Developing Measurement Science for SLP of PV Polymeric Materials:

Interdependent Multi-Stress Effect on PV Laminate and Backsheet Degradation

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Acknowledgments: Eric Byrd, Debbie Jacob, Lakesha Perry, Jason Garver, Li-Piin Sung, Tinh Nguyen, Don Hunston, Chris White, Stephanie Watson; Brian Dougherty, Behrang Hamadani, Matthew Boyd; NIST-Industry PV Consortium

NIST 2m SPHERE

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NIST PV Array Gaithersburg, Maryland

National Institute of Standards and Technology
U.S. Department of Commerce

Interdependent Multi-Stress Effect

❑ What is it?

- The interdependent multi-stress effect is that the effect of one environmental stress on the response of materials depends on another environmental factor, *and **vice versa***.
(*Therefore, simultaneous/combined test could be substantially different from single test or sequential test.)*

-Such multi-stress effects can be **synergistic**, or **competing,** depending on the stressors and the material properties.

❑ Why does it happen?

- Multiple weathering factors can be interactive *(e.g., UV light and H_2O)*
- Polymer physical properties (*e.g., O_2 diffusion*) depend on the phases *(glassy, molten, etc.)*
- Polymeric material degradation usually involves multiple degradation modes, and their sensitives to changes in stressors could be different. *(e.g., photo-yellowing and photo-bleaching)*
- For accelerated laboratory testing, not all factors are accelerated to the same level, therefore, the leading degradation mode could change.

❑ Discussion limited to the Key Environmental Factors (UV (I, λ), T, RH, σ/ε) on polymer degradation from our SLP study.

NIST Service Life Prediction of PV Polymers and Components

Reliability-based Methodology

Laboratory Exposure

- Identify key environmental factors and quantify their effects on polymer degradation (UV (I, λ), T, RH, σ/ε …)
- Full factorial design, not only extremes; simultaneous.

Outdoor Exposure

(UV Spectral irradiance, T, RH recording)

linkage

Cumulative Da
M

utdoor
s

- ❑ **Multi-property measurements**
- ❑ **Failure mode and degradation mechanism study**
- ❑ **Database; Predictive models**

- **The parameters of predictive models are estimated solely from laboratory experiments; outdoor exposure results are used to verify the models using field exposure conditions as input (*Prediction models suitable to different climates*).**

- ➢ **To predict the service life of a polymeric material/component.**
- ➢ **To develop standards for accelerated laboratory testing for PV polymers.**

Laboratory and Field Exposure

SPHERE Exposure Condition

Outdoor Exposure

Materials and Components

(Full Factorial Design)

RH / Temp	0% (dry)	30%	60%
45°C	X R	X	X
65°C	X R,W	X	X
75°C	X W		
85°C	X W R	X	X R W

- UV Intensities (ND): 40, 60, 80 and 100 % of the sphere flux
- Wavelengths (WL): 306, 326, 354, 452 nm (band pass filters)
- No light

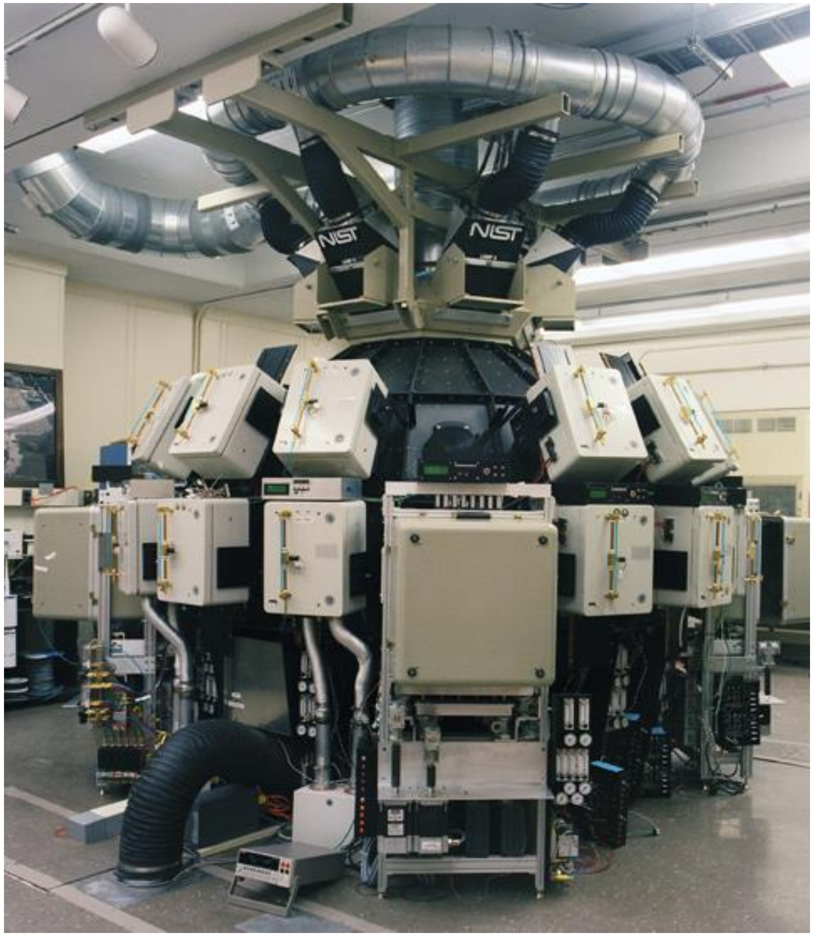
Indoor results are used to obtain parameters for SLP models.

