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Article

Performance Analysis Based on Thermal Aging Tests of Sol-Gel and Polymer Insulated Wires by Enameling and Extrusion Technology

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Abstract: This research activity aims to evaluate electrical insulation system (EIS) intended for electrical machine winding wires. The evaluation is based on mechanical, thermal, and electrical properties tests following international standards for enameled wires. Dielectric parameters such as dissipation factor, partial discharge inception voltage (PDIV), parallel capacitance, and parallel resistance behavior of different insulator configurations on twisted-pair samples are observed during thermal aging tests. Those configurations are formed of different combinations of dielectric layers based on conventional polymers (polyester-imide (PEI), polyamide-imide (PAI), polyimide (PI)) used as coating wires. A study using two mineral varnishes (silica-based) obtained by the sol-gel process integrated on these classical enamels an outer layer of extrusion of thermoplastic polymer with and without mineral fillers. Given the high consumption of energy and the use of unsustainable materials involving the production of wires, the principal interest of this work is to exploit new configurations of coating wire produced, with less environmental impact than conventional ones. This work investigates the impact of filled resins on the performance of insulating samples and the influence of the use of sol-gel solutions (mineral varnishes) on the insulated wire to increase the thermal class.

Keywords: thermal aging; enameled wire; extrusion; sol-gel; thermoplastic polymer; mineral fillers; dielectric parameters



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1. Introduction

The enameled wires used for conventional windings are insulated with polymers and their application processes require toxic solvents and high energy consumption. Advances in insulation technologies and application processes are of ecological and economic interest [1]. Electrical machines are exposed to many stresses that contribute to the degradation of the electrical insulation systems (EIS), such as thermal stress [2]. In this context, the design of new winding insulation systems is guided by the interest in improving the performance of electric motors, by increasing the thermal class of insulated wires, that make up their coils and reducing the environmental impact caused by the enameled wire production.

Several standards define the insulated wires for machine coils, including the conductor diameter, the thickness of the insulating layer, and the various mechanical and thermal tests of the wires [3]. The thermal class of electrical insulators is classified by international standards [4]. This characteristic of wire insulation defines its performance. The material with

high thermal endurance can support electrical and mechanical constraints under higher temperature circumstances. This is interesting for electrical and electronic applications.

This work presents the development of extruded enameled wires based on a sequence of tests using different polymers and mineral varnish obtained by the sol-gel process on the enamel layer and different resins on the extrusion layer. The proposed wires are manufactured in a laboratory-scale enameling line present in LSEE. The new insulation structures tested are in development and offer a promising, more environmentally friendly solution for electrical insulation systems for winding wires. Their analyses are based on mechanical, thermal, and electrical test specifics for enameled wires. The resistance to the thermal aging test is the main factor of analysis to investigate the insulator material's ability to improve the thermal index of the enameled wire.

2. Literature Basis

2.1. High-Temperature Resistant Polymer Dielectric (HTPDs)

The polymer materials that possess enough thermal endurance for high-temperature operations (above 175 °C) are called high-temperature-resistant polymer dielectrics (HTPDs) [5]. For the HTPD used in electrical applications, the principal required characteristics are high thermal stability, high dielectric strength, and good adhesion to the copper conductor (in the case of enameled wires) [6]. They have those favorable properties because of their high-energy chemical bonds, strong inter- and intramolecular interactions, and conjugation action along the polymer chain [7]. The most common dielectric polymers used in electrical insulation systems for high-temperature applications are [5,8]

- polyimide (PI) films, varnish, plastics, and tapes;
- polyamide-imide (PAI) plastics and varnish;
- polyetheretherketone (PEEK) film;
- polyesterimide (PEI) film;
- polyphenylquinoxaline (PPQ) varnish; and
- polybenzimidazole (PBI) plastics.

Polymers imide enamels (PEI, PAI, PI) are used to insulate the winding wires of electrical machines. Their favorable properties for this application are high thermal and oxidative stability, high dielectric strength, and high mechanical strength [5,9].

- The polyimide (PI) varnish undergoes a chemical reaction during the coating process called imidization. The solvents are evaporated (by the annealing process as described in Section 3.1), and the precursor is dehydrated, resulting in the closure of the rings, and the PI coating [6]. The thermal class of this polymer is 220 °C and can reach 240 °C for 25 µm thickness.
- The polyamide-imide (PAI) combines the good properties of PI and good processability. The PAI varnish is characterized by a 220 °C thermal class and is frequently used on topcoats in electrical insulations of wires with a PEI underlay [5,9].
- The polyesterimide (PEI) is a modified version of PI, with lower thermal stability due to its flexible ether linkage [9]. This polymer is characterized by a 200 °C thermal class.

2.2. Sol-Gel Technology

Sol-gel is a process that allows the synthesis of materials from precursors in solution. The main application of this process is the application of thin films, which were initially used for the manufacture of low-temperature glasses. The exploitation of this process has opened space for the development of hybrid materials (nanocomposites) [10]. Materials derived from the sol-gel method can be produced from various substances such as silica, metals, organics, cellulose, and carbon [11].

Nanoparticles have an impact on the macrostructure that starts with the polarization generated at the atomic scale, which affects the morphologies of the material, followed by their electromagnetic interactions affecting their characteristic (complex parameter, complex permittivity, and many other dielectric parameters) [12].

Sol-gel is an attractive method to obtain the dispersion of nanoparticles and improve the properties of the enamel wire [13]. Interesting observations have been made on nanocomposites polymers containing different contents of silica particles obtained by the sol-gel process (from 0% to 20%) [14]. These observations include:

- The increase in thickness for the same amounts of coating applied to the wire using higher silica content. These effects were correlated with the increase of the viscosity of higher silica contents particles in the varnish compared with lower content, and neat polymer.
- The glass transition temperature is also increased by the high silica content coating.
- The improved surge-resistance properties if the nanoparticles are uniformly dispersed.

Previous works using the sol-gel process for coating wires have shown such good properties as breakdown voltage resistance with thin coats [15]. These properties have also been observed after thermal degradation at high temperatures [16]. Given these advantages, sol-gel solutions were chosen to be used in the insulation wire preparation with classical varnishes as a primer before the extrusion layer. This process is detailed in Section 3.1 below.

2.3. Extrusion Resins

Some studies have been carried out with extruded polymers for high-voltage wire; the interest of this technology is to improve the insulation performance between turns to avoid some of the other insulators that make up the motors by reducing their weight. Some materials such as polyetheretherketone (PEEK), polyphenylene sulfide (PPS), liquid crystalline polymer (LCP), and polytetrafluoroethylene (PTFE) are crystalline resins classified as being high-temperature resistant. Those works show that the extruded thermoplastic [17] high-temperature polymer with a base enamel insulator (PAI) had an improved performance compared with the conventional enameled wires combined with interphase paper insulators [18].

Additives in Polymers

Different additives (particles, fibers, platelets) are used with polymers to improve the properties of the material (processability, electrical, mechanical, thermal characteristics) depending on its application. For the processability properties, the most influential factor is the homogeneous distribution of the fillers on the material. It can be a powerful source of strategic property improvement, but for large-scale exploitation, there are many obstacles such as environmental reasons, high costs, and knowledge gaps [19].

