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Test method

A comparative study on the thermo-oxidative stability of polyethylene

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ABSTRACT

The main goal of this work is to establish a correlation between the oxidation induction time (OIT) and the oxidation induction temperature (OIT*), which are frequently used to assess the thermo-oxidative stability of polyolefins, either as raw-materials or as finished products. Several tests have been performed at different temperatures (200, 210 and 220 °C) using a set of polyethylene (PE) samples from different sources. The correlations found - based on a quadratic regression – make it possible to estimate the OIT value based on experimental data of OIT* and vice-versa. This method can be quite useful in cases when the PE is highly stabilized so that the OIT test at 200 °C is expected to last for hours. Additionally, an Arrhenius plot has been developed to obtain the activation energy (E_a) associated with the beginning of the thermo-oxidation for each PE, which also allows us to determine OIT from calculated Ea. All estimated values, obtained by the developed regression models, using unknown specimens, have been confirmed by experimental testing. The regression models have also been checked by a residual analysis as a way to evaluate their reliability. The contribution of the uncertainties of measurement and calculation has also been evaluated, leading to increased reliability of the findings.

1. Introduction

Due to the structure of the linear molecules, high density polyethylene (HDPE) has high crystallinity, which results in high rigidity and low permeability. The balance of its good physical properties in the solid state and its chemical inertness, as well as its low cost and ready processability, make PE the material of choice for a wide variety of uses [1], in particular for the manufacture of pipes for conducting water and gas. For these applications, the thermoxidative stability of the polyethylene is a key feature. Thermo-oxidation results from the combined action of oxygen and heat. It is a source of free radicals and other reactive chemical species which decrease the regularity of the chemical structure of the polymer by breaking down the polymer chains through the creation of branching and the crosslinking of polymer molecules, and reduce the molecular weight, the crystallinity and, consequently, the mechanical properties of the polymer. The degree of thermoxidative stability of PE is influenced by several factors, in particular the degree of aging, the manufacturing process, as well as the content and nature of antioxidant additives. The thermoxidative resistance of PE is also affected by the presence of recycled polymer. The thermoxidative stability of PE is assessed by differential scanning calorimetry (DSC) using two distinct parameters [2]: the induction time to oxidation (static OIT), which is a slower and less economical method, and the oxidative induction temperature (dynamic OIT), for which the determination is fast and expeditious, and thus more economical. However, the product standards [3,4], suggest only the first of these methods, setting a minimum OIT of 20 min at 200 °C, in order to ensure that the pipes resist oxidation during the manufacturing process and during their service life [5]. It is very useful to determine a reliable correlation between these two parameters and also

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