B.9.2. Optimisation des procédures physico-chimiques d'analyses destinées à l'étude du vieillissement du polyéthylène, isolant pour câbles d'énergie

DUCHATEAU F. & Co-auteurs,
EDF/DER/ERMEL, Moret-sur-Loing,
France

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DUCHATEAU F. & Co-authors,
EDF/DER/ERMEL, Moret-sur-Loing,
France

Résumé

L'étude du vieillissement du polyéthylène, terme général utilisé pour les isolants PEBD, PEHD et PRC, a nécessité la maîtrise de méthodes physico-chimiques de caractérisation de ce matériau afin de détecter d'éventuelles évolutions morphologiques, chimiques ou électriques. C'est pourquoi un groupe de travail français comprenant des fournisseurs de matières premières, des fabricants de câbles et EDF dont la finalité était l'étude du vieillissement multi-contraintes du polyéthylène a d'abord travaillé sur la mise au point de méthodes d'analyses, comprenant des mesures électriques, l'analyse d'impuretés et la caractérisation morphologique, afin de les rendre fiables, reporductibles et indépendantes du laboratoire réalisant les mesures.

Abstract

The study of the intrinsic polyethylene ageing, polyethylene being a general term for the LDPE, HDPE and XLPE insulations, was possible only after the mastery of physical and chemical methods of the material characterization in order to detect any morphological, chemical or electrical evolution. That is why a french working group including raw material suppliers and cable manufacturers and EDF had the finality to study the polyethylene multi-stress ageing; they began to carry out the development of analysis methods including electrical measurements, impurity analysis and morphology characterization, to obtain reliable and reproducible measurements which are independant on the laboratory.

1-Introduction

Doing an analysis seems to be particularly easy when it is carried out with a simple and routine technique. Therefore, each analysis must be done carefully and with the mastery of the knowledge of the studied domain. The experiments initiated by the working group on cables insulations proved it.

The objective of this working group was to visualize the property evolution of the insulations during ageing, to propose ageing tracers and end-life critera. This study needed a certain number of more or less sophiticated analytical techniques. For these techniques which had been used daily for a long time by each member of the working group for development, qualification or expertises; we discovered that we were unable to compare our results, thereby to share them. During the first investigations, we noticed deviations of the results obtained in different laboratories from equivalent samples with the same techniques, and sometimes with apparatus from the same suppliers. We concluded that experimental parameters must differ from one laboratory to another.

To reach this objective, we decided to begin the study of experimental parameters of several of these techniques : Differential Scanning calorimetry (DSC), moisture content (Karl-Fisher), Infrared microscopy (FTIR) and short-time dielectric breakdown field (Go). These techniques have been investigated in order to determine the main parameters, to fix some of them, and to give recommendations on the measurements and cautions that lead to quality results. Round robin tests have also been performed in order to validate this study.

2-Optimization of physical and chemical procedures

2.1-Electrical measurement "Short-time dielectric breakdown measurement"

2.1.1 Statistical treatment method

WEIBULL MODEL

In the light of what we know presently on breakdown it appears that this variable has strong statistical character on extruded cables. This is the reason for which statistical treatments have appeared essential to characterize the dielectric strength.

In a system of n identical components, breaking probability of the Pc chain is bound to the breaking probability Pm of a link by the relation :

$$Pc = p^{n/m}$$

In the formalism of Weibull, the quantity $\log(\ln \frac{1}{1-P(X)})$ is expressed linearly as a function of the logarithme of the variable X determined by way of experiment, where X is either the variable of time (t) or the gradient (G), and can be written under the linearized form :

$$\log(\ln \frac{1}{1-P(X)}) = \alpha \text{ Log } (X) + \text{Log } (K).$$

The nominal gradient shall be called Go, value for which the density of probability is maximum.
Gs is the value of the gradient below which the probability of breakdown is zero.
α is the slope of the curve.

Model with two or three parameters

In our study, the results have been obtained after treatment by the two statistics methods of calculation of the curve : method of the least error squares and method of the maximum likelyhood. For most of the population, the method of the maximum likelyhood gives, visually, a better estimation. The method of the maximum likelyhood has only been retained.

2.1.2 Parametral study