- Simultaneous temp (25-85 °C), RH (0-75% RHs) and UV (~x1-3 Suns)
- Factorial Experiment design (4T, 3RH, 4 UV-I, 4 UV-WL)
- Reciprocity study
- Wavelength effect Study
- Temperature Effect
- Moisture Effect
- Degradation mechanism and failure mode study

- NIST building Roof; Florida; Arizona.
- T, RH, UV Spectral Irradiance measurements

$$S(t) = \int_0^t f[\mathrm{Temp}(\tau)]g[\mathrm{RH}(\tau)] \int_{\lambda_{min}}^{\lambda_{max}} E(\tau,\lambda)\left[1 - e^{-A(\lambda)}\right]\phi(\lambda)d\lambda d\tau$$

Accelerated Laboratory Exposure using NIST SPHERE:
Simultaneous UV/T/RH Exposure

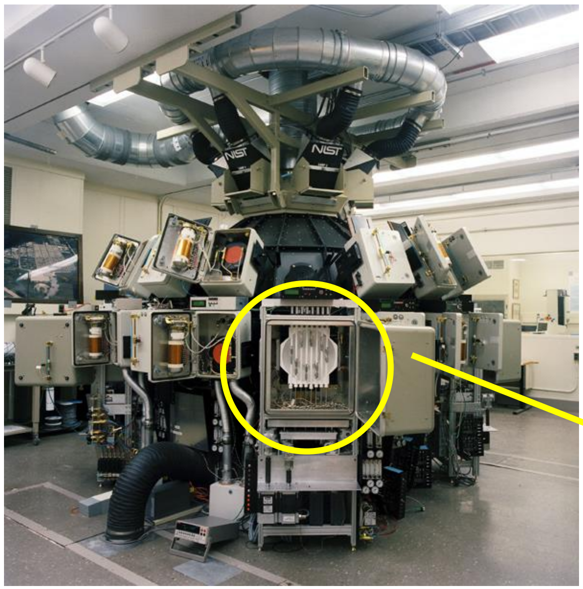


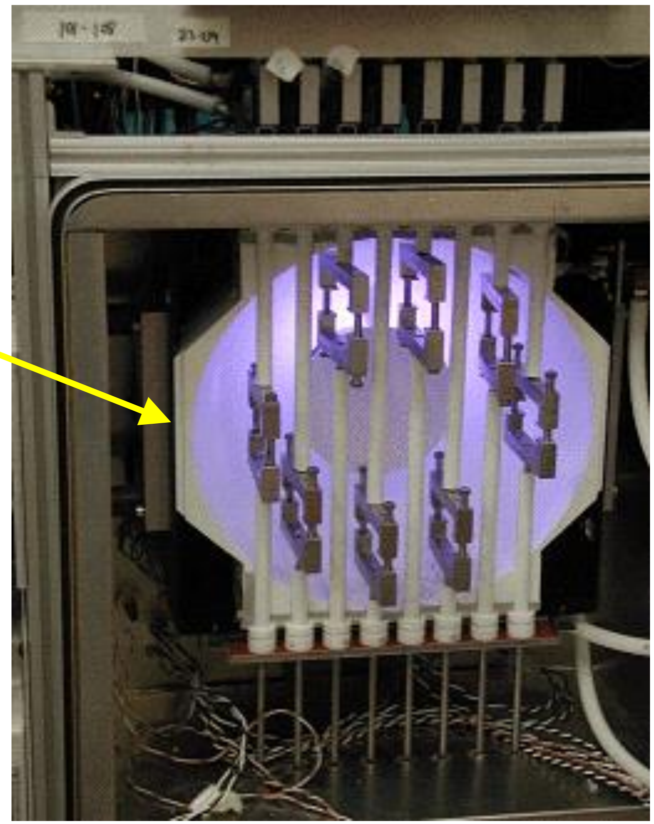
- Simulated Photodegradation via High Energy Radiant Exposure (SPHERE)
- 2 m integrating sphere-based UV chamber
- High Power Mercury Lamp
  8400 W UV
- 95% exposure uniformity
- Low wavelength <290 nm removed
- Most visible and infrared radiation removed
- Exposure conditions of 32 chambers can be individually controlled (UV, RH,T)
- Capability for mechanical loading

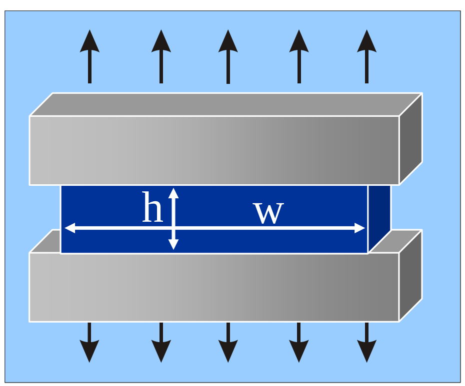
- *Martin and Chin, U.S. Patent 6626053*
- *Chin et al. Review of Scientific Instruments, 75(11), 4951-4959, 2004.*

NIST National Institute of Standards and Technology

Sealant Test Chambers







Test geometry ASTM C719

MUSIC – Labsphere's Commercial Versions

Labsphere can provide fully functioning systems that provide all the capabilities of SPHERE

