the real part of permittivity showed to be correlated with the concentration of the degradation species, investigated through FTIR. On the contrary, the dielectric breakdown at high frequencies showed to be independent from the corresponding values of the dissipation factor ( $\tan\delta$ ). This behavior would imply that thermal runaway is not the principal phenomenon leading to the dielectric failure.

**Keywords**—Power electronics, thermal stress, breakdown, polyester

## I. INTRODUCTION

Nowadays, in most industries, the final energy conversion stage is done through power electronic converters. Often in automotive and energy sectors, the mechanical to electrical conversion (and vice versa) is performed with a drive, comprising a converter and a rotating machine. The use of converters leads to the generation of overvoltages, an issue that is aggravated by the increased voltages required as a result of the higher power demands [1]. The high power involved causes both significant voltages and currents and consequently electrical and thermal stresses. Hence, the proper design of the electrical insulation is a key step for ensuring long service lifetimes of the equipment with sufficiently high levels of reliability.

The electrical stress from power electronics does not solely present increased amplitudes due to the mentioned overvoltages but also features different waveshape characteristics. One of the main distinctions to the sinusoidal AC grid voltage is the increased harmonic content of the converter-produced waveform. The high-frequency harmonics can enhance the stress on the insulation system, and increased dielectric losses can contribute to premature failures of the system [2]. Besides, it is sometimes unclear how to evaluate the condition of the insulation, since methods developed for AC 50Hz/60Hz sinusoidal systems may provide information unrelated to the failure mode.

In this context, research efforts have been focusing on the development of innovative design solutions involving new insulations with high performance and endurance. Among others, polyethylene terephthalate (PET) is widely used as primary insulation due to its good thermal stability and excellent insulation resistance. This article discusses the effects of thermal stress on PET films focusing on its impact on the capability of the insulation to withstand voltage waveforms with an elevated frequency content. In order to properly mimic the degradation, the material may face during its service life, thin insulation films (50  $\mu\text{m}$ ) were subjected to accelerated thermal aging at 180 °C in cycles lasting 10 days each.

Dielectric spectroscopy was performed to locate the dielectric losses peak at different aging stages. To simulate the worst-case scenario, the frequencies associated with those peaks (up to 40 kHz) were selected and used for the AC breakdown tests, thus assessing the evolution of the PET dielectric strength with aging.

## II. EASE OF USE

### A. Materials and aging

Commercial PET films with thickness of  $\sim 50\mu\text{m}$  were provided by IDI Fabrication EMT. Specimens are obtained by cutting 3x3 cm sheets from the delivered commercial foil.

To simulate long-term aging conditions, accelerated aging was performed in air oven at temperatures 60°C higher than the reference temperature for the class (120°C), corresponding to 180°C, for 30 days. Samples were removed from oven for testing every 10 days (Table 1). Prior to analyses, samples were put in vacuum oven at 50°C for at least 24h to remove moisture and contaminants from the surface.

TABLE I. AGING INFORMATION

Sample name	Aging duration (days)	Aging temperature
Unaged	0	180°C
Aged 1	10	
Aged 2	20	
Aged 3	30	

## B. FTIR measurements

Fourier Transform Infra-Red (FTIR) spectroscopy was performed on both neat and aged samples to investigate the oxidation stage throughout aging. Analyses were performed using a Bruker Alpha analyzer in ATR mode. As ATR measurements are not quantitative, FTIR spectra were normalized using the  $-CH_2-$  peak at  $1472\text{ cm}^{-1}$ , which is not expected to be modified by aging.

From this analysis, it is possible to derive the carbonyl index as:

$$CI = \frac{A_{1715\text{cm}^{-1}}}{A_{1472\text{cm}^{-1}}} \quad (-) \quad (1)$$

where  $A_{1715\text{cm}^{-1}}$  is the absorbance peak height of the carbonyl group and  $A_{1472\text{cm}^{-1}}$  is the absorbance peak height of the  $-CH_2-$  group. This quantity provides a quantitative insight of the oxidation level inside the polymer, and it has been widely used in the literature [3-5].