Precedent works have shown an improvement in fracture behavior and other mechanical properties of insulations that contains mineral microfillers [20,21]. Part of the structure insulation of the magnet wires studied in this research activity are extruded thermoplastic resins. The presence of mineral microfillers is also tested to observe their impact on the insulation performance.

3. Materials and Methods

3.1. Conception of Insulated Wires

The process used to apply the electrical insulation to the copper wire includes the steps shown in Figure 1. The process starts by passing the bare copper wire through a first annealing furnace at 450 °C [22]. The temperature of this furnace is adjusted according to the wire speed. The wire is then cooled as it passes through a tank of demineralized water where its impurities are removed (the wire must be cooled quickly to avoid oxidation), and the water is blown away. During this phase, the copper is cleaned and softened, and its elasticity is improved by 40%. This step is important so that the wire can acquire the necessary flexibility to withstand the winding operations.

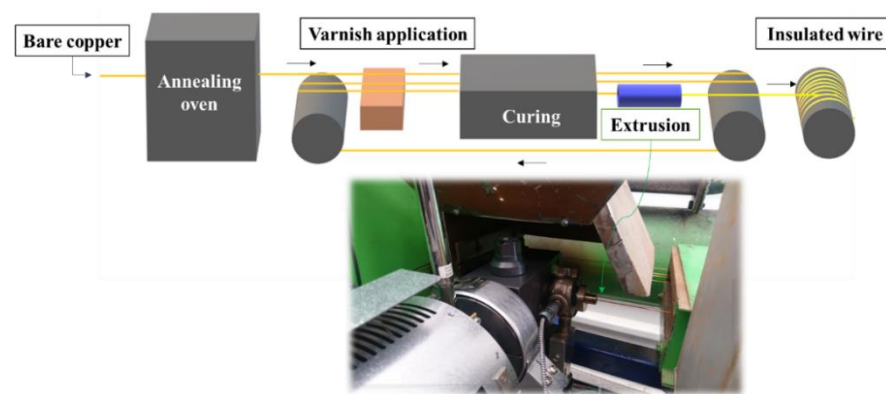


Figure 1. Insulation layer application process.

Next comes the varnish application phase, followed by an oven passage (curing process), where the solvents of the varnish will be evaporated, and the dry extract (the dried enamel) will remain on the wire surface. The coat application and curing process are repeated several times, to apply several layers of the enamel to the conductor. The evaporated solvents are passed through a catalyst where they are burnt and the heat from the catalyst is reused by the enamel baking oven.

Finally, the enameled wire arrives at the extruder, where a layer of resin is applied in a single pass.

The different systems for insulated wires used in this work are shown in Figure 2.

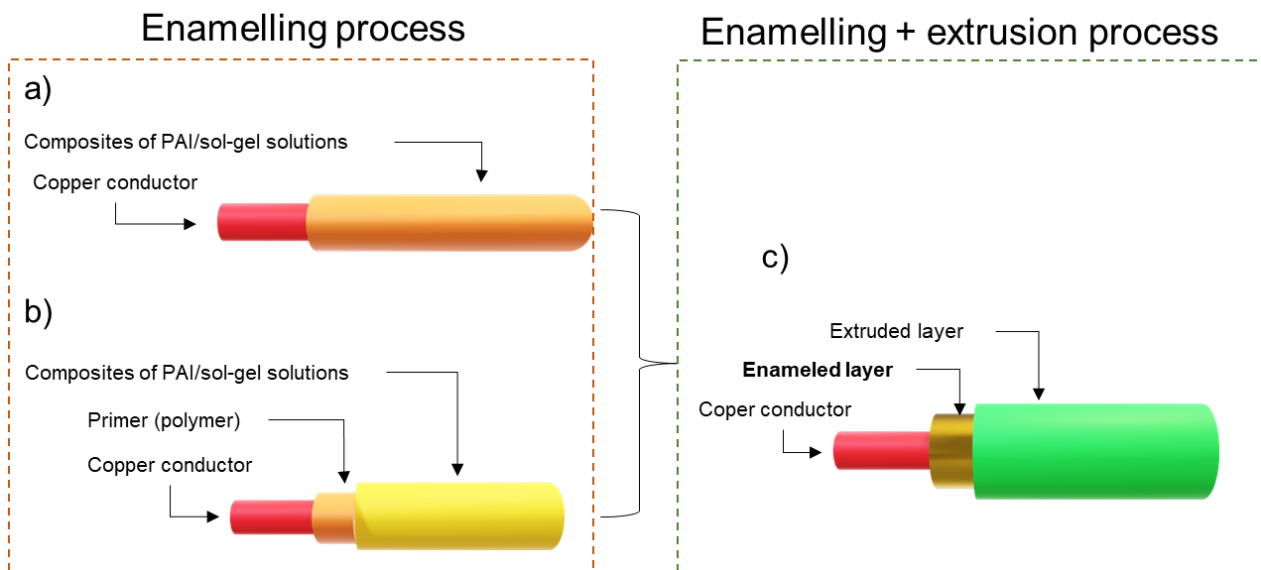


Figure 2. Wire insulation structures. (a) Enameled layer structure of composites of PAI/sol-gel solutions. (b) Enameled layer structure composed of a polymer primer and second layer composites of PAI/sol-gel solutions. (c) The global structure, enamel, and extrusion coat.

- (i) The enameling coatings with these primers were obtained with different varnishes in solvent (described in Section 3.2) including PEI, PAI, PI, and composites of PAI/sol-gel solutions (sol-1 and sol-2). The thickness of the enameled layers was in the range of 20–25 μm .
- (ii) Topcoat: thick layers obtained with extruded polymers and their composites (with fillers). The thickness of the extruded layer was in the range of 50–75 μm .

3.2. Materials Insulators

For the first part of the experiments, four types of insulating materials were applied to 0.95 mm diameter copper conductors. The temperature of the curing process used was 500 °C and the enameling speed was 20 m/min. The insulation systems consist of a primer (in solvent) applied by a classical enameling system followed by a topcoat extruded layer. The conventional polymers (used as a thick primer) used as the first coat are PEI and PAI. The solid content (dry extract) of used primers was fixed at 25%. The dilution varnishes were optimized to mainly ensure the adhesion of the extruded layer.

In a second step, the extruded coatings were applied to the primer including a pure thermoplastic polymer and the same thermoplastic polymer filled with 25% of mineral microfillers.

For Section 4.2, the extruded polymers (and its composites) layers were kept constant (as above), and the primers were varied by adding silica sol-gel solution (sol-2) to the PAI varnish, aiming to increase the thermal class of the insulation system and studying the effect of silica (from sol-gel solution) on the performances of our systems.

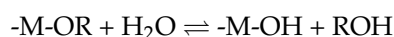
To compare the new system, we used sol-gel solution-PAI as a primer, with a classical high thermal class PI varnish; the latter was used as a primer and overcoated with both extruded layers.

For the last part, Section 4.2.1, the new primer with composites PAI-sol 1 was evaluated without extrusion layers, and the results are given for simple comparison and the work with the extruded layer needs more investigation.