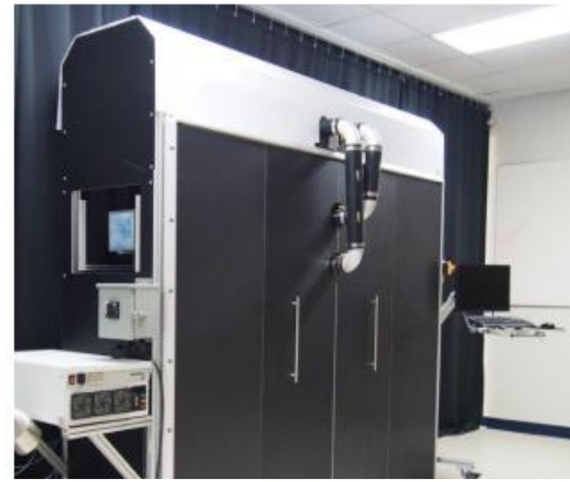
M – Multi-Sample

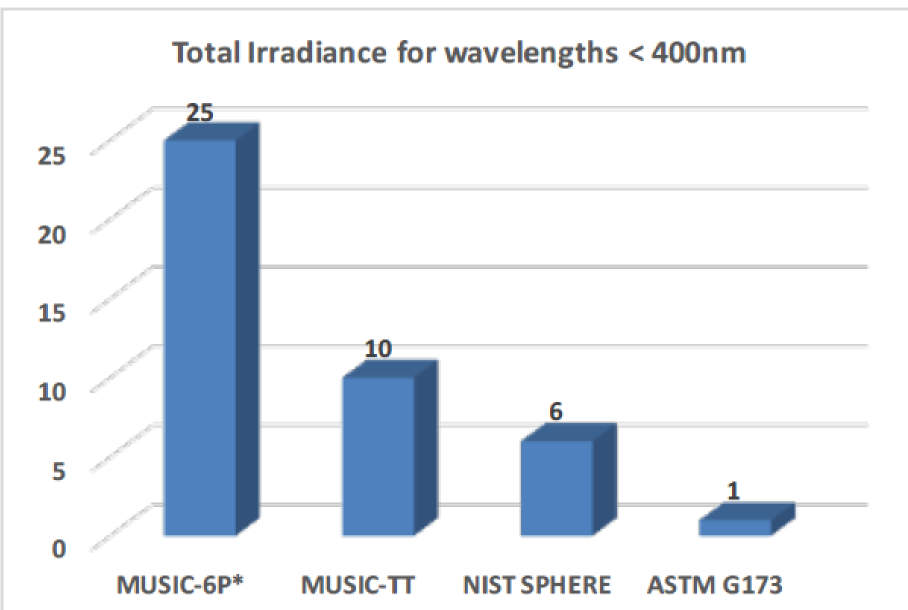
U – Uniform

U – Ultraviolet

6 Port

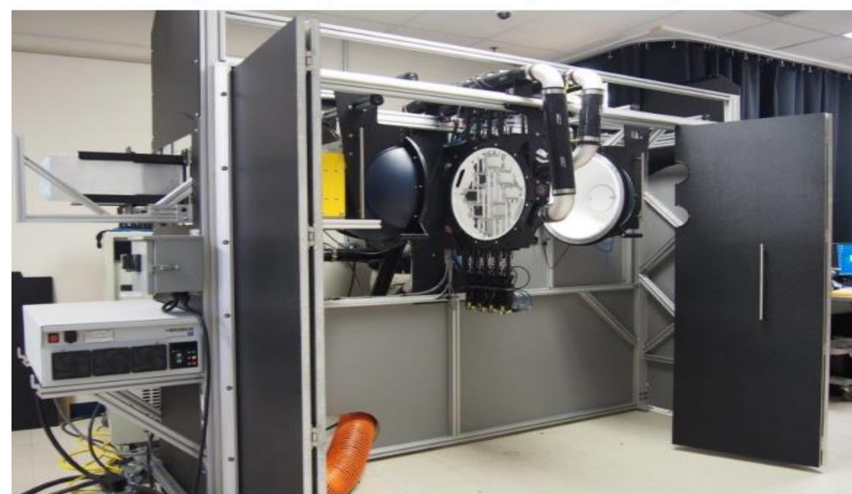
Tensile Tester





MUSIC-6P is an estimated value

System Opened with Sphere Separated

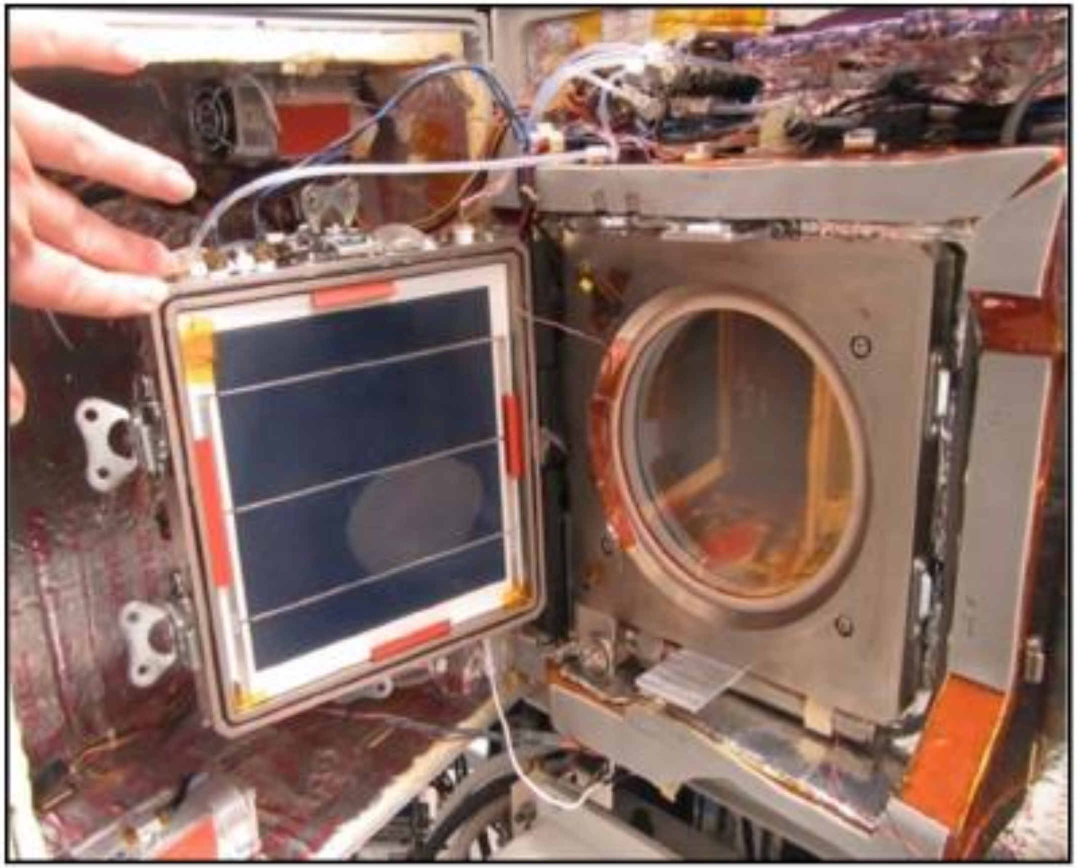


(ECU - Environmental Control Unit not visible)

T- RH – E_e Challenges

Overview

- Ongoing NIST PV module testing using MAC's Prototype HEWC on NIST's SPHERE UV source for Accelerated Weathering
- Measurement & Control of interactive parameters
  - High Temperature
  - High Humidity
  - High Irradiance



- MAC's Prototype HEWC operating range:
  - 35°C to 105°C, ± 1°C
  - -40°C dp to 90%RH @ 85°C, ± 2%RH
  - Full Envelope

Measurement and Control Challenges

In

