Degradation Analysis of Poly(ethylene terephthalate) via Fluorescence Spectroscopy

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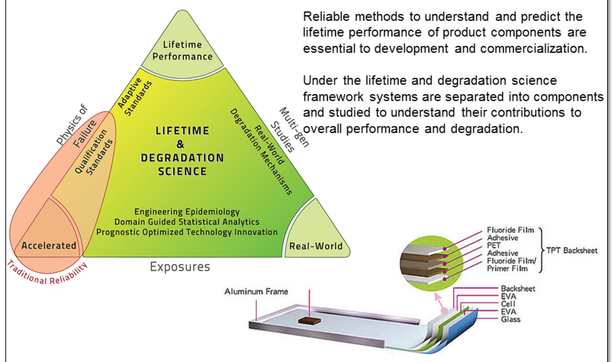
Introduction



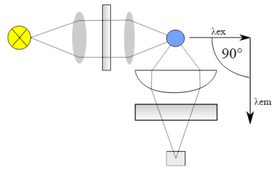
Reliable methods to understand and predict the lifetime performance of product components are essential to development and commercialization.

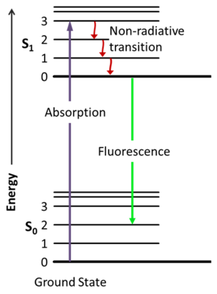
Under the lifetime and degradation science framework systems are separated into components and studied to understand their contributions to overall performance and degradation.

Fluorescence Spectroscopy

- Sample excitation by light source followed by detection of emission radiation
- Probes molecular structure
- Can be used to monitor chemical changes in materials

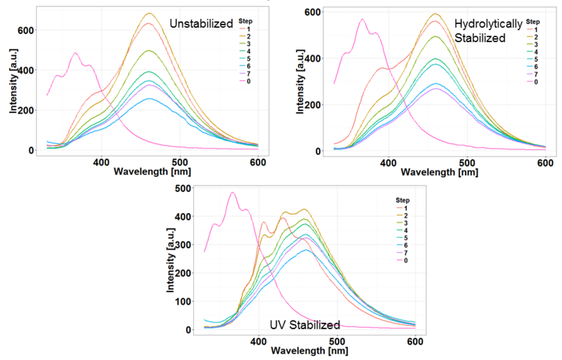
Excitation: $S_0 + h\nu_{ex} \rightarrow S_1$
Emission$_{(Fluorescence)}$: $S_1 \rightarrow S_0 + h\nu_{em} + \Delta$



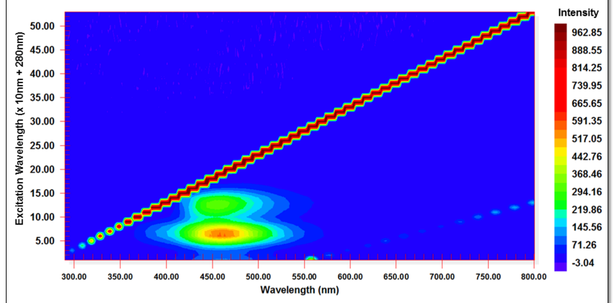


Modified ASTM-G154 Exposure Results

- Decrease in PET fluorescence intensity – Loss of distinctive PET peaks
- Redshift in fluorescence emission in all grades



Future Work: Three-Dimensional Matrix Fluorescence Spectra

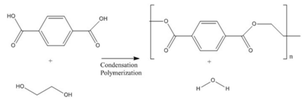


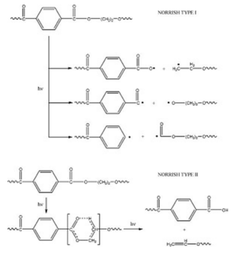
Objectives:

- Measure and plot three-dimensional excitation and emission matrix spectra to maximize data collection and information.
- Identify PET Degradation mechanism pathways for various exposure types.