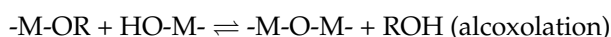
The sol-gel solutions sol-1 and sol-2 were used as received, without further modifications, from Materia Nova and Esix, respectively. Both sols were obtained by hydrolysis of Si metal alkoxide precursors under acidic conditions. The solid content of the sol-1 and sol-2 were measured at 260 °C after 1 h, giving values of 40% and 12%, respectively. The sol-2 contains an adhesion promotor reacting as a plasticizer to improve its mechanical properties.

The process of using sol-gel solutions is included in a mixture of silicon sol with PAI varnish as an enamel layer. In this process, we studied two different sol-gel solutions (sol-1 and sol-2) incorporated into the PAI varnish. Both sols were obtained by hydrolysis of Si metal alkoxide precursors by using tetraethoxysilane $\text{Si}(\text{R}_1\text{O})_4$ and $\text{R-Si}(\text{OR}_3)_3$, where R_1 and R_2 and R_3 are alky groups. The mechanism of the two main reactions during hydrolysis and polymerization is presented in the equations below.

- Hydrolysis:



- Condensation:



where M denotes the metal (such as Si, Ti, Zr, Al, etc.) atom and R the carbon chain (Methyl, Ethyl, etc.).

3.3. Analysis Method of Insulations Performance

After the conception of the wires, the basic properties are verified, and the thermal aging is initiated with additional dielectric measurements.

3.3.1. Characterization of the Insulated Wires

The first step is to validate whether the application of the new insulation complies with the requirements of the standards for enameled wire used in the windings of electrical machines [3]. The property verifications are described below.

- We examined the dielectric strength by measuring the breakdown voltage. The breakdown voltage measurement is made in five twisted-pair samples for each type of

insulated wire. The device illustrated in Figure 3a is following the standard IEC 60851-5 [23]. An alternative voltage (50 Hz) is applied to the sample, starting from 0 V, and increasing until the breakdown of the sample. The speed of voltage increase is uniform and defined by the standard depending on the amplitude voltage of the breakdown expected. When the breakdown occurs, the voltage is collected, and the test is made on the other samples. Figure 3a shows a photo of the local breakdown in a sample after the test.

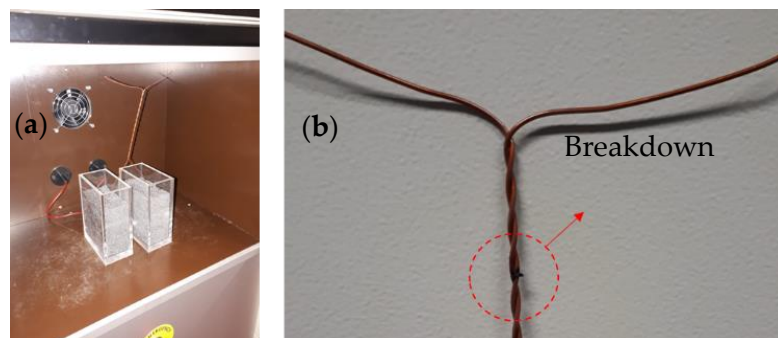


Figure 3. (a) Breakdown voltage device and (b) sample broken down.

- Mechanical properties using tensile stress, peel tests, and flexibility are performed on three samples per insulated wire. Figure 4 shows the flexibility and adhesion tests. Those tests are specified in the standard IEC 60851-3 [24].



Figure 4. Mechanical tests. (a) Flexibility tests samples. (b) Peel test device.

- The tensile stress or snap test consists of stretching the insulated wire until it breaks. The wire is observed and if it cracks or stripped parts are visible, the wire is considered to have failed the test (KO); otherwise, the sample is considered good (Ok). This test is applied to a wire of 25 cm in length; this allows us to check the quality of adhesion insulation to the copper.
- The “peel test” is used to check the adhesion of the insulation to the copper. The insulated wire of 50 cm in length is stretched under a 25 N load defined for the wires of 1 mm to 1.4 mm in diameter. The top and bottom parts of the insulation are removed. The insulated wire is rotated at a speed of 80 revolutions per minute, and the number of revolutions required to reach the loss of adhesion is counted.
- Flexibility is achieved by winding the wire around its diameter until at least 10 turns are made. The sample is classified as good if there are no cracks visible under the binoculars (zoom 6×). If the sample fails this test, more tests are made in a reduced constraint whereby the wire is wrapped around a support corresponding to two times its diameter or more.
- Thermal and thermomechanical properties were evaluated by a cut-through (thermo-plastic flow) test. This test was carried out based on the ASTM D1676—03 standard [25]. This test consists of measuring the temperature necessary to deform the material and allowing an electrical contact. Each sample is composed of two insulated wires crossed

and in contact. Once the test is running, a temperature is applied to the point of contact between the two round wires and increased until it reaches the cut-through temperature of the round film-insulated.

- The tangent delta characteristics as a function of the temperature of each wire are also collected based on the standards IEC 62631-1 [26] and ASTM D 150—98 [27]. The device dissipation factor measurement is composed of an oven, and the value of the dissipation factor is collected while the temperature of the oven is increasing, as presented in Figure 5. One sample for each insulated wire is measured. Before inserting the sample into the oven, a graphite solution is applied around the wire (the specific zone of contact with the measurement device) and dried.

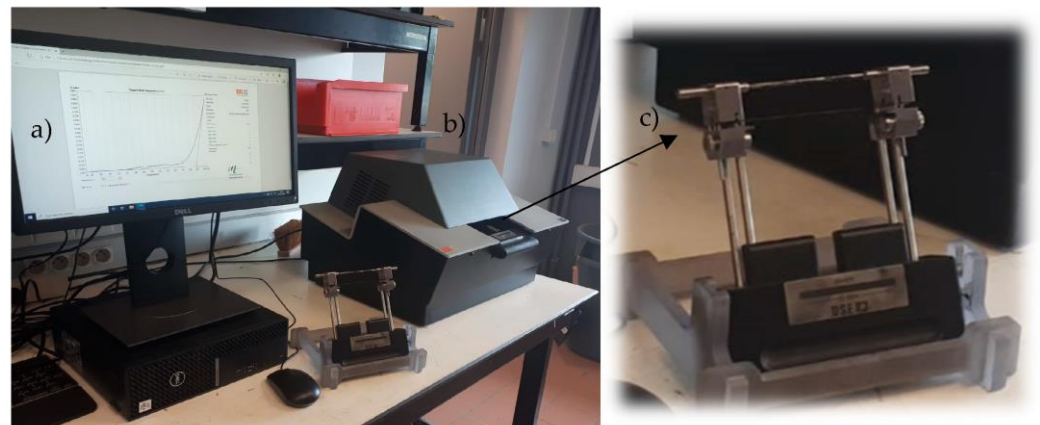


Figure 5. Dissipation factor measurement device: (a) Display of collected data; (b) Circulating air oven and, (c) sample with a graphite coat.

3.3.2. Thermal Aging Test

The thermal aging tests are executed following the international standards [28–32]. The main steps of the thermal aging test are described below.

