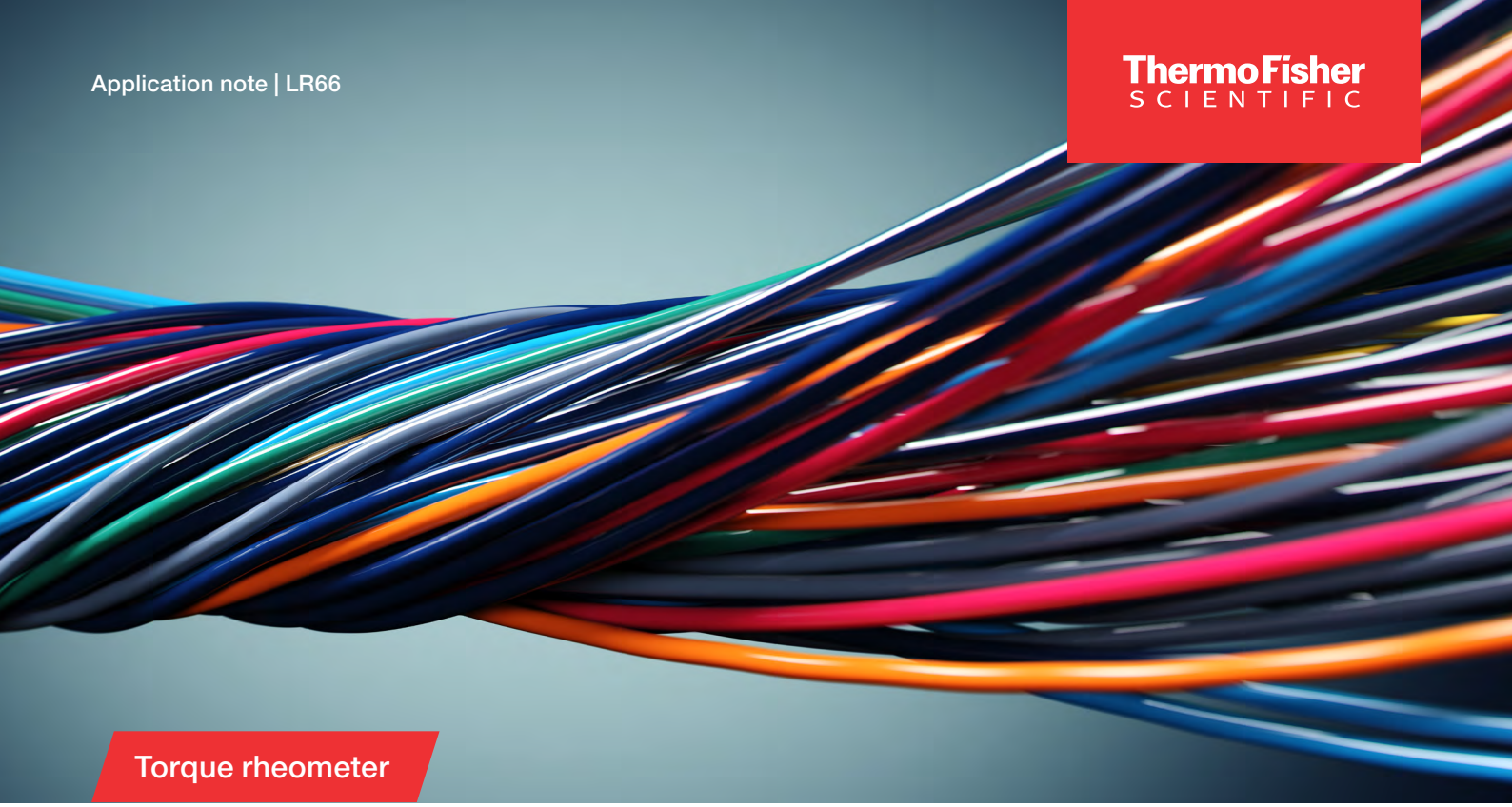

Torque rheometer

Examining the fusion and degradation behavior of PVC dry blends with the HAAKE PolyLab QC

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Keywords

Mixer test, PVC dry blend, fusion and degradation behavior

Abstract

PVC dry blends often have the tendency to exhibit greatly differing melt and degradation characteristics after undergoing comparatively small alterations in their recipes. This can lead to a variety of problems during processing such as reduced impact strength or the coloring or degradation of the extrudate. The Thermo Scientific™ HAAKE™ PolyLab™ System offers a quick and reliable method of examining these material characteristics.

Figure 1: HAAKE PolyLab QC with Rheomix 600 Laboratory Mixer

Introduction

The aim of the test was to differentiate between two PVC compound formulations, which only differ in composition by 0.1 % stabilizer content.