## C. Dielectric spectroscopy measurements

Dielectric response over frequency was measured by means of Novocontrol Alpha-A Dielectric Analyzer with applied voltage of  $3V_{rms}$ . The analyzed frequency range is  $10^1$ - $10^6$  Hz, allowing the investigation of both the lower-frequency region of dipolar polarization mechanisms along with the higher-frequency region of interfacial ones. Prior to testing, samples were metallized in gold through plasma cold sputtering to guarantee efficient adhesion between the material and cell electrodes.

## D. AC dielectric strength measurements

Dielectric breakdown values were obtained by means of a resonant generator capable of reaching 10kV for frequencies in the range of 10kHz to 45kHz. The samples were fed with the high-frequency sinusoidal voltage while immersed in a vegetal oil bath to avoid the inception of partial discharges, that would otherwise contribute to the degradation through surface erosion. The samples were positioned between a round HV electrode featuring a 5 mm diameter and a ground plate. The voltage was increased in a ramp at a ratio of 100 V/s, according to standard [6] and the breakdown voltage was recorded at the instant prior to the short circuit. The voltage value was collected by a Tektronix P6015A scope probed to a Picoscope 4262 oscilloscope.

# III. RESULTS AND DISCUSSION

## A. FTIR results

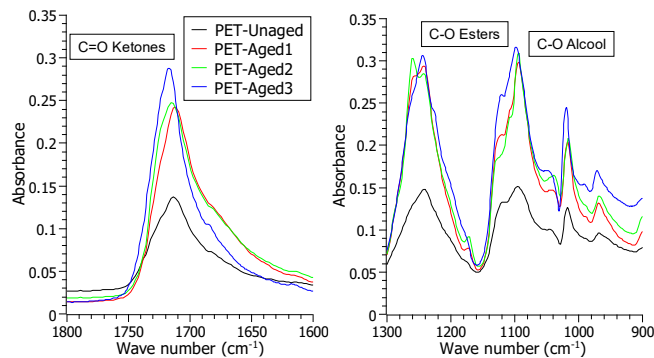


Fig. 1. FTIR spectra related to (a) Ketones, (b) Esters and Alcohols groups for the different aging levels.

In this section, the results coming from FTIR analysis are presented and discussed. This provides an outlook of the degradation level of the tested materials with aging. For the sake of brevity, normalized spectra related to oxidation functional groups only are reported (Figure 1).

From Fig.1 it is possible to notice an increase of the absorbance peak height of the different degradation groups with aging. This behavior confirms a significant oxidation level of the material by the formation of various oxidized groups namely: ketones, esters, and alcohols. In particular, the biggest increase of the peaks occurs during the first aging period. Additional aging slightly modifies the peak distribution with the formation of an additional peak related to esters at  $1243\text{ cm}^{-1}$  and the corresponding disappearance of the peak at  $1260\text{ cm}^{-1}$ . This behavior, already investigated in literature [7], may be related to the different morphology of the ester group arising during aging.

For an easier comparison, the values related to the carbonyl index were normalized by the initial value (Figure 2). Figure 2 confirms the abovementioned trend, and it is evident how the first aging level causes a significant increase of the oxidized groups which then experience a slower growth.

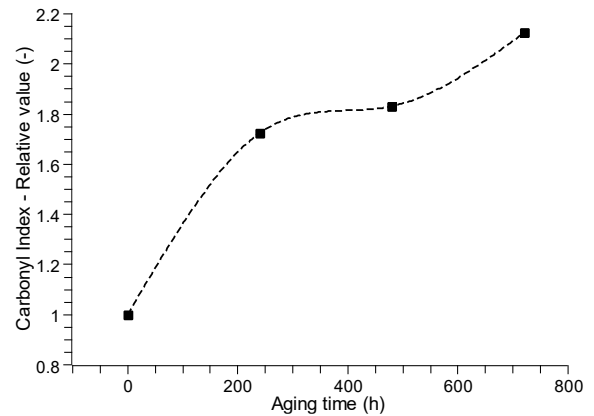


Fig. 2. Carbonyl index as a function of aging time.

## B. Dielectric spectroscopy results

Figure 3 reports the trend of the real part of permittivity (Fig.1.a) and  $\tan\delta$  (Fig.1.b) as a function of frequency for the different tested materials.

