

Thermal Analysis

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Polystyrene Oxidation Using a TGA Hyphenated to a Gas Mixing Device (GMD)

Introduction

Thermogravimetric analysis can be used to study the effects of gas sorption on materials, redox reactions at the surface of a material, and the thermal stability of materials all under

a control environment. To carry out such studies a Thermogravimetric Analyzer (TGA) can be hyphenated with a Gas Mixing Device (GMD 8000), which can provide a custom gas environment. The PerkinElmer GMD 8000 allows a user to blend up to three separate gases and introduce them to the sample chamber as needed. The unit is controlled through standalone software giving the user the flexibility to use other instruments including the PerkinElmer DSC 8000/8500, STA 6000/8000, or other third party instruments.

Materials

To demonstrate the effects of varying gas concentrations on materials, a PerkinElmer GMD 8000 and the PerkinElmer TGA 8000™ were used to analyze a low density polystyrene sample. The polystyrene sample was exposed to different concentrations of oxidizing gas as the sample was heated in the TGA. The oxidative degradation of the polystyrene as it relates to the gas concentration was studied. The sample size was limited to 9 mg ± 1 mg.

For simplicity, mixture of air (78.09% N₂, 20.95% O₂, and <1% others) and nitrogen (99.99% purity) was used in the study. The concentration of air ranged from 100%-0% of the total gas mixture.

GMD 8000 Method (Part Number N5320014):

1. Set channel A to nitrogen
 - a. Set the percentage of nitrogen desired for each individual run
2. Set channel B to air
3. Set total flow rate to a constant 40 mL/min

TGA 8000 Method (Part Number: N5320021):

1. Set sample purge flow to nitrogen @ 40 mL/min
2. Set balance purge flow to nitrogen @ 60 ml/min
3. Initial temperature 50 °C
4. Heat from 50 °C to 500 °C @ 30 °C/min
 - a. Trigger GMD 8000 ON at the start of the heating ramp
 - b. Switch the sample purge flow to 0 mL/min at the start of the heating ramp
5. Hold for 1 minute @ 500 °C
 - a. Trigger GMD 8000 OFF at the start of the heating ramp
 - b. Switch TGA sample gas flow to 40 mL/min at the start of the heating ramp

Results/Conclusions

With help of the GMD 8000, precise mixtures of gas were introduced into the system to study the effect on oxidative degradation of polystyrene. The data clearly shows increased concentration of the oxidation agent leads to early onset of thermal degradation in polystyrene. The onset temperature shifted from 418 °C with a 100% nitrogen environment to 390 °C with 100% air. A closer look at the first order derivative of the weight loss signal showed a decreasing decomposition rate with increased presence of the oxidation agent. As the results suggest, the incorporation of GMD 8000 to any oxidation mass loss study can be vital to a material's characterization profile. With its independent controls and software the GMD 8000 can be incorporated with any instrument or study which require complex mixtures of gases.