Test sample

PVC dry blend compound with:

- PVC19STB = 1.9 % stabilizer
- PVC20STB = 2.0 % stabilizer

Test equipment

A HAAKE PolyLab Torque Rheometer configuration (Figure 1) with the following components:

- Thermo Scientific™ HAAKE™ PolyLab™ QC Torque Rheometer
- Thermo Scientific™ HAAKE™ Rheomix 600 Laboratory Mixer
- Roller rotors
- Thermo Scientific™ HAAKE™ PolySoft Mixer Software

Test conditions

- Mixer temperature: 170 °C
- Rotor speed: 60 rpm
- Sample weight: 65 g

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Test result and evaluation

Figure 2 illustrates the results of the mixer test of the PVC compound formulation with 2.0 % stabilizer.

Basic curve discussion

The initial filling of the mixer results in the first Torque Peak or Loading Peak (L). This peak serves as the reference point for the calculation of the various substance characteristics. At the time of this peak, the sample is distributed in the hot mixer chamber and additives with a low melting point do melt. Because of this, the torque value drops to a minimum value known as the Valley (V). Due to friction and heat the PVC powder starts to agglomerate. This increases the compound viscosity and causes a second rise in torque. The agglomeration process is finished when the compound has formed a homogenous melt. In the torque curve this can be seen as a second maximum, the Fusion Maximum (F).

After the fusion, the torque drops again until the melt temperature of the sample comes to equilibrium between the mixer temperature and the heat generated by shearing. The resulting second minimum (Mi) is a relative value for the melt viscosity.

The stabilizers added to the PVC compound have the task to catch chlorine ions which were separated from the polymer chains. A stabilizer has a certain capacity to bind those chlorine ions. After the stabilizer is saturated, the free chlorine ions will cause the PVC degradation.

This degradation comes along with a crosslinking reaction which can be detected as a third rise of the torque curve. The point where the torque value starts to rise again is called the Onset of Degradation (On). This characteristic point is usually defined as 10 % of the increase of the torque signal after having passed the Minimum Torque Value. The so-called Stable Point (St) is reached by locating this 10 % value before the Minimum. The time between the Stable Point and the Onset of Degradation point (Stable Time) is generally taken to be a relative measure for the compound stability against thermal degradation.

Results and discussion

The torque / time curves of both PVC compounds are shown superimposed in Figure 3. One can clearly see that the sample with a higher stabilizer content of 2.0 % has a longer Stable Time. The graph also shows that the difference of 0.1 % in stabilizer had no influence on the Minimum nor on the melt viscosity of the compound. Very important is the fact that the change in stabilizer content does show a significant effect on the fusion behavior of the PVC compound. The compound with the higher stabilizer content needed a longer time to reach the fusion maximum.

During production, this would have a significant influence on the degree of the compound gelation and so it would also affect the mechanical property of the final product.

Summary

As can be seen from this example, twenty minutes spent on prior sample testing can prevent subsequent time-consuming and costly production problems.

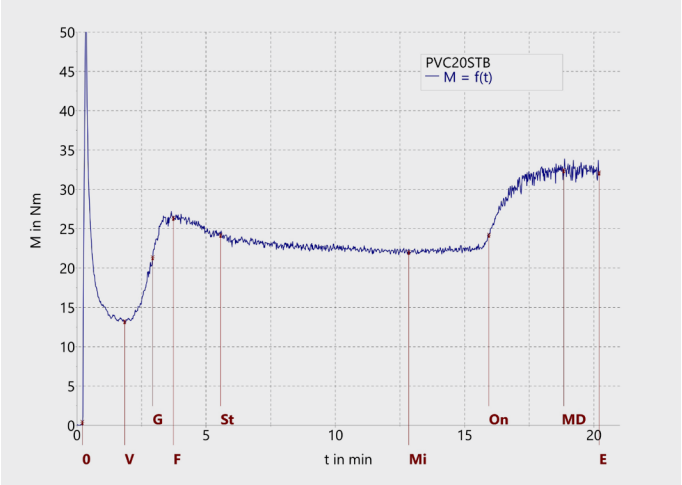

Figure 2: Mixer Rheogram of PVC fusion and degradation behavior.

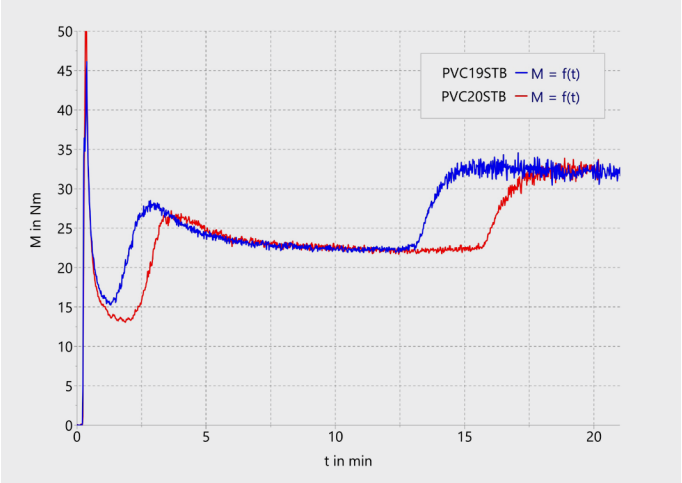

Figure 3: Comparison of two PVC-Masterbatches with different stabilizer content.

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