TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Determination of Carbon Black Pigment in Nylon 66 by TGA*

Number TA-122

Summary

Carbon black is a common pigment used in plastics such as Nylon 66. The ability to rapidly assess the level and proper dispersion of carbon black in a plastic is important to ensure that the mechanical properties and cost of the final material are not adversely affected. Thermogravimetric Analysis (TGA) provides a convenient method for making the determination of carbon black content.

Introduction

Nylon 66, a polyamide, has been used for many years in the plastics industry in a variety of application areas as diverse as fibers, apparel, molded components, glass reinforced laminates and fasteners. In these applications, Nylon 66 is often pigmented. The most common pigment used to produce a black color is carbon black, with relatively small amounts of carbon black (less than 3%)capable of producing a consistent black color.

Pigmentation of Nylon 66 is not always a straight forward process and producing a homogenous mix of carbon black and polymer prior to processing is sometimes difficult. Hence, quantifying the amount of carbon black in the Nylon 66 is important as a check of consistency to ensure that there are no areas of very highly pigmented polymer. High concentrations of pigment can have a detrimental effect on the mechanical properties of the Nylon 66 producing localized weak points. A second important consideration is cost. Carbon black is expensive relative to the base polymer and it is important to ensure that no more carbon black is used than is necessary, as higher loadings of carbon black will increase the cost of the finished product and will not enhance the color.

Thermogravimetic Analysis (TGA) is an established technique used in the quantification of weight changes within a material as a function of temperature or time. TGA can be applied to such diverse application areas as compositional analysis, moisture content, volatility studies, determination of decomposition mechanisms, and ash content. In addition to temperature, purge gas (sample atmosphere) is another variable that can be used to affect the TGA weight change results. By varying the purge gas in TGA during the analysis, separation (and quantitation) of polymer additives and the bulk polymer is sometimes possible.

Experimental

In TGA, the sample is placed in an open pan suspended from a sensitive microbalance (Figure 1). A furnace surrounds the sample to provide accurate heating during the measurement. A thermocouple located near the sample monitors sample temperature so that the weight change can be followed as a function of temperature. Purge gas flow across the sample is horizontal to assure good sample/atmosphere interaction while minimizing buoyancy and chimney effects. The experimental data obtained is stored and subsequently analyzed using dedicated data analysis software.

TGA 2950 SCHEMATIC - SAMPLE AREA

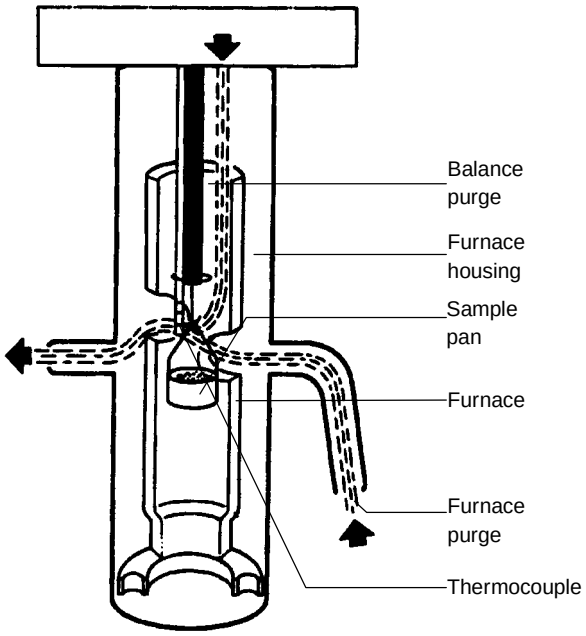

Figure 1

In this work, a Nylon 66 material containing carbon black pigment was analyzed from ambient to 1000°C using a TGA 2950 Thermogravimetric Analyzer. The programmed heating rate was 20°C/minute. The sample atmosphere (furnace purge) was nitrogen below 650°C and air above 650°C using flow rates of 100 ml/minute. Gas switching was automatically controlled by the Thermal Analyst 2100 using a Gas Switching Accessory.

Results

Since a certain amount of carbon char is produced from the thermal decomposition of the base Nylon 66, it is necessary to first quantify this amount in order to subtract it from the total carbon produced from the pigmented sample thereby obtaining the carbon black contribution.

Figure 2 shows the thermogram resulting from the analysis of 13.147mg of unpigmented Nylon 66. The results show a small and very gradual weight loss between ambient and 300°C. Across this same temperature range the derivative weight signal displays a very broad peak centered at 200°C. This weight change is most likely due to the evolution of traces of moisture, volatiles or unreacted monomer. TGA is a quantitative technique and will not give information as to the identity of evolved materials. By linking the TGA to a secondary analytical device such as Fourier Transform Infrared Spectroscropy (FTIR) or Mass Spectrometry (MS), however, it is possible to identify the evolved species.

Above 300°C the Nylon decomposes in a single smooth step as shown by the symmetrical derivative profile with peak temperature of 410°C. This weight change corresponds to the decomposition of the base polymer to leave residual carbon char from the polymer back bone. This carbon char is quantified as 1.53% at 590°C where the weight profile is at a plateau and does not change under the inert nitrogen atmosphere.