- A group of 11 twisted specimens of each type of insulation is made. They are placed at insulated support inside an oven.
- A temperature and a duration of exposition are chosen.
- After each thermal cycle, a voltage is applied, and the amplitude is chosen following the thickness of the insulation.
- The lifetime is defined when more than 50% of the samples are broken down.

3.3.3. Dielectric Parameters Monitoring during the Thermal Degradation

The dielectric parameters are collected from the 11 specimens per wire insulation type before starting the thermal aging cycle to verify the homogeneity of the specimens:

- Partial discharge inception voltage (PDIV).

The PDIV is collected respecting the requirements of the standard IEC 60,270 [33]. The measurement device used in the laboratory is shown in Figure 6. The device is composed mainly of a 1 nF coupling capacitor (CD) with one output for DP measurement and another for voltage measurement. The quadrupole (CIL/V) contains a 1- μ F capacitor which, with the CD, forms a voltage divider (1000/1). The results in a low-voltage signal are proportional to the high voltage applied. This signal will be used to synchronize the partial discharge detector and visualize the waveform's quality. A preamplifier (RPAI) is used to amplify the signals of small amplitudes of partial discharges so that the phenomenon is visible during the collection of measurements. All these components are mounted in a Faraday cage as presented in Figure 6a, to overcome external electromagnetic interference. The compact ICM allows the measurement and display of the apparent charge pulses of partial discharges on the sinusoidal signal when a voltage level is imposed, as shown

in Figure 6b. Once the test specimen is connected, a voltage is injected and increased until repetitive pulses characterizing the partial discharges appear on the ICM Compact (the PDIV).

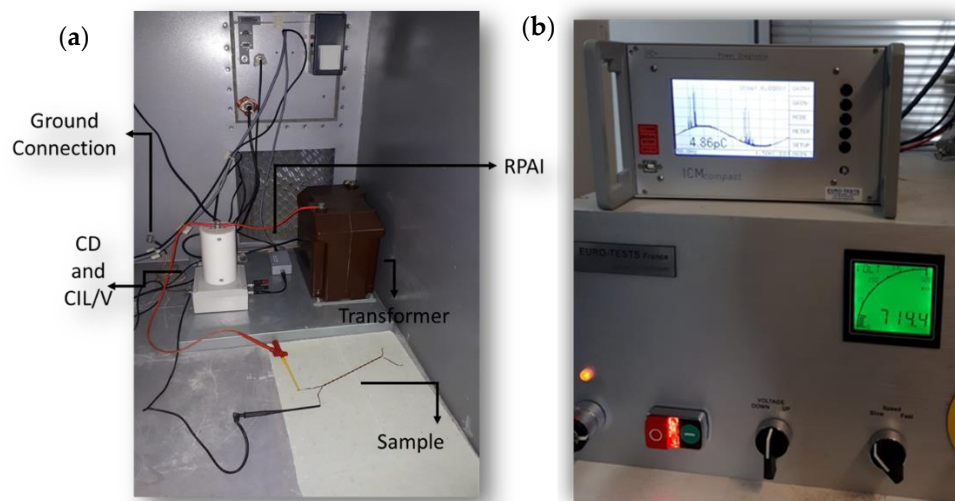


Figure 6. PDIV device. (a) Faraday cage. (b) ICM compact with the voltage control box.

- Impedance parameters (R_p , C_p , and $\tan \delta$):

The impedance parameters (R_p , C_p , and $\tan \delta$) are collected by using an Agilent 4294A Precision LCR meter based on the measurements methods defined by the standards IEC 62631-1 [26] and ASTM D 150—98 [27]. The frequency of measurement is selected as the accuracy indication by the catalog (10 kHz—0.08% of precision). The necessary corrections (open and short circuits) are made before the measurements to obtain the most representative values of the sample.

After each thermal cycle, the measurements are remade to monitor the evolution of those parameters during the thermal degradation. The main values of the C_p , $\tan \delta$, and PDIV parameters for the per insulated wire group of 11 samples are plotted to analyze these evolutions.

4. Results

This section presents the results of two experimental sequences. The first one is to investigate the influence of the thermoplastic polymer with mineral fillers on the extruded layer, and the second one is to observe the sol-gel solution influence on the enamel first coat insulation.

4.1. Polymers Enamelled with Thermoplastic Polymer Extrusion Layer

The four types of insulators and their results in basic property tests are described in Table 1.

Table 1. Basics characterizations of polymers enameled with thermoplastic on extrusion layer.

Samples	PAI + Thermoplastic Polymer	PAI + Filled Thermoplastic Polymer	PEI + Thermoplastic Polymer	PEI + Filled Thermoplastic Polymer
Enamel primer coat	PAI	PAI	PEI	PEI
Extrusion topcoat	Thermoplastic polymer	Thermoplastic polymer with 25% of mineral micro fillers	Thermoplastic polymer	Thermoplastic polymer with 25% of mineral micro fillers

Table 1. Cont.

Samples	PAI + Thermoplastic Polymer	PAI + Filled Thermoplastic Polymer	PEI + Thermoplastic Polymer	PEI + Filled Thermoplastic Polymer
Enamel/extrusion (μm) diameter over conductor	20/70	20/70	20/70	20/70
Snap test	OK	OK	OK	OK
Peel test	>240	200	>240	220
Flexibility 1D pre-stretch 20% length	OK	OK	OK	OK
Σ Breakdown voltage (kV)	7	7	6.8	6.1
Cut through (thermoplastic flow) ($^{\circ}\text{C}$)	>350	>350	>350	>350
Tangent delta temperatures ($^{\circ}\text{C}$)	101/240	103/241	104/227	101/246

With regard to the flexibility properties, all the structure insulators presented acceptable results. The wire was stretched to 20% of its length and turned around its diameter, and no broken parts were detected. For the first test of adhesion on cooper (snap test), the insulators showed no visible difference between them. After the snap test, it is observed the ruptured part of the wire, and the insulator didn't present any defaults.

Observing the peel test results, a higher rate of adhesion is obtained for the wires which are not filled, PAI + thermoplastic polymer and PEI + thermoplastic polymer. The presence of mineral microfillers could modify the interactions between the coats of insulation affecting the adhesion of the insulator. Previous studies show the effects of filler-matrix interactions in filled films when subjected to high constraints lead to the formation of cavities, resulting in stretchability reduced of composite films [34]. To accurately validate this relation between the presence of fillers and the adhesion of the insulator, more tests must be executed at the microscale also to deeply understand the changes in interactions between materials.

Observing the breakdown voltage mean values presented in Table 1, the insulator with filled resins shows lower results but is not significant if we consider the precision of measurement. The variations between samples of the same wire reach 0.45 kV, so the difference between wires cannot be observed from these tests; higher precision is needed.

Figure 7 refers to the curve of the sample of the wire PEI + thermoplastic polymer, the tangent delta temperature behaviour obtained is the same for the four types of wires. The first pic of loss near 100 $^{\circ}\text{C}$ represents the amorphous absorption part of the topcoat thermoplastic polymer (a semi-crystalline material). The last pic represents the absorption of the primer coat when the loss factor increases exponentially. The tangent delta temperature is frequently used as an indicator of the thermal class of the insulation system wire manufacturing [35]. The temperature values obtained don't show a significant difference between them. Seeing these similar values of tangent delta temperature in Table 1, a difference between the wire's thermal class is not expected.

