Correlations between Physico-Chemical Properties and Dielectric Behavior of Thermally aged XLPE Cables Insulation

Yasmina Sebbane, Ahmed Boubakeur, and Abdelouahab Mekhaldi

Abstract-The carried out investigations aim at clarifying the correlations between physico-chemical properties and the variation of dielectric losses of Cross-Linked Polyethylene (XLPE) as Medium Voltage cable insulation during thermal aging. The used cables were manufactured by ELSEWEDY CABLES. They have been installed in Algeria where they must resist high temperatures that could exceed 50 degrees in the south of the country. For this aim, thermal aging measurements were performed on XLPE cables samples at 80, 100 and 140 °C for aging time of 5000 hours. The chemical properties were evaluated through Fourier Transform Infrared Spectrum (FTIR). In addition, the effect of aging on the crystallinity ratio, the lamellar thickness and the melting peak temperature was assessed using Differential Scanning Calorimetry (DSC). Frequency domain dielectric spectroscopy has been conducted on the aged XLPE cables in order to study the evolution of the dielectric losses. The obtained results showed that the principal aging mechanism is thermal oxidation which heads to deteriorate the morphology and increase both the polarization and losses, principally at low frequencies. It has been found that the color changes caused by the chains scission indicate the increase of interfaces between the cable insulating and semi-conductive layers, which could head to increase the dielectric losses due to the interfacial polarization.

Keywords- Cross-linked Polyethylene, Cristallinity, Thermal aging, Dielectric losses.

I. INTRODUCTION

The Cross-linked polyethylene (XLPE) is used as an electrical insulating material in underground and transmission and distribution cables, due to its prime dielectric strength, low dielectric permittivity and losses factor [1]. Therefore, crosslinked polyethylene (XLPE) remains a material of choice for medium and high-voltage cables [2]. It has also a high operating temperature of about 90 °C [3]. However, it is now understood that irreversible changes may happen after the exposure of the cable to severe environmental conditions. The thermal constraint considered as the most impacting factors leading to irreversible chemical and/or morphological changes that affect the properties of XLPE, and produce a limit to the effective service life of the power cables [4]. To address these issues, many researchers have focused on the relationship between structural changes and dielectric strength of cables insulation under thermal oxidative aging [5, 6]. These studies showed that the dielectric parameters (dielectric losses factor and dielectric permittivity) are completely linked with the physical, chemical and structural changes of XLPE insulation occurring during service or accelerated testing [7]. However, further research is needed on the dielectric behavior of the aged cables.

This paper aims to evaluate the Physico-chemical and dielectric properties of the cable insulation samples before and after aging to investigate the degree of XLPE degradation. Besides, we aim

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to study the correlations between these different properties, leads to elucidate aging and degradation mechanisms.

II. SETUP OF EXPERIMENTS

A. Samples preparation and thermal aging procedure

Taking into account the dimensions of the ovens intended for the thermal aging, it was therefore necessary to prepare cables samples into 0.5 m long segments. Fig 1 shows the dimensions of the tested XLPE cable samples.



Fig 1: Dimensions of the tested XLPE cable samples.

Samples of 18 kV XLPE cable furnished by Elsewedy Cable were cut into 0.5 m in length taken as specimens for thermal aging tests. The samples consist of XLPE insulation (thickness~8mm).

The choice of the test temperatures derives from cable design and the need to obtain thermal degradation in a rational time. The maximum temperature assigned to conductive core of the cable in steady conditions is 90 °C. Also, higher temperatures are allowed in Short-circuit conditions (up to 250 °C) according to the cable technical sheet. All cable samples were aged in an air oven at three of aging temperature 80°C, 100°C and 140°C. Aging time of 5000 hours was selected for two level of temperatures 80°C and 100°C.While 1500 hours were used for 140°C.

B. Analytical measurement

B.1. FTIR spectroscopy measurements

The chemical properties brought about in XLPE insulation after the degradation generated by thermal aging were analyzed by a spectrometer (FTIR spectrum two 100302) with PerkinElmer software.

The XLPE films for FTIR analysis were cut up longitudinally on a slitting machine. In this method, the insulation layer of the cable was sliced into thin wedges to form a loop, as shown in Fig 2.



Fig 2: Cutting of cable insulation.

B.2 .DSC measurement

DSC analysis was performed using PERKIN ELMER PYRIS device under nitrogen gas flow.

B.3. SEM analysis

We analyze the surface morphology of aged and unaged XLPE samples by Scanning Electron Microscopy (SEM) using SU-8230 Electron Microscope.

