# A Supportive Approach into Life Testing and Characterization of PVC and XLPE-Insulated Cable Materials

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Abstract: ESR thermal analysis investigation of widely used insulation materials, namely PVC and XLPE, were conducted. The results show the thermal stresses and confirm the fact that PVC is rapidly degrades than XLPE. It is also noticed that colorants and cable's manufacturing processes enhance the thermal resistance of the PVC insulation material. A comparative investigation of weight loss experimentation, IEC Standard 216-procedure, and ESR-experimentation verify the powerfulness and importance of the ESR testing of insulation materials used in cable industries. Hence, ESR results were utilized to predict more accurate thermal life time curves of the studied patches. The comparison of he thermal endurance parameters obtained for insulated cable models with those derived in the past for XLPE cable models shows that the all the pervious tests of XLPE overestimate its thermal endurance characteristics.

# Introduction

During insulation manufacturing, plastics are exposed to heat that lead to degradation. Although degradation can not be eliminated completely, it must be held to a minimum to keep the high quality of the final products. However, the low thermal stability of PVC has been an area of intensive research, the relationship between microstructure and degradation behavior is still not fully understood.

There are many conventional and analytical testing methods available for thermal endurance characterization of insulating materials [1]. The main advantage of the analytical tests to that of the conventional methods are their abilities to reduce test times without losing accuracy and practical significance of the results. Electron spin resonance (ESR) method is one of the most accurate analytical method that has special potential in material degradation analysis. It is well known that ESR spectroscopy is the only technique established to detect radicals from different sources [2-4]. Also, it has been widely used in polymers degradation analysis [5,6]. However, it has been never used for thermal endurance characterization of manufactured and raw insulating materials used by cable industries yet.

The present investigation will present the results of the direct testing and evaluation of thermal aging on widely used insulation materials (PVC and XLPE). The determination of thermal endurance characteristics of raw

and manufactured samples from major national cable factories using ESR spectrophotometer will be conducted. The results can be extended from application of life tests to more improvement and higher quality of cable's insulation materials and its manufacturing processes.

# **Experimental Section**

**Materials:** The experimental tests were performed on the samples as received. The tested samples were made of tiny pieces, which were placed into glass tubes of 25 mm length and 2 mm inner diameter (ID). The tubes were tagged as given in Table 1, where the first character, R or M, stands for raw or manufactured samples, respectively, the second and the third characters of the codes were chosen to symbolize plastic's type, while the last character is for coloring condition; N for non-color and R for red-colored insulation materials.

 Table 1: Tagging of insulating materials used in the experiment.

Insulating material type	Tag	
Raw <sup>a</sup> PVC non-color	RPVN	
Raw PVC red-color	RPVR	
Manufactured <sup>b</sup> PVC red-color	MPVR	
Raw XLPE non-color	RXLN	
Manufactured XLPE non-color	MXLN	
	Insulating material type Raw <sup>a</sup> PVC non-color Raw PVC red-color Manufactured <sup>b</sup> PVC red-color Raw XLPE non-color Manufactured XLPE non-color	

<sup>a</sup> Raw materials used in cable industry.

<sup>b</sup> Manufactured power cables insulation.

**Thermal Aging Unit:** A well controlled thermal device was used to age plastic samples at 130, 170 and 220 °C. The device consists of a heater tape, sample holder inside a well-isolated container that is equipped with a preprogrammable heat controller. External thermometer was placed inside the sample holder to show the actual temperature of the heating environment of the aging process.

**ESR Spectroscopy:** Varian E-line series spectrometer was used to detect the electron spin resonance (ESR) spectra of the plastic samples. All the ESR-spectral properties of these samples were performed at room temperature using cylindrical cavity along with 9 GHz Microwave Bridge. The working parameters of the spectrometer are as follows: time constant = 655 ms, modulated frequency = 25 kHz, modulation amplifier = 1 mT, microwave frequency = 9.34 GHz, signal gain = 104, sweep time = 120 s, and sample temperature = 22 °C.

### **Results and Discussion**

**ESR Results:** Samples from the collected ESR-spectra of PVC and XLPE insulation materials during their aging process are illustrated in Figure 1.



Figure 1: ESR spectrographs of raw PVC-red colored (RPVR) and of raw XLPE-non colored (RXLN) as received (Non-aged) and after aging at 220 °C for about 23.5 hours

The results indicate that the major difference between the non-aged and aged spectral properties is the appearance of a singlet peak in the middle region in the graphs of the deteriorated samples. Similar ESR-pattern was reported during thermal degradation [7-9], photo degradation [10,11], or chemical dehydrochlorination [12] of PVC compound. It was also observed in the  $\gamma$ -irradiated polyethylene (PE) compound sample by doses of several thousands megarads [13-16]. This midel signal can be attributed to the formation of conjugated polyene macroradical systems (Scheme 1) as an essential thermal degradation product.

$$-CH_2 - CH - (CH = CH)_n - CH_2 -$$

Scheme 1: Proposed molecular radical of degraded plastic samples

This chemical change confirms physical changes, in general plastic degradation, which favors treeing inception and growth up to breakdown when voltage is applied [17,18]. Figure 1 also shows that the ESR-peak intensities, which are correlated with the amount of the radical contents of the studied sample, of the aged PVC is almost ten times the observed one of the aged XLPE

sample. This high intensity of the aged PVC samples compared to XLPE is attributed to the ease of PVC degradation and is correlated very well with known stability of XLPE insulation materials toward thermal degradation relative to PVC materials.

Series of spectral measurements were performed and then their peaks' height-to-through were plotted as a function of time of aging (Figure 2) to pin-point the starting of the degradation. The time of the onset points (point at which ESR-peak starts to appear) was selected as the end point or the failure time (time-to-end-point, or life).