Multiple Interactive & Synergistic Parameter Environmental Testing

John Sparks

Measurement Analysis Corporation
23850 Madison St. Torrance, CA

Examples of Interdependent Multi-stress Effects

❖ (UV (I, λ), T, RH, σ/ε)

(Full Factorial Design)

Temp \ RH	0% (dry)	30%	60%
45°C	X R	X	X
65°C	X R,W	X	X
75°C	X W		
85°C	X W R	X	X R W

- UV Intensities (ND): 40, 60, 80 and 100 % of the sphere flux
- Wavelengths (WL): 306, 326, 354, 452 nm (band pass filters)
- No light

Indoor results are used to obtain parameters for SLP models.

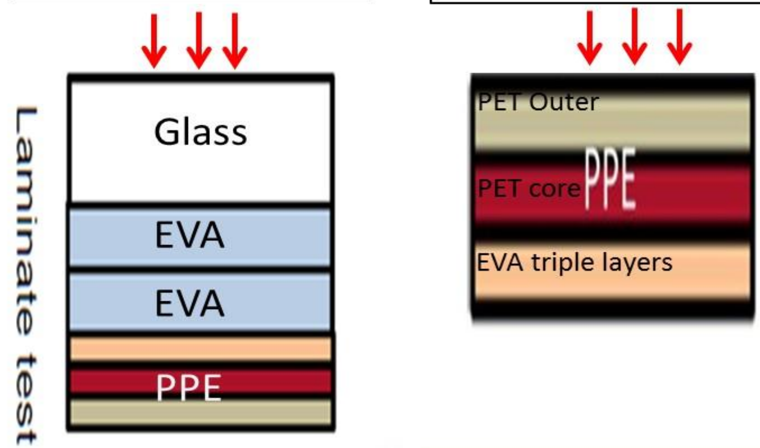
Samples and Materials

Glass/EVA/PPE Laminate

UV Through glass

PPE Free Film

Direct UV radiation on PET Outer Layer



- Yellowing Index (UV-vis; glass/EVA/PPE; PPE)
- Chemical Changes (ATR-FTIR; PPE) (Carboxylic Acid Formation, Chain Scission)