The real part of permittivity experiences a significant increase during the first aging period throughout the analyzed frequency region. Further aging does not cause appreciable variation of the property, being almost constant at  $\sim 3.9$  at 50Hz.

The dielectric losses, here reported as  $\tan\delta$ , exhibit a polarization peak at  $\sim 500$  kHz, which may be linked to the relaxation of dipolar species in the polymer (dipolar polarization response). This peak undergoes a continuous decrease with aging, suggesting the concentration reduction of the corresponding species.

On the contrary, the dielectric response related to the interfacial polarization ( $<10$  Hz) depicts a different behavior. The first aging periods lead to the increase of the losses in the low frequency, while the 3<sup>rd</sup> aging level causes the reduction of the dielectric loss value. This trend is likely imputed to a different arrangement of interfaces inside the polymers e.g., crosslinks and additive consumption.

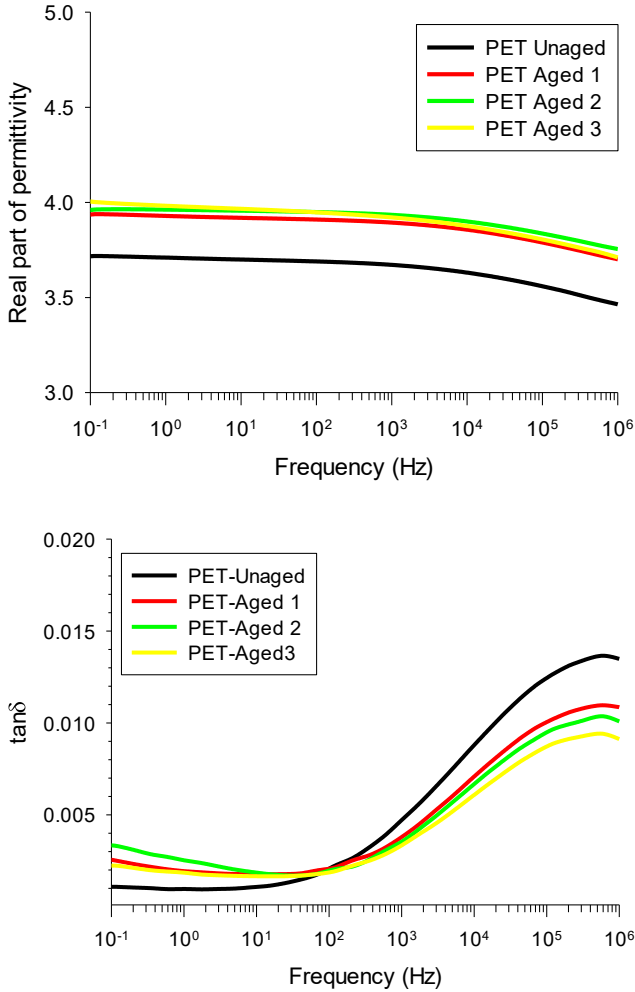


Fig. 3. (a) Real part of permittivity and (b)  $\tan\delta$  as a function of frequency for the different aging levels.

### C. AC dielectric strength results

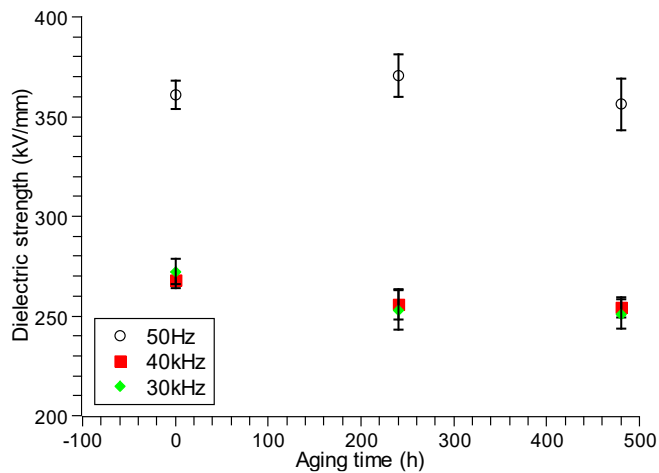


Fig. 4. Dielectric strength as a function of aging time under AC voltage at 50 Hz ( $\circ$ ), 40 kHz ( $\square$ ) and 30 kHz ( $\diamond$ ).