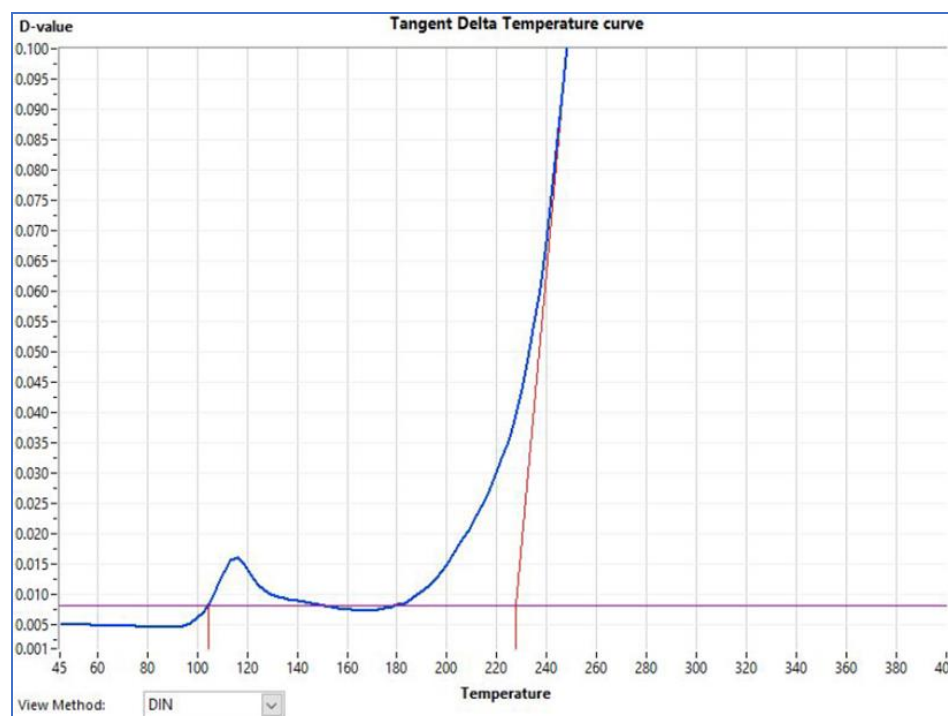


Figure 7. Tangent delta behavior for the wires PEI + thermoplastic polymer.

4.1.1. Thermal Aging Performance

Before starting the thermal aging test, a verification of the thickness of insulation wire with an optical microscope (Carl Zeiss—AXIO, Zeiss International Germany) had been performed as shown in Figure 8. The thickness that is shown in Table 1, 90 μm , is referent to the insulation diameter over the copper conductor. Thickness measurements were taken on all four sides around the copper conductor, with a 50 \times zoom. The eccentricity is an important property of the insulated wire [36]. The homogeneity of the insulation was checked (variations of two microns between measurements). The test voltage between cycles was chosen according to the IEC 60,172 standard [4], which is 1000 V.

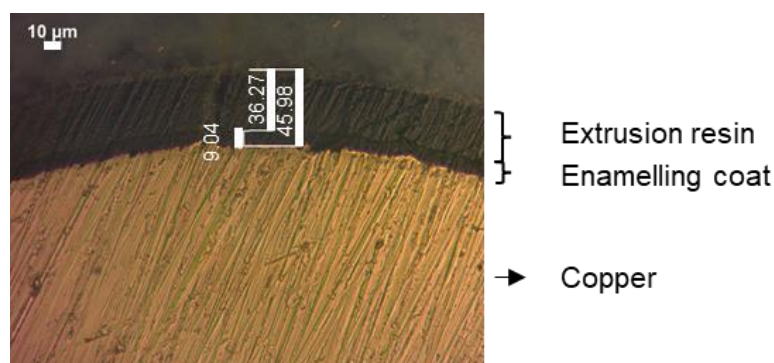


Figure 8. Microscopic view of insulated wire (dimensions in μm).

A temperature of 250 $^{\circ}\text{C}$ was chosen for the thermal aging cycles test, and it was considered that

- the thermal class for each insulation material is 190 $^{\circ}\text{C}$ for the thermoplastic polymer, 220 $^{\circ}\text{C}$ for PAI, and 200 $^{\circ}\text{C}$ for PEI; and
- the tangent delta curve, which presented a maximum temperature pic slightly below 250 $^{\circ}\text{C}$.

Regarding the thermal class of the primer coats, it's expected that the wires enameled with PAI would resist a longer period than the others. But in this experimentation, the

results show in Table 2 that the filled resin-extruded wires presented a bigger lifetime, consequently influencing a bigger thermal class. The best performing wire showed a significant difference in lifetime compared to the others.

Table 2. Lifetime at 250 °C aging test.

Samples	PAI + Thermoplastic Polymer	PAI + Filled Thermoplastic Polymer	PEI + Thermoplastic Polymer	PEI + Filled Thermoplastic Polymer
Lifetime (h)	420	708	348	492

4.1.2. Dielectric Parameters Collected between Cycles of Thermal Degradation

The dielectric parameters measured between thermal aging cycles (at constant and ambient temperature) are presented in Figure 9.