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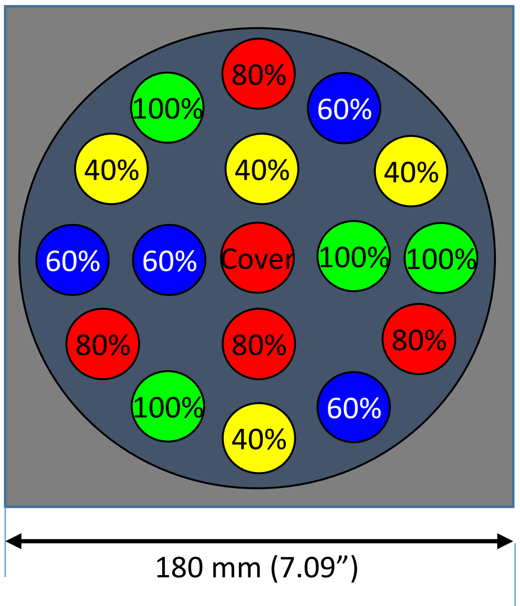
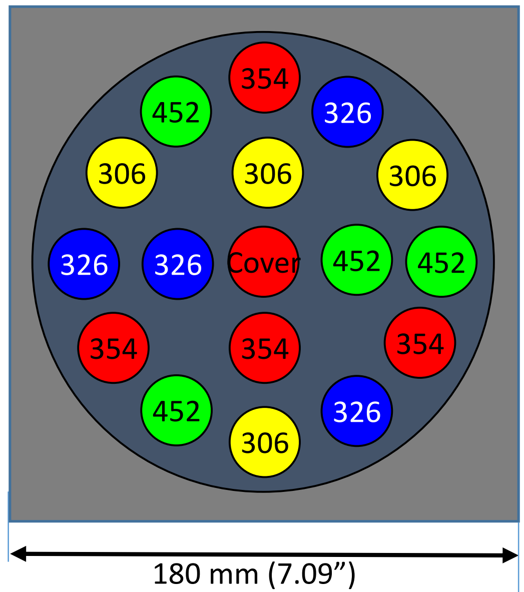
Using Filters to Vary Light Intensity and Wavelength

1) Reciprocity Study
Neutral Density (ND) filters



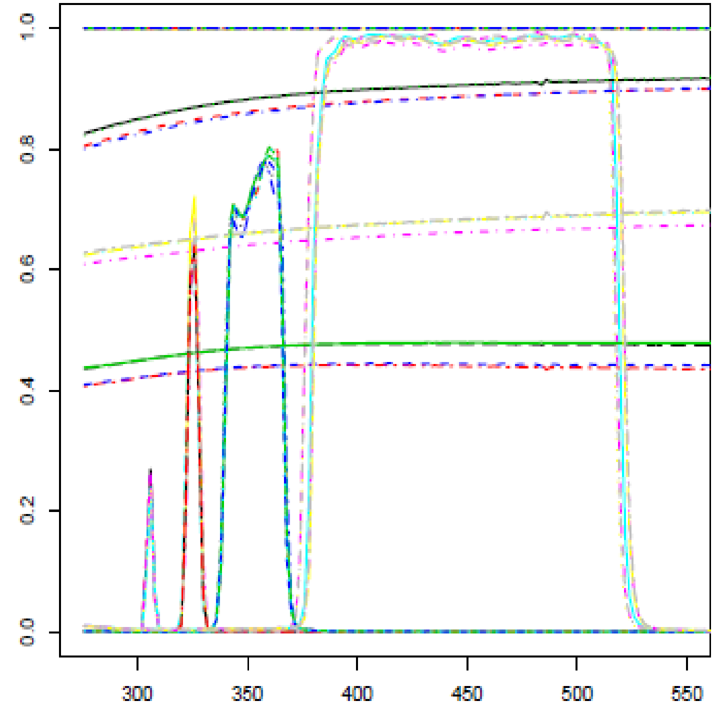
Varying light intensity with 40, 60, 80, and 100 % of ND filters
irradiance: 170 W/m^2 (300-400 nm)
$\rightarrow$ 68, 102, 136, 170 W/m^2

2) Wavelength Study
Band Pass (BS) filters



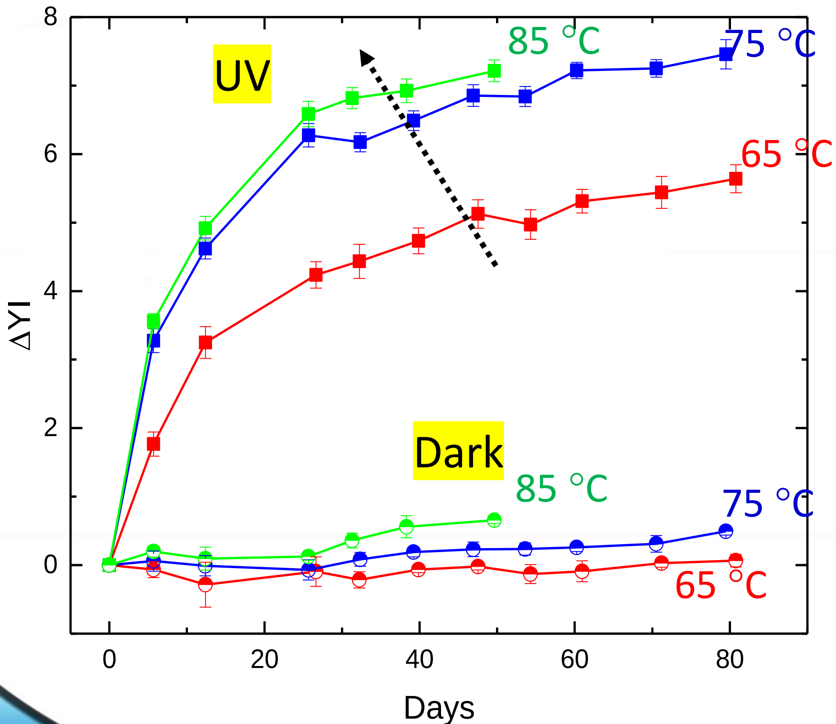
Varying wavelength range with narrow band pass filters
(306 $\pm$ 3) nm
(326 $\pm$ 6) nm
(354 $\pm$ 19) nm
(452 $\pm$ 80) nm.