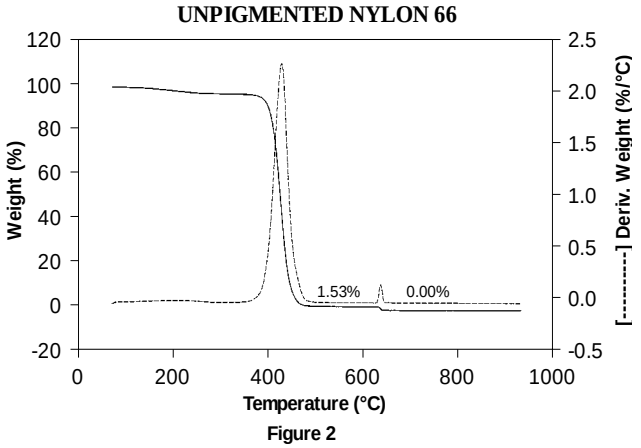

Figure 2

At 650°C switching the purge gas to air results in the oxidation of the carbon char to leave inert inorganic residue. The value of the weight at 730°C is 0.00% which proves that the sample is entirely organic with no inorganic filler or pigment.

A carbon char content of 1.53% is a large amount relative to the quantity of pigment that may have been added. It is important, therefore, that the char content be quantified reproducibly. This is checked by running a duplicate sample under exactly the same conditions as above. Figure 3 shows the overlaid thermograms from the duplicate analyses. The weight loss profiles are identical.

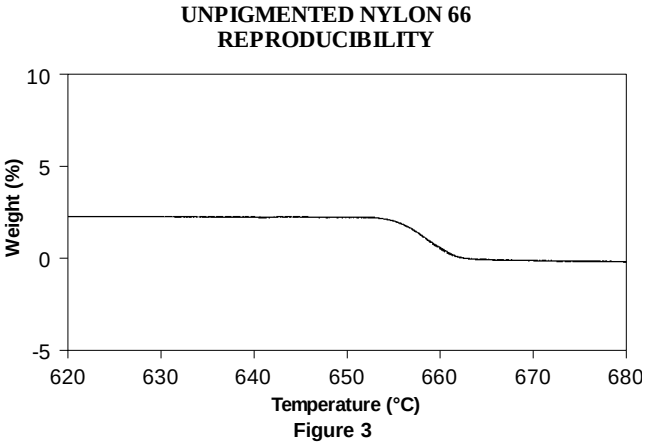

Figure 3

Figure 4 shows the TGA decomposition curve for 13.06 mg of the pigmented Nylon 66. Since this sample foams during the main decomposition and can contact the instrument thermocouple, platinum foil was wrapped around the sample during evaluation. This results in subtle changes (shoulders) in the TGA derivative curve, as well as lower intensity derivative peaks. Both of these subtle differences in the derivative curve are related to the lower surface area exposed to thermal and purge gas effects. This may reflect a change in decomposition mechanism, but has no effect on the carbon black determination. The plateau value in nitrogen before air switchover is 3.05%. Subtracting the 1.53% char due to the base polymer yields 1.52% as the weight of carbon black. This value agreed well with the pigment loading targeted by the manufacturer as providing the optimum combination of pigmentation and cost. Furthermore, additional runs on samples taken from different sections of the material yielded similar values indicating uniform pigment distribution throughout the polymer.

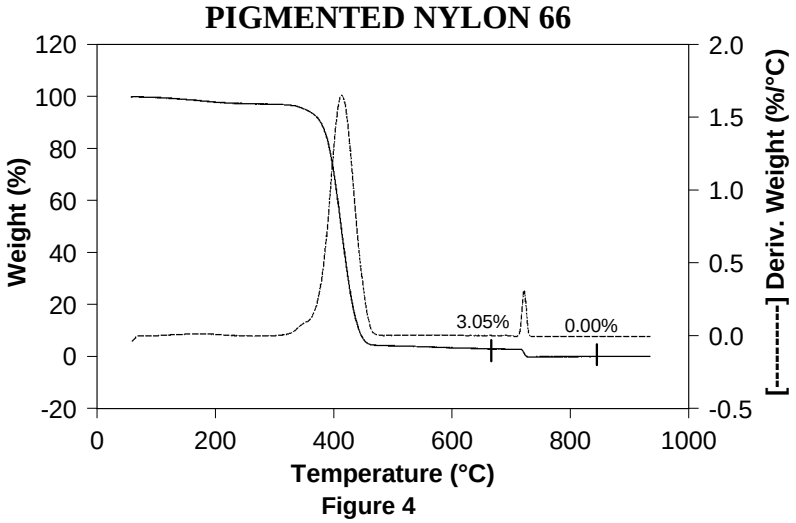

Figure 4

*Acknowledgment

This brief was developed by I. Groves, T. Lever, and N. Hawkins of TA Instruments Ltd. (U.K.)

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