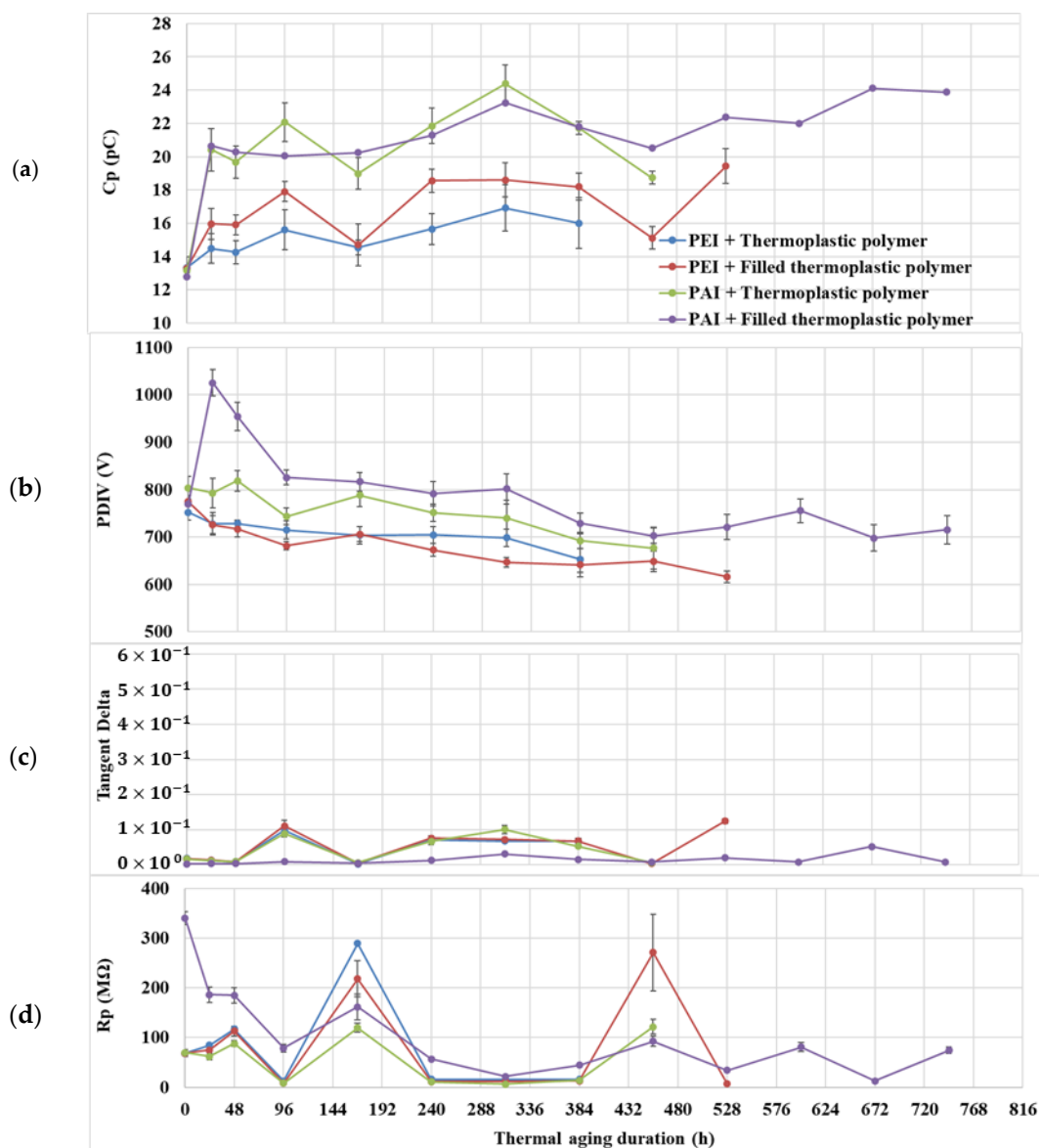


Figure 9. Mean values of dielectric parameters collected during the thermal aging test at 250 °C. (a) Parallel capacitance (Cp). (b) Partial discharge inception voltage (PDIV). (c) Dielectric loss factor (tangent delta). (d) Parallel resistance (Rp).

- The increase in parallel capacitance of the insulators composed by PEI in the primer enamel layer is lower than those composed by PAI in the primer enamel layer. This coincides with the difference between their respective relative permittivity, 3.15 [9] and 4.5 (at 1 kHz) [37]. The relative permittivity of PEI is lower than that of PAI, and this characteristic is seen after thermal aging.
- The wire PAI + filled thermoplastic polymer presents higher values of PDIV.
- The tangent delta of the PAI + filled thermoplastic polymer resin had lowered means values during this test.

The influence of the temperature on the parallel capacitance C_p of insulated wires needs more investigation, but in the literature [38–41] some works have been carried out and show an increased evolution of this parameter during thermal aging. The turn-to-turn capacitances collected in the samples grew up during the aging test, as expected.

For the partial discharge phenomena observed on twisted-pairs samples [42], some works [40,41] show that PDIV decreases during the thermal degradation of the insulation, as the results obtained in this experience.

The parallel resistance had performed inversely with the tangent delta value. That is coherent at low frequencies when the tangent delta is affected principally by the ohmic losses.

The dielectric parameters could give some indications during the first cycles of aging, with high values of PDIV and lower values of tangent delta for the wire enameled with PAI and extruded with the filled thermoplastic resin.

After the aging tests at 250 °C, the filled thermoplastic polymer had shown an improved aspect as presented in Figure 10. The PAI and PEI + thermoplastic polymer had shown a very damaged aspect of adhesion, which can be related to the degradation of polymers during the thermal aging caused by alterations in the molecular chains.

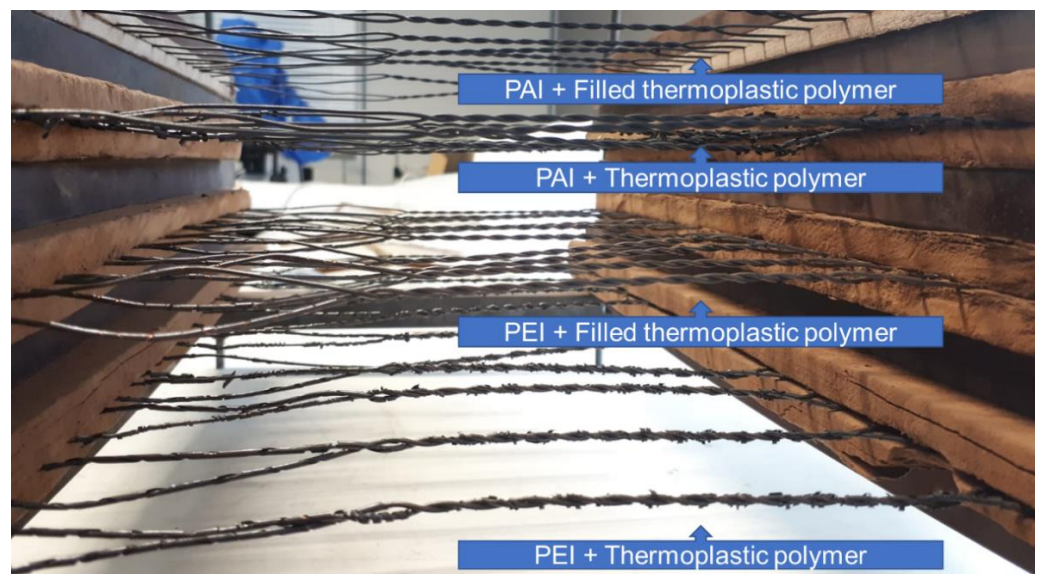


Figure 10. Improved aspect of filled thermoplastic polymer extruded layer after aging test at 250 °C.

4.2. Polymers with a Sol-Gel Solution in Enameled Coat under Thermoplastic Polymer Extruded Layer

To explore the varnish made from the sol-gel process as a primer coat under a thermoplastic extrusion resin, the structure insulations had been made as illustrated in Figure 2, Section 3.1. Preliminary tests have been performed with enameled insulated wires before selecting the structures to be analyzed under the extrusion layer.

4.2.1. Preliminary Analysis of Different Enameling Structures and Sol-1 Concentrations

The term “sol-1” is used to represent one of the varnish solutions made from the sol-gel process described in Section 3.2. Table 3 shows the different wire insulations tested at 330 °C and the lifetime (h) obtained. The parameters of change between them are

Table 3. Characterization tests of enameled wires using Sol-1.