UV-vis Transmission Spectra of ND and BS Filters

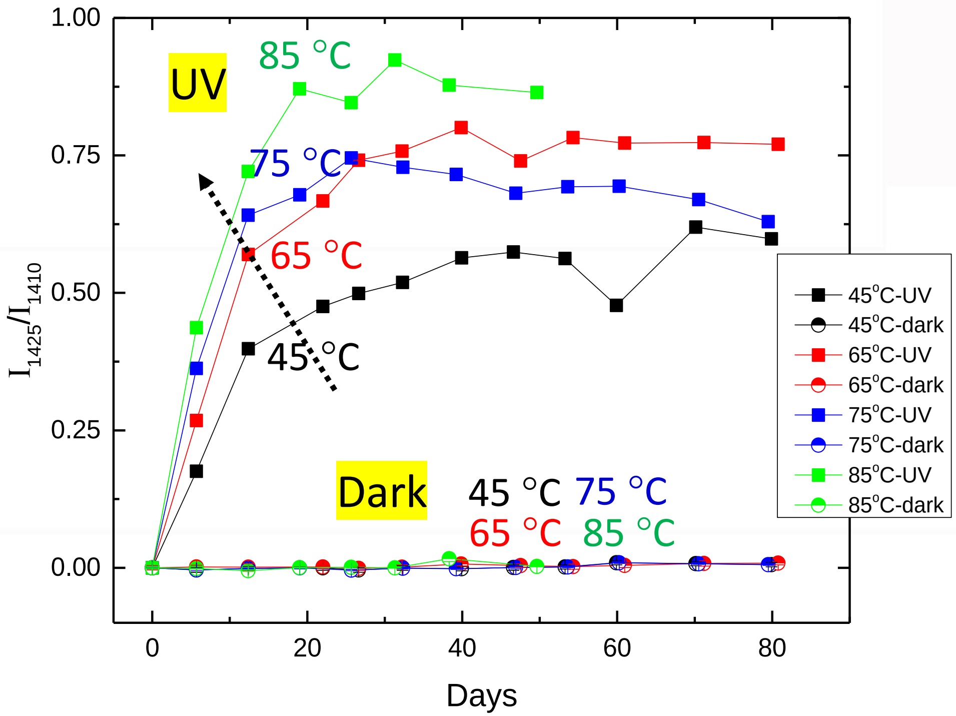


1. Synergistic Effect of UV and Temp on Yellowing and Chemical Degradation of PPE Films

Yellowing, UV/T vs. No UV/T, dry



Acid formation, UV/T vs. No UV/T, dry

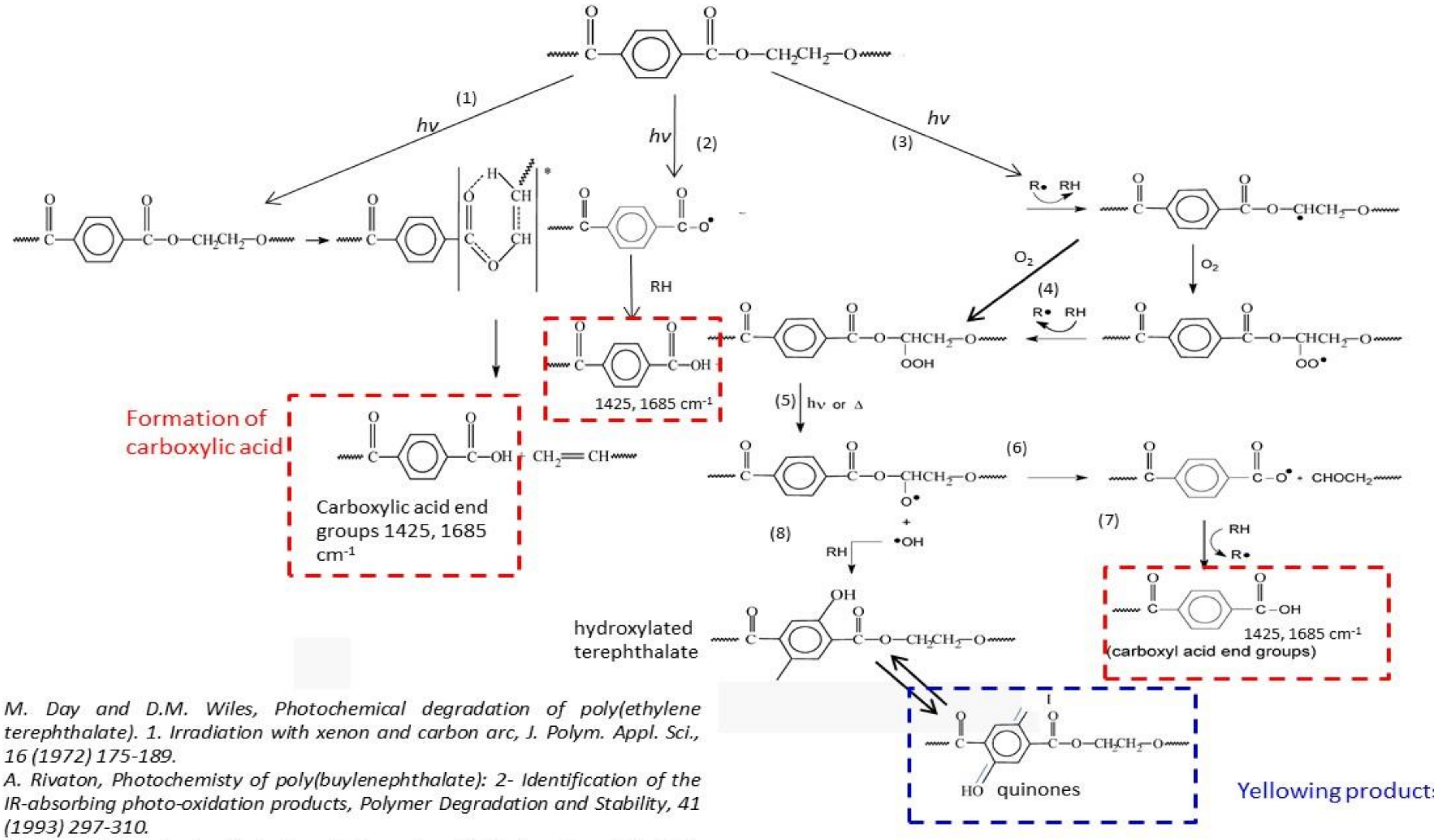


UV irradiance: 170 W/m²
(295-385 nm)

With UV,, Ea ≈ 14 KJ/m²
No UV, Ea ~ 120 KJ/m²
(Acid formation)

Proposed Reactions for Aromatic Acids Formation and Yellowing of PET Outer Layer Exposed to UV/T

engineering

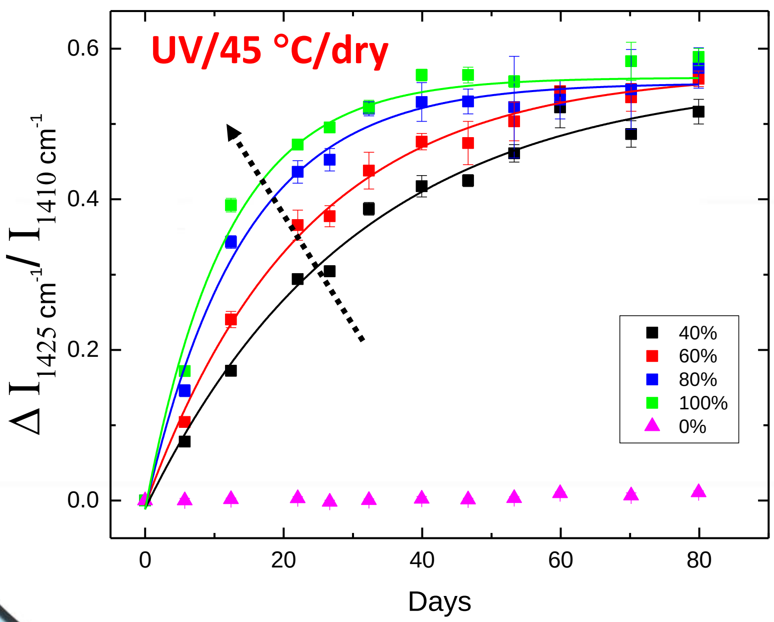