Figure 4 reports the trend of dielectric strength as a function of aging time for three different frequencies, namely: 50Hz, 40 kHz and 30 kHz. As expected, the increase of frequency by three orders of magnitude brings to the significant reduction of the dielectric strength ( $\sim -30\%$  of the

reference 50Hz value). On the contrary, negligible differences are present comparing the 40 and 30kHz responses.

The performed aging highlights little modifications of the breakdown values particularly at lower frequencies (50 Hz). Higher frequencies cause a more evident decreasing of the property which, however, experiences a limited reduction ( $\sim -8\%$ ).

## IV. DISCUSSION

Accelerated thermal aging has proven to modify most of the investigated material properties. Given the oxidizing environment in which aging is performed, it is possible to consider both chain scission and oxidation as major degradation phenomena. As known, thermal stress mainly leads to the breaking of the long polymer chains into shorter reactive ones (chain scission). The resulting chains are free to bond with environmental oxygen or each other, leading to stable groups. In particular, during the first phase of aging, oxidation is limited by the presence of antioxidants which are supposed to be present in adequate quantity in the commercial film. After running out of antioxidants, oxidation can be incepted leading to the arising of new species whose concentration is believed to increase throughout aging. From the presented FTIR analysis (Fig. 1), it can be stated that the degradation species formed during aging are mainly: esters, ketones and alcohols. However, due to chemical kinetics reasons, the increase in concentration of these species is occurring at a slower pace after the first aging period, as already seen in [8].

These species are characterized by a very high dipolar moment [8]. Thus, their response to an external electric field (polarization) is supposed to modify the dielectric spectra, as confirmed by Fig. 2. Similar to FTIR spectra, the real part of permittivity significantly increases during the first aging period and then it slows its rise. This confirms the strict correlation between the concentration of oxidized species and their dipolar response, as already proven in [3, 8].

On the other hand, the dielectric losses do not follow the abovementioned trend, starting a continuous decrease of the dipolar polarization peak.

Indeed, the initial consumption of antioxidants may lead to a reduction of the polarization peak due to their conversion to species starting a lower dipolar momentum. Additionally, oxidized chains, which are derived from the initial chain scission, are smaller dipolar molecules whose dielectric relaxation may be placed outside the tested frequency region ( $> 1\text{MHz}$ ), further reducing the relaxation peak (Fig.2.b).

Similar reasoning may be done focusing on the interfacial polarization frequency region ( $<10\text{Hz}$ ). Broken chains which do not react with oxygen may randomly arrange themselves into different structures modifying the interfacial arrangement in the polymer. As a result, the molecular relaxation related to the interfacial polarization may differ along with aging, depicting a non-monotonic variation of the property (Figure 3.b).

Despite the advanced oxidation state of the investigated polymer, the dielectric strength does not appear to appreciably change from its initial value. In the case of dielectrics without defects, dielectric breakdown may be triggered by a variety of causes, one of these being thermal runaway due to thermal instability inside the polymer [9]. For this reason, the analysis of the dielectric losses can be a good indicator for the prediction of the endurance of the insulation system. Given the results reported in Section III, it is evident how this correspondence may be considered the case for tests

performed at power frequencies (50Hz), for which no change is seen for both the  $\tan\delta$  values and the dielectric strength. Referring to the high frequencies, the decrease of the dielectric losses (Figure 2.b) would have suggested an improvement (increase) of the dielectric strength due to lower amount of energy thermally dissipated. Nonetheless, it does not apply to the tested materials (Fig.4). This peculiar behavior suggests that the dielectric losses, for the aging level here considered, are not sufficiently high to provide significant contribution to the thermal breakdown, possibly explaining the almost constant BD values.

## V. CONCLUSIONS

In this article, the chemical degradation of thin PET films is presented and correlated with some of the most important electrical properties related to their applications into power electronics.