Samples	PAI + PAI (80%)/ Sol-1 (20%)	PAI + PAI (50%)/ Sol-1 (50%)	PAI (50%)/Sol-1 (50%)	PAI (80%)/Sol-1 (20%)
Structure insulation	(b)	(b)	(a)	(a)
Primer polymer	PAI	PAI	-	-
Concentration (%)	PAI—80%/sol-1—20%	PAI—50%/sol-1—50%	PAI—50%/sol-1—50%	PAI—80%/sol-1—20%
Thickness layers diameter over conductor (μm)	5 μm/40 μm	5 μm/40 μm	45 μm	45 μm
Flexibility	4D	5D	3D	5D
Σ Breakdown voltage (kV)	7.3	7.6	9.6	7.9
Cut through (thermoplastic flow) (°C)	500	480	470	>520
Tangent delta temperatures (°C)	283	297	272	274
Lifetime (h) thermal aging at 330 °C	12	60	60	12

- the concentration of PAI/sol-1 varnish solutions enamel; and
- the structure of insulation (a) and (b) with the enameled coats, as described in Section 3.1.

The four types of wires have 0.5 mm in diameter and are manufactured under the same conditions of the enameling process, presented in Table 3.

Observing the results presented, we can note that the following.

- The structure insulation (b) with a pure polymer (PAI) in primer, presents a lightly lower breakdown voltage than structure (a). The insulation without PAI in primer present a higher content of sol-1; this may contribute to this property as shown in the literature in Section 2.2. Otherwise, the wires characterized by structure (b) present higher tangent delta temperatures.
- The higher concentration of the varnish made from the sol-gel process for the wires PAI + PAI (50%)/sol-1 (50%) and PAI (50%)/sol-1 (50%), can contribute to the thermal aging resistance as shown by their long lifetime.
- The higher concentration of PAI shows higher cut-through performance.
- All configurations containing the sol-1 had bad results in the flexibility tests. The absence of breaks could be seen when the constraint was reduced. Instead of one diameter, the samples only could resist when they were turned around at support of 3, 4, or 5 times the diameter size.

4.2.2. Enameled Wires Using Sol-2, PAI, and Extruded Layer

Other types of varnish made from the sol-gel process have been tested in lower concentrations to minimize the degradation of the mechanical properties of the insulation. In Table 4, we can see the different structures with the “sol-2” (the second varnish solution made from the sol-gel process as described in Section 3.2). The factors to be observed are the structure of insulation (a) and (b), the presence of fillers on resin extrusion, and the PI enamel coat in comparison with the PAI/sol-2 for the enamel extruded layer. Observing the wires composed with the insulation presented in the three first columns, we find the following.

Table 4. Basics characterizations of polymers with a varnish produced by a sol-gel process on the enameled layer with a thermoplastic polymer extrusion layer.

Samples	PAI + PAI/Sol-2 + Thermoplastic Polymer	PAI + PAI/Sol-2 + Filled Thermoplastic Polymer	PAI/Sol-2 + Thermoplastic Polymer	PI + Thermoplastic Polymer
Structure insulation enamel	(b)	(b)	(a)	-
Enamel primer coat	PAI	PAI	PAI/sol-2 (18%)/PAI (82%)	PI
Enamel intermediary coat	PAI/sol-2 (18%)/PAI (82%)	PAI/sol-2 (18%)/PAI (82%)	-	-
Extrusion topcoat	Thermoplastic polymer	Filled thermoplastic polymer	PPS	PPS
Thickness layers over a conductor (μm)	5/20/55	5/20/55	25/75	25/75
Snap test	OK	OK	KO	OK
Peel test	180	60	KO (>60)	120
Flexibility 1D	OK at 20% elongation	OK at 20% elongation	KO	OK at 20% elongation
Σ Breakdown voltage (kV)	11.4	10.1	7.3	5.9
Tangent delta temperatures ($^{\circ}\text{C}$)	105/248	110/260	100/259	102/260

- The snap test results, peel test, flexibility, and dielectric strength, show a low performance for the wire PAI/sol-2 + thermoplastic polymer which is composed of a structure (a). In this experience, this structure shows less performant mechanical properties (adhesion and flexibility) which could affect the breakdown voltage of the insulator.

Comparing the wires which are composed of a polymer on the interface with the cooper conductor and PPS on the extrusion layer, we find the following.

- The PI + Thermoplastic polymer is characterized by a lower breakdown voltage but has good mechanical properties.
- The PAI + PAI/sol-2 + filled thermoplastic polymer shows a lower performance on the peel test compared with the same non-filled insulated structure. Otherwise, the PAI + PAI/sol-2 + thermoplastic polymer without fillers has a slightly lower temperature of the tangent delta.
- The tangent delta temperatures don't show an important difference between the wires.
- The less performant wire on these characterizations is PAI/sol-2 + thermoplastic polymer, which means that the polymer in primer can be a factor in the contribution of the performance insulation for the extruded enameled wires.

4.2.3. Thermal Aging Performance

The temperatures of the tangent delta are around 260°C for the majority part of those wires; the chosen temperature of the test is 300°C . A group of samples of the wire PAI + filled thermoplastic polymer which presented a longer lifetime on the precedent aging test had been included in this test, in addition to the wires presented in the first columns of Table 4. The lifetime obtained is shown in Table 5.

Table 5. Lifetime at 300°C aging test.

Samples	PAI + PAI/Sol-2 + Thermoplastic Polymer	PAI + PAI/Sol-2 + Filled Thermoplastic Polymer	PAI/Sol-2 + Thermoplastic Polymer	PI + Thermoplastic Polymer	PAI + Filled Thermoplastic Polymer
Lifetime at 300°C (h)	168	168	24	168	72

The short lifetime obtained by the wire PAI/sol-2 + thermoplastic polymer is coherent with its lower performance on the mechanical tests. No difference between filled and non-filled resins had been noted in this aging test result.

The sol-gel solution-PAI structure presented longer lifetimes than the PAI + filled thermoplastic polymer, which means that

- the varnish made from the sol-gel process in the enamel insulator may strengthen the lifetime insulation and consequently the thermal class. It's a possibility of insulation having less polymer, and better performance compared with entire polymer insulation. The mineral part generated by sol-2 in the PAI enamel leads to an increase in wear and thermal resistance during the aging tests. These observations agree with the literature.

4.2.4. Dielectric Parameters Collect between Cycles of Thermal Degradation

Observing the dielectric parameters between cycles (at constant and ambient temperature) during the thermal aging tests for the wires which resist longer time the thermal aging test (Figure 11). The following can be noted.