- M. Day and D.M. Wiles, Photochemical degradation of poly(ethylene terephthalate). 1. Irradiation with xenon and carbon arc, J. Polym. Appl. Sci., 16 (1972) 175-189.
- A. Rivaton, Photochemisty of poly(buylenephthalate): 2- Identification of the IR-absorbing photo-oxidation products, Polymer Degradation and Stability, 41 (1993) 297-310.
- T. Grossetete, et al., Photodegradation of poly(ethylene terephthalate)-modified copolymer, Polymer, 41 (2000) 3541-3554.

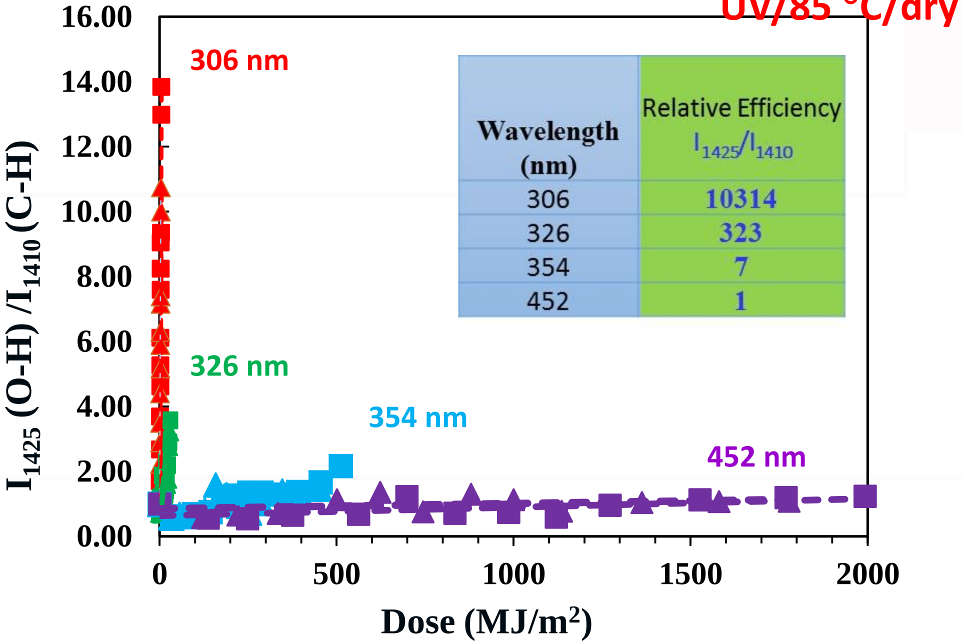
+ Photobleaching under O_2 and long wavelengths