To obtain smooth surfaces, we immerse the aged and unaged samples in a liquid Nitrogen bath at a temperature of -30°C, and then we cut them using a LeicRM2265 microtome.

B.4. Dielectric measurement

Two sets of devices were put for the characterization of the cables samples.

B.4.1. Dielectric spectrometer

The dielectric measurement (dielectric losses factor tanð) was performed with the Broadband Dielectric Spectroscopy (BDS) using a spectrometer Type Novocontrol (high resolution Alpha-N dielectric analyzer) with WinDETA software. The cable samples were covered with copper tape in order to increase the surface of the electrode and reduce the connection resistance; then, the measurement was started after calibrating the device at low capacity and the measurement frequency range from 100 Hz to 1 MHz

B.4.2. Schering Bridge

The dielectric losses factor (tan δ) was measured in function of that at the temperatures higher than the melting point of XLPE the voltage by using a Schering bridge (AG type TG-3MOD) (105 °C) ease the oxygen diffusion between the under the ambient temperature and the frequency of 50 Hz. A test cell consisting to place the conductive core on the HV oxidative reactions.

terminal of the transformer and the metal screen to earth, then gradually increase the test voltage up to the specific cable voltage, which is 18 kV. A filter containing an inductor and two parallel capacitors was used to reduce background noises and a layer of semi insulating paint was applied through the surface of the cable ends to reduce the leakage currents.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Color changes

In Fig 3, we present the obtained changes in aspect of XLPE films from cutting of cable at different aging phases. We remark that the color changed from gray before aging to light yellow after 5000 h at 80 °C, to light brown after 5000 h at 100 °C and then a dark brown after 1500 h at 140 °C.



Fig 3: Color change of XLPE films.

These color changes have been ascribed to the formation of vinylidenes and vinyl groups caused by thermal oxidation [8, 11]. It could attribute to the antioxidant reaction during the oxidation [12].

B. FTIR spectroscopy analysis

In Fig 4, we present the FTIR spectra of XLPE samples before and after thermal aging.

The group CH_2 manifest at a wavenumber of 720 cm⁻¹, and it is affirmed by its peaks appeared at 730 cm⁻¹, 1463 cm⁻¹, 2848 cm⁻¹ and 2915 c [13, 14].

After aging, an increase of the absorption peaks intensities in the XLPE characteristic bands (720, 1463, 2848 and 2915 cm^{-1}), could be attribute to the molecular chains scission. At the aging temperature of 140 °C we remark that the formation of C=O carbonyl groups and an increase of the IR absorption band between 1500 cm⁻¹ and 1800 cm⁻¹.In addition, the C=C bond of the vinylene group responsible for the color change can be found between 1560 cm⁻¹ and 1600 cm⁻¹ [15]. Moreover, a slight increase in the absorption of the -OH function of the hydroxyl groups could be observed after aging in the region between 3000 and 3500 cm^{-1} [16]. The significant increase of the carbonyl groups observed in the case of the aging temperature of 140°C. It is known in the literature that at the temperatures higher than the melting point of XLPE (105 °C) ease the oxygen diffusion between the oxidative reactions.

The acceleration of chemical reactions after thermal aging, the aging rate can be supposed proportional to the rate of chemical reactions. It can easily be anticipate that the thermo-oxidation phenomenon in the thermally aged XLPE cables may influence their dielectric behavior.



Fig 4: FTIR spectrum of XLPE samples plotted in different ranges after 5000 hours for the aging temperatures of 80° C, 100° C and after 1500 hours for the aging temperature of 140° C.

Enlargements of various partial spectral ranges of Fig 4 are presented in Fig 5, 6 and 7.



Fig 5: Partial FTIR spectrum from 400 to 1500 cm^{-1} .





Fig 7: Partial FTIR spectrum from 1600 to 3600 cm^{-1} .

C. SEM analysis

Fig 8 show the micrographs of unaged and aged XLPE samples at aging temperatures of 80°C, 100°C and 140°C, using Energy Dispersive Spectroscopy technique.

The SEM micrographs show many micro-cracks on the XLPE surface after thermal aging. These micro-cracks are more accentuated when the aging temperature increases; therefore, the unaged XLPE has a smooth surface. We also notice that these cracks take the form of needles which appear on the surface of the thermo-oxidized material at all aging temperatures 80°C, 100°C and 140°C [17].



Fig 8: SEM-EDS micrographs of the samples.(A) NA, (B) Aged at 80°C for 5000 h ,(C) Aged at 100°C for 5000 h ,(A) Aged at 1400°C for 1500 h.