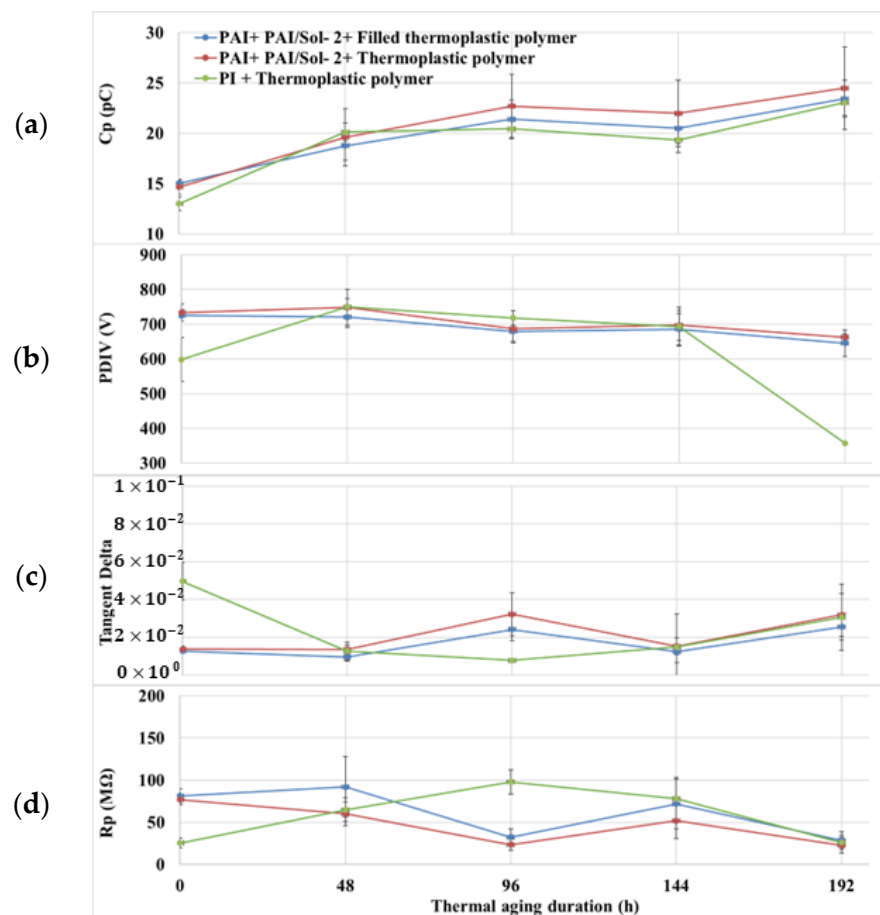


Figure 11. Mean values of dielectric parameters collected during the thermal aging test at 300 °C: (a) Parallel capacitance (Cp). (b) Partial discharge inception voltage (PDIV). (c) Dielectric loss factor (tangent delta). (d) Parallel resistance (Rp).

- The evolution of the parallel capacitance is increasing as the previews experience analysis. The difference between wires is not very significant.
- The PDIV has an increasing evolution for all groups of samples. The PI+ thermoplastic polymer extruded layer has a bigger augmentation after the first cycle, which can be explained by a resumption of polymerization if it was not finished during the enamel process. This phenomenon is also shown in the PDIV curves of Figure 9 referent of the 250 °C thermal aging.
- The tangent delta values don't show any different behavior that can be related to the end of life. This parameter variates inversely to parallel resistance as in the preceding experiment.
- The evolution parameters are similar for the wires PAI + PAI/sol-2 + filled thermoplastic polymer and PAI + PAI/sol 2 + thermoplastic polymer, which contains the

same material of enamel. That may indicate the parameters are more influenced by the interface of contact copper and enamel, rather than extrusion topcoats.

- The fillers on the extrusion layer didn't show any impact on this aging test as the precedent test.

5. Discussion

The first part of the experimentation which analyses the extrusion layer with thermoplastic polymers and mineral fillers had been in coherence with the literature consulted. Two references showed improvement in mechanical properties:

- The presence of 25% of mineral fillers showed an increase in crystallinity of the material in ref. [21] and, it had been used as a solution to improve the toughness of the polymer in ref. [20].

The microfillers affected the lifetime of extruded resins without affecting their tangent delta temperatures. The tangent delta temperature is not enough to predict a performance aging test for extruded enameled wires with the presence of mineral microfillers.

Concerning the lifetimes obtained by the thermal aging test, the extruded wires' lifetimes exceed the commercial ones, which have a thermal index of 210 °C. Commercial wires enameled with PEI and PAI coat had also aged at 250 °C as shown by previous studies [43]. The lifetime obtained for these commercial wires was 252 h, inferior to the lifetimes obtained for all the enameled extruded wires in this work.

The dielectric parameters could give some indications about the performance of the wires. In the first cycles of aging degradation, high values of PDIV and lower values of the tangent delta are collected for samples of PAI + filled thermoplastic polymer insulated wire.

In the second Section of experimentation Section 4.2, it could be observed the good performance of the insulations with a bigger concentration of varnish made from the sol-gel process, (except in terms of mechanical properties for non-extruded wires). This can be correlated with the good dispersion of silica for the sol-1 (to be confirmed with the supply varnish). This influence is not noted for the followed tests with the sol-2 which presented better performance when composed by a PAI in primer.

For the extruded insulations, after one cycle of thermal aging, the augmentation of PDIV could be observed, which indicates some changes in the material, and possible finalization of polymerization. This hypothesis can be confirmed by deeper material analysis as a differential scanning calorimetry analysis. That means the curing process must be eventually optimized to improve the conditions of polymerization during the varnish application process.

For both experimentations, the following could be observed.

- The coherence of the literature and the variation of parallel capacitance and PDIV during the aging test.
- The measurements of parallel resistance presented an inverse evolution of tangent delta ($\tan \delta$) This is very close to the sample measurement model represented by a capacitor in parallel with a resistance possessing the following Equation,

$$\tan \delta = \frac{1}{\omega C_p R_p}$$

where, ω = the angular frequency (rad/s), C_p = parallel capacity (C), and R_p = parallel resistance (Ω).

During thermal aging at temperatures above 200 °C, copper can suffer from oxidation. This phenomenon causes overheating in the area where it occurs, the excessive heating causes oxidation to increase at a faster rate. It is known that lifetime is strongly impacted by oxidation [44]. Complementary analyses on wires after aging must be done to deeper understand how the results obtained by the thermal aging could be influenced by copper oxidation.

6. Conclusions

In this work, we have demonstrated a great interest in the use of a new process combining classical varnishes, sol-gel solutions and free solvent topcoat using extruded polymers. We have studied three different systems of insulated wires and new processing. The samples were prepared and characterized, and the full results were discussed.

The observation of the dielectric parameters during thermal aging can give a preview of the performance of the wires. These parameters can help to make decisions for the conception of wires before the end of the aging test.

The improved performance during the thermal aging degradation is evident for the enameled wire, which presents higher contents of the silica sol (sol-1). This demonstrated that this technology can be a strategic source of improvement for HTTD insulators, although the flexibility needs to be improved. For this purpose, we believe that the extrusion part as top layers could be an appropriate solution for the complete system.

The mineral microfillers show an evident improvement for the extruded thermoplastic polymer resin during thermal aging, but can have a similar performance to a non-filled resin depending on the enamel coat underneath. A deeper analysis at the molecular scale may answer the reasons for this different behavior and explain the interaction between the top extruded and the underneath coat.

The wires are made in a laboratory, and better process conditions can be studied to exploit the technologies of the sol-gel process for the enamel layer and extrusion resin in the topcoat. The combination of enamel produced by the sol-gel process using silica sol and the extruded thermoplastic polymer is a promising option for the development of eco-friendly magnet wires. The enameled extruded wires developed and studied in this work demonstrated an improved thermal aging resistance compared to conventional enameled ones.

More investigations are being made to analyze the copper oxidation impacts during the thermal aging tests, and to choose materials that can reduce this effect.

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