The performed accelerated thermal aging resulted in the arising of new degradation species starring oxygen groups e.g., ketones, alcohols, and esters. The biggest increase in concentration of these groups occurred during the initial stages of aging, while additional aging slightly modified their concentration.

Dielectric spectroscopy results provided useful insights on the dipolar response of the aged material. In particular, it has been demonstrated that the arising of these polar groups considerably affects the real part of permittivity. On the contrary, the dielectric losses experienced a reduction of the peak related to the dipolar polarization probably due to the shifting towards higher values of the relaxation frequencies of the created degradation species.

Despite the reduction of the dielectric losses at ~40kHz, the corresponding AC dielectric strength did not experience a significant modification from the initial value. This behavior may be imputed to the fact that (a) the reported dielectric losses are not high enough to cause a thermal instability during the electric field ramp applied during testing, or (b) the principal phenomenon causing the breakdown is not related to the thermal losses associated with the dissipation factor, but it may be linked to different physics e.g., electromechanical, or electronic breakdown.

By way of explanation, the reported study proved that the performed accelerated aging did not significantly worsen the electrical properties of the material. This outcome may imply that in real applications e.g., power electronics, the polymeric material may deplete its mechanical performances at a faster

pace than its electrical ones during application life. Thus, it may still fulfill its role as insulation system as long as its mechanical properties are sufficiently good to ensure proper operating conditions.

For this reason, future work on this topic will include the analysis of the conduction current, mechanical properties, and electrical endurance tests on the same materials.

## REFERENCES

- [1] M. Kaufhold, H. Aninger, M. Berth, J. Speck, e M. Eberhardt, «Electrical stress and failure mechanism of the winding insulation in PWM-inverter-fed low-voltage induction motors», *IEEE Trans. Ind. Electron.*, vol. 47, fasc. 2, pp. 396–402, apr. 2000, doi: 10.1109/41.836355.].
- [2] A. Rumi, J. Marinelli and A. Cavallini, "Converter Stress Impact on Thermally Aged Resin for Low-Voltage Machines," *2021 IEEE Conference on Electrical Insulation and Dielectric Phenomena (CEIDP)*, Vancouver, BC, Canada, 2021, pp. 40-43, doi: 10.1109/CEIDP50766.2021.9705422.].
- [3] S. V. Suraci, D. Fabiani, S. Roland, and X. Colin, "Multi scale aging assessment of low-voltage cables subjected to radio-chemical aging: Towards an electrical diagnostic technique," *Polymer Testing*, p. 107352, Oct. 2021, doi: 10.1016/j.polymertesting.2021.107352.
- [4] M. C. Celina, E. Linde, and E. Martinez, "Carbonyl Identification and Quantification Uncertainties for Oxidative Polymer Degradation," *Polymer Degradation and Stability*, vol. 188, p. 109550, Jun. 2021, doi: 10.1016/j.polymdegradstab.2021.109550.
- [5] C. Rouillon et al., "Is carbonyl index a quantitative probe to monitor polypropylene photodegradation?," *Polymer Degradation and Stability*, vol. 128, pp. 200–208, Jun. 2016, doi: 10.1016/j.polymdegradstab.2015.12.011.
- [6] ASTM D149-20 - Standard Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies.
- [7] M. Da Cruz, L. Van Schoors, K. Benzarti, and X. Colin, "Thermo-oxidative degradation of additive free polyethylene. Part I. Analysis of chemical modifications at molecular and macromolecular scales," *J. Appl. Polym. Sci.*, vol. 133, no. 18, p. n/a-n/a, May 2016, doi: 10.1002/app.43287.
- [8] S. V. Suraci, X. Colin, and D. Fabiani, "Multiscale Modeling of Permittivity of Polymers With Aging: Analysis of Molecular Scale Properties and Their Impact on Electrical Permittivity," *IEEE Transactions on Dielectrics and Electrical Insulation*, vol. 29, no. 5, pp. 1795–1802, Oct. 2022, doi: 10.1109/TDEI.2022.3193869.
- [9] L.A. Dissado, J.C. Fothergill, "Electrical Degradation and Breakdown in Polymers (Materials, Circuits and Devices)", *The Institution of Engineering and Technology*, 1992.