D. DSC Results

The thermograms of the heating and cooling cycles of unaged and aged samples are presented in fig 9, 10 and 11.

The critical parameters are listed in Tables 1 and 2, where T_C is the crystallizing peak temperature, T_m is the melting peak temperature, and χ (%) is the crystallinity ΔH_m is the melting enthalpy and ΔH_c is the crystallization enthalpy.

The lamellar thickness was calculated based on Thompson-Gibbs equation as follows [13]:

$$T_{m} = T_{m0} \begin{pmatrix} 2\sigma_{e} \\ \Delta H_{m} \cdot L \end{pmatrix}$$
(1)

Where L is the lamellar thickness; σ_e is the surface-free energy per unit area of basal face; ΔH_m is the melting enthalpy of an ideal polyethylene crystal per unit volume T_{m0} is the equilibrium melting temperature of an infinitely thick crystal. The values used for calculation were as follows [13]: $\Delta H_m = 2.88 \times 10^8 \text{J}. m^{-3}$ $T_{m0} = 414.6$ K, and $\sigma_e = 93 \times 10^{-3} \text{J}.m^{-2}$.



Fig 9: DSC heating thermograms of unaged and aged samples (first heating cycle).



Fig 10: DSC heating thermograms of unaged and aged samples (second heating cycle).



Fig 11: DSC heating thermograms of unaged and aged samples (first cooling cycle).

 Table I

 RESULTS OBTAINED FROM DSC MEASUREMENTS (First cycle)

Parameters	NA	80°C-5000 h	100°C-5000 h	140°C-
				1500 h
T _m (°C)	106.26	111,36	109.04	63.99
T _c (°C)	94.59	92.65	92.08	53.48
$\Delta H_m(J. g^{-1})$	95.42	94,29	56.45	41.98
$\Delta H_c(J, g^{-1})$	-108	-92.73	-57.9	-42.66
X (%)	33.21	32,81	20.00	17.04
L (mm)	70,60	69,60	70.00	41.83

 Table II

 RESULTS OBTAINED FROM DSC MEASUREMENTS (Second cycle)

Parameters	NA	80°C-5000 h	100°C-5000 h	140°C-
				1500 h
T _m (°C)	105.93	110.57	107.00	78,77
T _c (°C)	94.58	92.66	92.06	52.99
$\Delta H_m(J, g^{-1})$	86.41	86.80	40,50	27.16
$\Delta H_c(J, g^{-1})$	-62.70	-92.63	-55.36	-42.44
X (%)	30.07	30.21	14.00	9.40

We remark in (Fig 9) that the melting peak widens and shifts toward lower temperatures. This phenomenon is ascribed to the chain scission caused by the oxidation process at high temperatures [13].

We remark in (Fig 10) that the ridges appeared during the first heat cycle disappear.

For the cooling cycle (Fig 11), we remark that at the aging temperature of 140°C the depression in crystallization temperature suggests that the cable sample underwent severe deterioration, due to the chain scission [13].

We can distinguish two phase according to the aging temperatures. The first one matches to aging temperatures below the melting point (80 and 100 °C) where the oxidation mainly affects the amorphous regions. However, oligomers could seem in the amorphous regions due to the chain scission. These latter crystallize. This is named as a post-cross-linking phenomenon [18]. The second phase matches to the higher aging temperatures (140 °C), the oxygen diffusion is accelerated and the crystalline parts disappear [18].

E. Dielectric behavior

To study the correlation between the dielectric behavior of the thermally aged XLPE cables and the physico-chemical changes, the samples aged at 80°C, 100°C and 140°C were taken to measure their dielectric losses factor, in the frequency range from 10 Hz to 300 kHz and in the voltage from 5kV to 18kV.

Fig 12 presents the plot of tan δ versus frequency for the unaged and aged XLPE cable samples of thermal aging at 80°C.100°C and 140 °C in the range from 10 Hz to 300 kHz.



Fig 12 : Plot of tan δ versus frequency for the unaged and aged XLPE cable specimens at 80°C.100°C and 140 °C in the range from 10 Hz to 300 kHz.

Enlargements of various partial plot frequencies ranges of Fig 12 are presented in Fig 13, 14 and 15.



Fig 13: Tan delta versus frequency in the range from 10 Hz to 500 Hz.



Fig 14: Tan delta versus frequency in the range from 500 Hz to10 kHz.



Fig 15: Tan delta versus frequency in the range from 10 kHz to 300 kHz.