Figure 2: Accumulation of polyene radicals per sample (raw PVC-noncolored; RPVN, raw-PVC-red-colored; RPVR, manufactured PVC-redcolored; MPVR, raw XPLE-non-colored; RXLN, and manufactured XLPE-non-colored; MXLN) during aging at 130 oC.

Results in Figure 2 used to estimate the effect of cable processing and coloring additives to the insulation materials. Generally, nonlinear thermal behavior of the studied plastic samples is observed, except; (1) onset of the degradation process and (2) intensity of the ESR-peak within the reported aging period. The detected earliest onset of degradation of raw PVC (Figure 2) compared to the other types of plastic can be linked to the nature of additives, colorant or stabilizer, and their thermal stability. Firstly, this confirms the observed high intensity of PVC-ESR-signal compared to XLPE (Figure 1) that was attributed to the plastic stability toward thermal degradation. Secondly, the early onset of the PVC-noncolored compared to PVC-colored provides evidence on the selection criteria of pigments used in cable industry that is to enhance polymer stability. Moreover, a close analysis of the peak-to-through results in Figure 2 around eight hundred hours of aging period indicates that rawPVC contains about 2.5 more radical than the manufactured samples, while around and after 1500 hours of aging period, similar heights are obtained by all PVC samples. The enhanced degradation of non-manufactured PVC samples may be attributed to the production of the corrosive HCl-molecular system as bi-product but with much large quantity than those produced from the manufactured system.

Life Time Analysis: In accordance with IEC standards 216, insulating materials are classified on the basis of their "Temperature Index" (TI) [19], temperature corresponds to a time to reach the end point, failure criterion, of 20,000 hours. Another quantity is proposed for this rationale is the "Thermal endurance profile" (TEP) that is used to drive an endurance coefficient (B) where the higher the endurance coefficient is the greater should be the thermal endurance. One of the basic diagnostic tests to derive these endurance parameters is the aging test, that will be carried out on cable specimens to age their plastics without waiting for their failure. According to Dakin [20] the use of so called Arrhenius thermal life equation the failure time can be explained using:

$$L_o = L \exp\left(\frac{B}{T_o}\right)$$
 (1)

B = W/k, where W is the activation energy, k Boltzman's constant and the higher the activation energy is the greater should be the thermal endurance. T<sub>o</sub> is the absolute room temperature and L' is a constant for the material under consideration that equals the life of material when temperature tends to infinity. Equation (1) has been modified to represent the "conventional" thermal stress and then can be used for life time analysis (L) at any applied temperature (T) to produce deterioration:

$$L = L_o \exp(-B cT) \quad (2)$$

Where,

$$cT = \frac{1}{T_o} - \frac{1}{T} = \frac{T - T_o}{TT_o}$$

The conventionality is derived from choosing the room temperature as the reference one, where the thermal stress is explicitly represented by cT.

To justify the reliability of the ESR-procedure, thermal aging and hence life curve derivation are investigated for the PVC and XPLE insulation materials using ESR spectral results and weight loss (IEC standard method) results. Figures 3 and 4 show the output of these methods of the manufactured PVC and XLPE, respectively, against aging time.



Figure 3: The onset of the degradation based on Weight loss (A) and ESR (B) results at 170 oC ( $\blacktriangle$ ) and 130 oC ( $\bullet$ ) of Manufactured red-PVC sample from medium voltage cable.



Figure 4: The onset of the degradation based on Weight Loss (A) and ESR (B) results at 170 oC ( $\blacktriangle$ ) and 130 oC ( $\bullet$ ) of Manufactured noncolor-XLPE sample from medium voltage cable.

The time scale in the graph was placed into logarithmic scale because there is a possibility of putting very short and very long times in the same graph and as shown in Equation (1) the life parameter of thermal stress is of exponential type. Following the IEC Standard 216 of failure criteria (0.5 % loss of weight), the end point of thermal stress is identified of all the samples and compared to the onset time of the plastic deterioration on the bases of their radical's contents in the ESR spectra.

Table 2: Failure time in hours of PVC and XLPE insulations.

Test		-	Samples	S	
	RPVN	RPVR	MPVR	RXLN ,	MXLN
Weight loss:					1.1
Failure time at 170 °C	32	37	53	400	350
Failure time at 130 °C	230	380	410	3000	3800
ESR:					
Failure time at 170 °C	21	23	30	110	60
Failure time at 130 °C	120	200	230	1200	1050

The listed results in Table 2 indicates that the weight loss experiment overestimates the predicted failure times compared to those values achieved by ESR experimentation, where the estimated aging time of PVCsamples from weight loss is almost twice their correspondence from ESR and it is about three times higher of XLPE-sample at the tested temperatures.



Figure 5: Life curves (Upper graph) and temperature index (lower graph) of manufactured XLPE and PVC systems using different analytical methods: weight loss (WL), ESR, and electric strength (ES)

### Conclusion

Thermal history of cable insulations is essential criteria to predict cable's life. It consists of two factors; 1) temperature and 2) exposure time. This ESR-spectral results add a third factor; molecular properties of the studied samples. ESR-results show that the thermal behavior of XLPE insulation is better than PVC insulation. The results also confirm that colorants enhance the thermal stability of PVC insulating material. This analytical procedure is the most direct one yet to pin-point the starting time of the accelerated thermal degradation. It is also remove the doubt on finding the end-point criterion using different conventional methods, e.g., residual electric strength, tensile strength, and/or decrease of weight, to achieve a unique thermal endurance graph which can be related to degradation and loss of reliability of cables in service conditions. Finally, it has been observed that ESR-measurements can provide thermal endurance characterization consistent with or even better than those obtained by the other conventional methods for any insulation cable models; this could promote a more extensive use of such techniques.

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