A NEW METHOD FOR MONITORING THE ANTIOXIDANT CONTENT IN CABLE COMPOUNDS DURING E-BEAM CROSSLINKING

Matthias **MEYER**, Inge **DUEMMLER**, Nexans Research Center, (Germany), Matthias.Meyer@nexans.com

ABSTRACT

Within this article we give a short introduction into a versatile method called HPTLC, which allows the monitoring of antioxidants with high accuracy. We have quantitatively shown with HPTLC, that antioxidant amount decreases with increasing radiation dose, but it was shown, that the antioxidants have different stability. Monitoring of antioxidant content can be used to optimize the amount and the mix of antioxidant in modern cable compounds.

KEYWORDS

HPTLC; antioxidant; e-beam; crosslinking.

INTRODUCTION

During e-beam crosslinking of cable compounds polymer chains are connected with each other and this results in crosslinked cables. The cable compound has to be crosslinked with the right choice of radiation dose and additives for crosslinking. It is very important to obtain a high quality cable which fulfills the mechanical requirements and also has a good ageing protection during cable life time.

High-molecular antioxidants could not be detected in a proper way with GC-MS (Gas Chromatography-Mass Spectrometry), because of the high molecular mass. In our study we describe a method for monitoring the antioxidant content as a function of different e-beam doses by use of HPTLC (High Performance Thin Layer Chromatography). This test-method can be used for different antioxidants ranging from low to high molecular weight [1]. The key of successful analysis is the right choice of the solvent combination and the right measurement parameters.

These basic studies regarding the consumption of antioxidants during e-beam crosslinking give important information for manufacturing high quality cables [2], which have good mechanical properties and good ageing protection.

DETERMINATION OF ANTIOXIDANT CONTENT WITH HPTLC IN POLYMER MATRICES

HPTLC is a versatile method for determination of different chemical substances and of their amount and purity [3]. This method is less expensive than other techniques like GC-MS, LC-MS and HPLC. HPTLC can be used to detect e.g. high molecular phenolic or phosphorus based antioxidants and also lower molecular weight substances like BHT, but the list is not complete and can be enhanced by a huge range of substances.

Extraction of antioxidants

Before the measurement with HPTLC it is necessary to extract the antioxidant from the polymer matrix. This task is the most important step during sample preparation, because a good and quantitative extraction of antioxidant is the key for a reliable analysis. The extraction yield depends on the right choice of solvent. In case of non-crosslinked samples the polymer can also be solved in a solvent, but this is not useful for the quantification of the antioxidant. A good solvent should extract the antioxidant in a quantitative fashion and the polymer itself should not be solved. We extracted in our case 16 hours with ideal solvents in a soxleth apparature.

Separation of antioxidants with HPTLC

After extraction we have an extract containing the antioxidant and also other soluble ingredients from the polymer compound (e.g. plasticizer). The aim of the analysis is the quantification of the antioxidant content as a function of different radiation conditions and because of this we need a good separation of the antioxidants from the other ingredients. This can be realized by the right choice of the HPTLC-plate and the right choice of solvent combination as eluent. We used in our case a combination of toluene/acetic acid. The antioxidants have been separated from each other and also from the other compound ingredients. Figure 1 summarizes the steps from extraction to quantification.

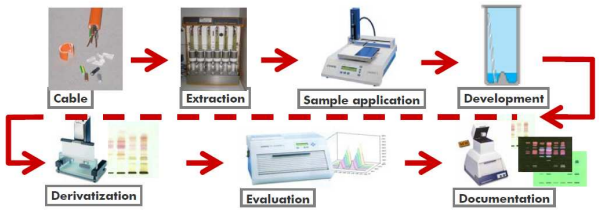

Fig. 1: Working steps from extraction to antioxidant quantification.

Quantification of the antioxidants

The quantification can be done with a HPTLC scanner by monitoring the spot area of each substance under UV irradiation. In case of certain antioxidants we can also add a derivatization step in order to make these substances visible. The area of each substance spot can be integrated with the software and additionally we receive the UV-spectra for each substance as additional information. The calibration was done with the corresponding reference standards. Figure 2 shows an HPTLC-plate with different separated substances under UV-irradiation for visualization of the substance spots.