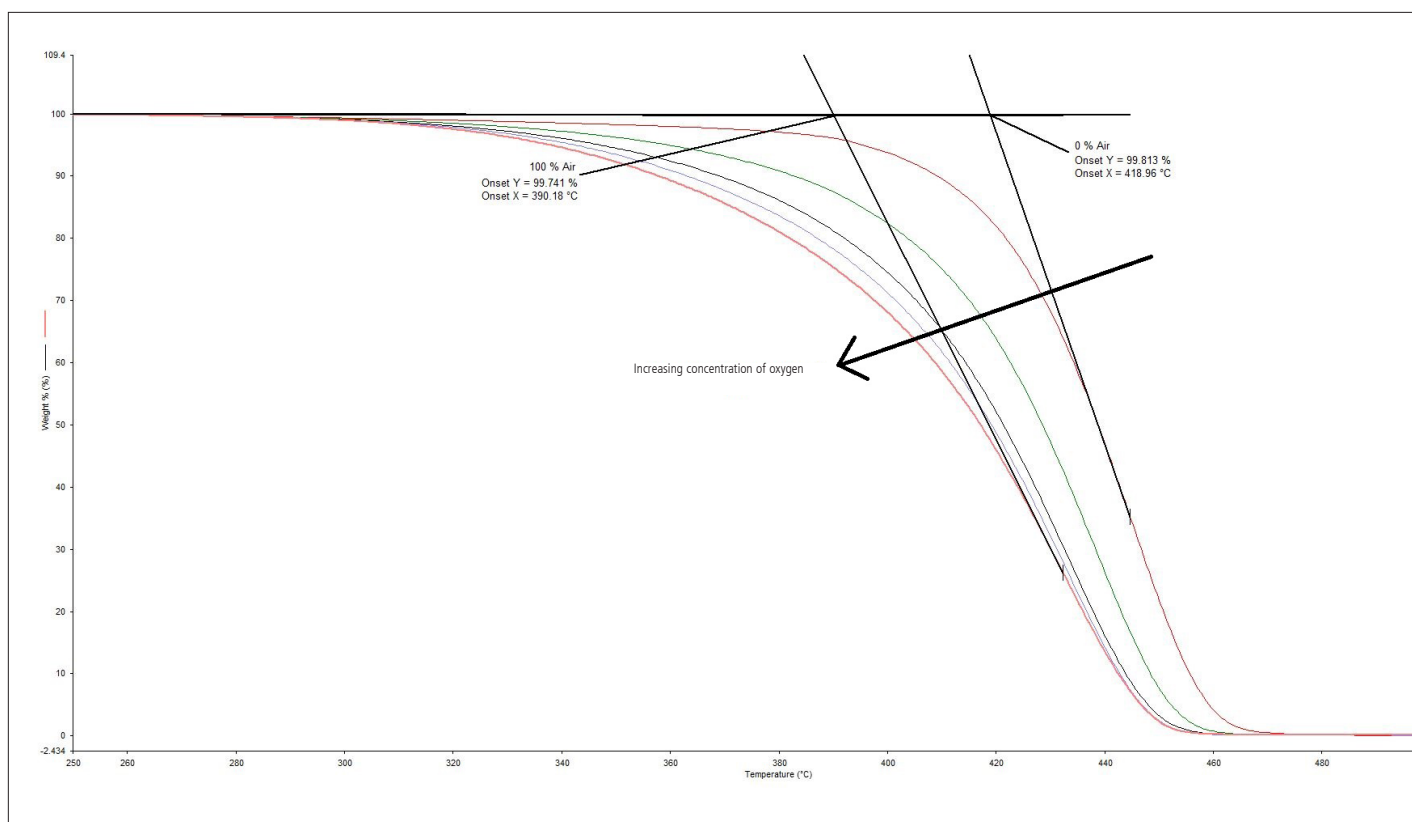


Figure 1. Weight loss curve of polystyrene as a function of oxygen concentration as acquired using the TGA 8000.

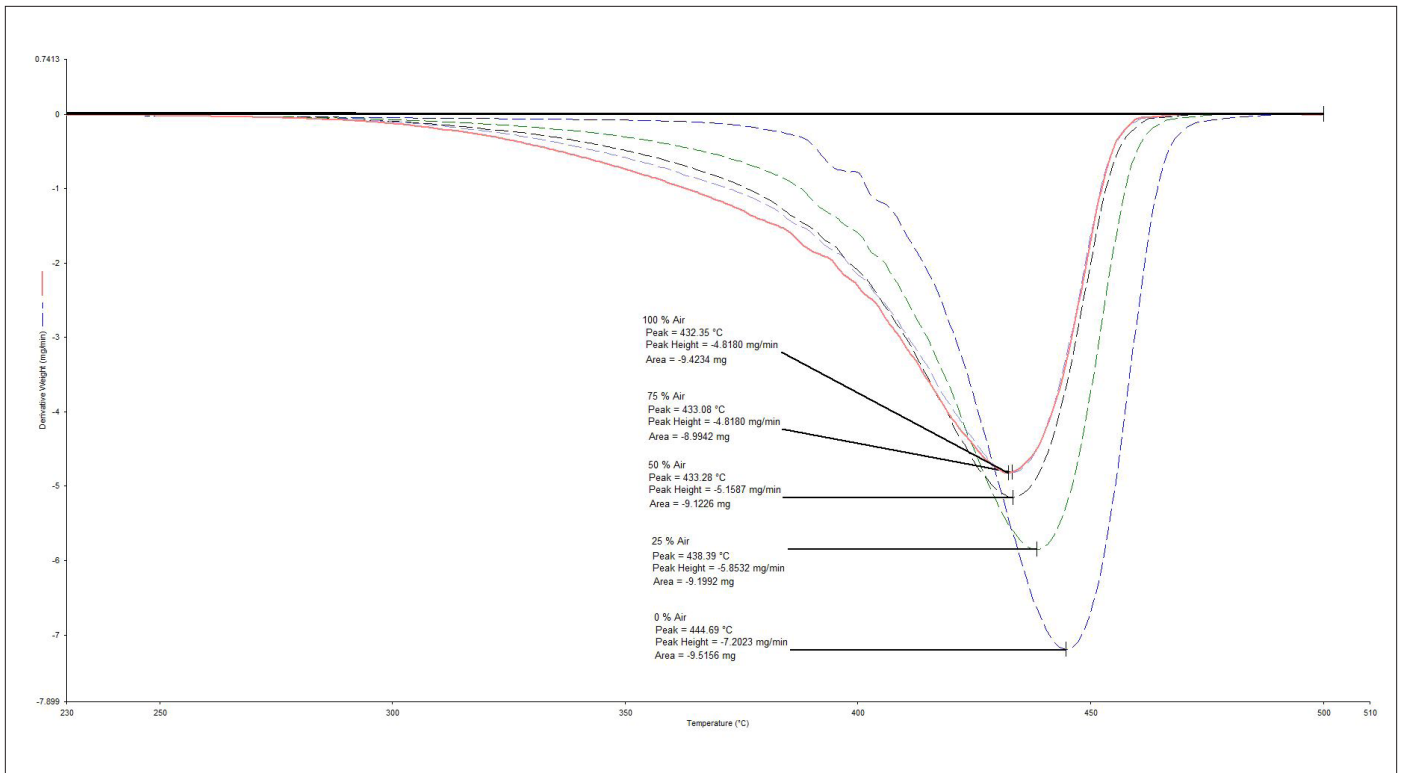


Figure 2. First order derivative of the weight loss signal highlights the decrease in degradation rate as higher concentration of air is introduced into the system.