We remark from the Fig 12 that dielectric losses factor of the thermally aged cable samples in the frequency ranging from 500 Hz to 10 kHz becomes higher with the aging temperatures and the fast decrease of tan δ in the frequency range between 100 kHz and 300 kHz.

We observe in Fig 13 that a dielectric losses peak appears in the frequency domain from 10 Hz to 500 Hz. As the aging temperatures becomes higher, the dielectric losses peak make higher and its position shifts to the higher frequency region.

The higher peaks of the dielectric losses factor of the cable samples aged at 100°C for 5000 hours and 140°C for 1500 hours can be due to its carbonyl groups confirmed by FTIR results and the displacement of the dielectric losses peak towards high frequencies (as indicated by arrow Fig 13) could be attributed to the morphology of the material (Fig 8) [19]. The change of the XLPE color seems to indicate the effect of increasing the interfacial zones between XLPE insulation and semi conductive layers and then promoting the interfacial polarization.

According to the reference [20] the presence of relaxation peak reveals the existence of several absorption bands. The dielectric losses peak in the frequency domain from 10 Hz to 500 Hz may be related on the one hand, to the presence of the interfaces (interfacial polarization) between XLPE insulation and semi conductive layers, on the other hand, to the thermo oxidation.

Mecheri and *al* [21] showed that the rapid decrease in the dielectric losses factor can be probably caused by the improvement of the crystal morphology of the XLPE and the volatilization of the crosslinking. For example, as confirmed in our DSC results, the thickness of the crystalline lamella remains practically the same compared to that of the sections of not aged cables (for the aging temperature of 80°C the thickness is 69.6 mm and for not aged cable it is 70.6 mm).

It is known, tand is strongly influenced by the oxidation phenomenon in the case of Polyethylene [22]. Therefore, the tand depends on the losses polarization such as dipole polarization [23]. The increase of tand can be attributed to the formation of electrically asymmetric carbonyl compounds due to the oxidation, these later are the mainly factor in increasing of dielectric losses [24]. This dielectric behavior may be ascribed to the presence of the carbonyl groups confirmed by FTIR analysis and the spatial charges due to the thermo-oxidation in the XLPE insulation [7]. The increase in dielectric losses factor for a not aged sample cable in overall frequency range is due to the ohmic effect.

Fig16 presents plot of tan δ versus voltage levels for the unaged and aged XLPE cable specimens at thermal aging of 80°C, 100°C and 140°C in the range from 5kV to 18 kV.



Fig 16: Plot of $\tan \delta$ versus voltage levels for the unaged and aged XLPE cable specimens at 80°C, 100 °C and 140°C in the range from 5kV to 18 kV.

We remark in Fig 16 that the tand increases with respect to voltage levels and this increase is accentuated when the aging temperature increases.

The increase is attributed to the losses caused by partial discharges occurring in the material [24]. The tan δ increase can be attributed to the great mobility of charge carriers. According to [25], thermo-oxidation plays a predominant role by the formation of polar groups and the chain scission lead to a decrease in the viscosity of the polymer and then, polar groups promote the mobility of charge carriers.

Tan δ increases considerably from a voltage level of 15kV which would be the ionization threshold, at beyond which partial discharges arise in the material yielding significant dielectric losses [26].

IV. CONCLUSION

In this paper, we have investigated the evolution of thermal, chemical, morphological and dielectric behaviors of XLPE MV cables insulation under thermal aging. The obtained results led to the main conclusions listed as below:

• The experimental results suggest that dielectric losses factor is strongly responsive to thermal aging effects and that it [16] can be contemplated as a diagnostic parameter for guessing the degree of thermally aged XLPE cables deterioration,

• FTIR characterization results show that the chemical degradation is accompanied by the generation of polar groups like that carbonyl and hydroxyl groups,

• The color change of XLPE to dark brown may throw back the beginning of the material deterioration. This change is ascribed to the formation of vinylidene and vinyl groups,

• The low frequencies range seems more adequate for ^[19] assessing the advanced degradation of XLPE cables samples,

• At low frequencies, the interfacial polarization due to the space charge accumulated between the XLPE insulation and the semi-conductive layer is the origin of dielectric losses,

• The color changes that show an increase of interfaces in the material could conduct to an increase of the dielectric losses,

• Micrographs obtained by the SEM analysis show that the deterioration of the dielectric properties is directly related to the thermo oxidation phenomenon,

• DSC results show that the recrystallization of many parts of XLPE leads to enhance the thermal and dielectric properties for the aging temperature of 80°C.

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