Poly(ethylene terephthalate)

- Condensation polymer of terephthalic acid and ethylene glycol
- Useful as core layer in PV backsheets because of its high dielectric breakdown strength and low cost
- Degrades under exposure to environmental stressors
- Stabilizers added to mitigate or prolong degradation processes

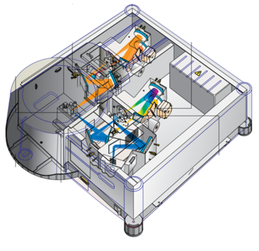


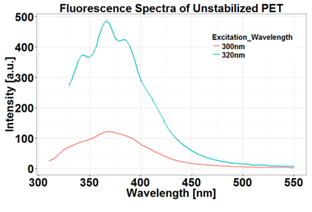


Material	Thickness
Unstabilized PET	75 µ
UV-Stabilized PET	50 µ
Hydrolytically Stabilized PET	125 µ

Fluorescence Spectroscopy of PET

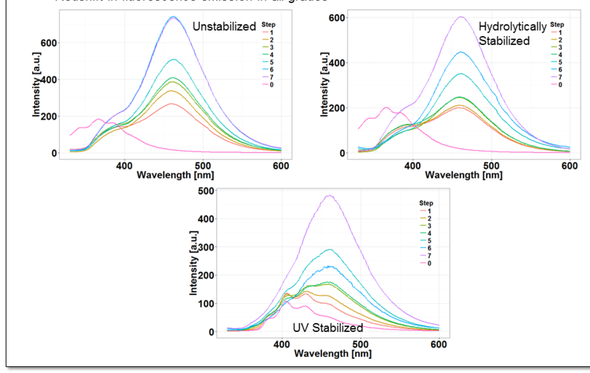
- Instrumentation Agilent Cary Eclipse Fluorescence Spectrophotometer
- Allows characterization of PET and degradation products
- Performed with 300 nm and 320 nm excitation wavelength
  - Note the differences in spectra
  - However, both demonstrate same trends with exposure steps





ASTM-G154 Exposure Results

- Increase in PET fluorescence intensity - Loss of distinctive PET peaks
- Redshift in fluorescence emission in all grades



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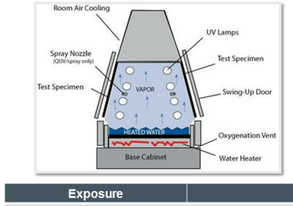
  - Ronald Steffen

Accelerated Weathering

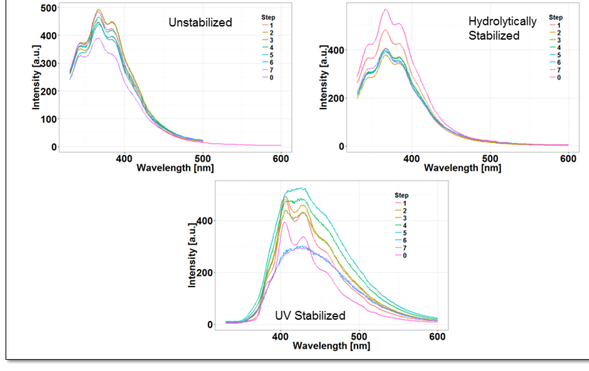
- Exposure protocols in Q-Lab QUV Accelerated Weathering Testers and Cincinnati Sub-Zero Environmental Test Chamber
- Administered combinations of heat, humidity and UV irradiance



Exposure	Description
ASTM G-154	8 hours of UVA exposure of 1.55 W/m^2 at 340 nm and 70 °C followed by 4 hours of condensing humidity at 50 °C in the dark
Modified ASTM G-154	Continuous UVA exposure of 1.55 W/m^2 at 340 nm at 70 °C
Damp Heat	Continuous exposure at 85 °C and 85% relative humidity

Damp Heat Exposure Results

- Decrease in PET fluorescence intensity - Loss of distinct PET peaks in UV Stabilized grade



Conclusions and Work in Progress

Conclusions:

- Fluorescence properties of unstabilized and stabilized grades of PET were characterized
- Changes in fluorescence spectra were observed after accelerated weathering
  - Provide insight into the molecular structure changes of PET as a result of degradation
- Observed different fluorescence responses as a function of weathering exposure type
  - Provide insight into degradation mechanism and synergistic effects of exposures
- Observed function and effect of UV and hydrolytic stabilizer additives
- Of the exposure types, the cyclic combination of heat, humidity, and UV irradiance exposure proved most damaging

Work in Progress

- Full fluorescence spectrum measurements for luminescence intensity mapping
- FTIR measurements to correlate results with specific structural changes
- Larger study of transparent and opaque samples
- Nanoindentation to correlate degradation with loss of mechanical properties