Effects of Light Intensity and Wavelength on Chemical Degradation of PPE Films

Carboxylic Acid Formation



Carboxylic Acid, FTIR Peak Ratios
(I_{1425} (O-H) /I_{1410} (C-H))

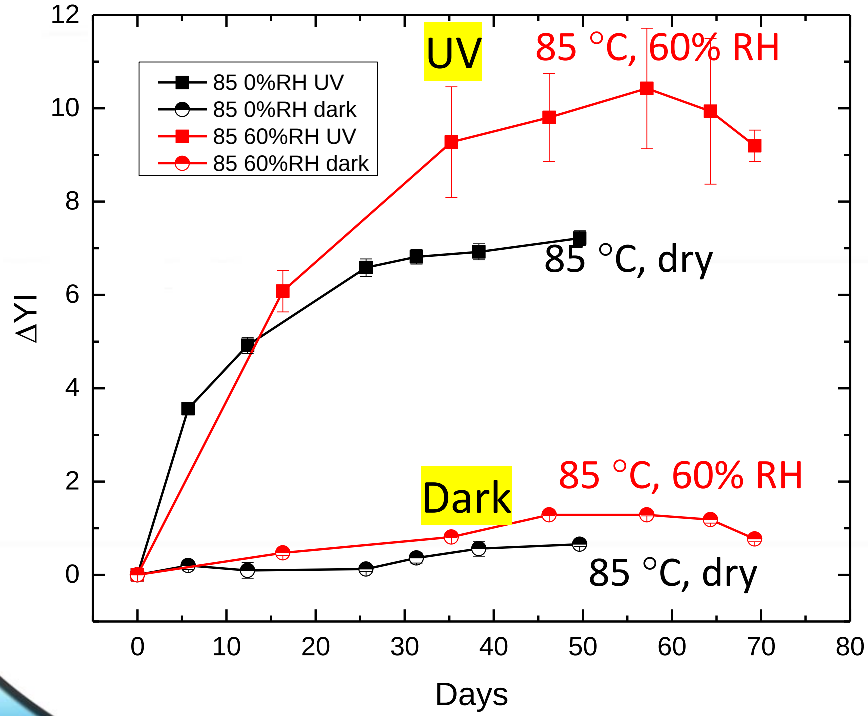


Wavelength (nm)	Relative Efficiency I_{1425}/I_{1410}
306	10314
326	323
354	7
452	1

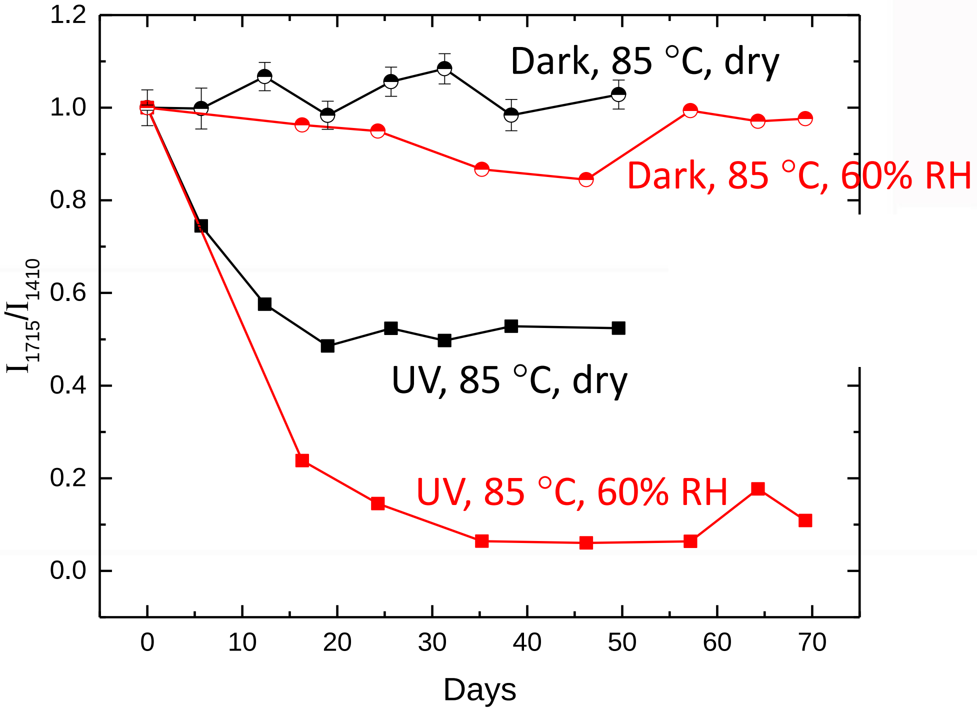
- ➢ Results have shown the effects of UV (intensity and wavelength) and temperate on PPE degradation.

2. Synergistic Effect of UV and RH on Chemical Degradation and Yellowing of PPE films

Yellowing, UV/RH vs. No UV/RH



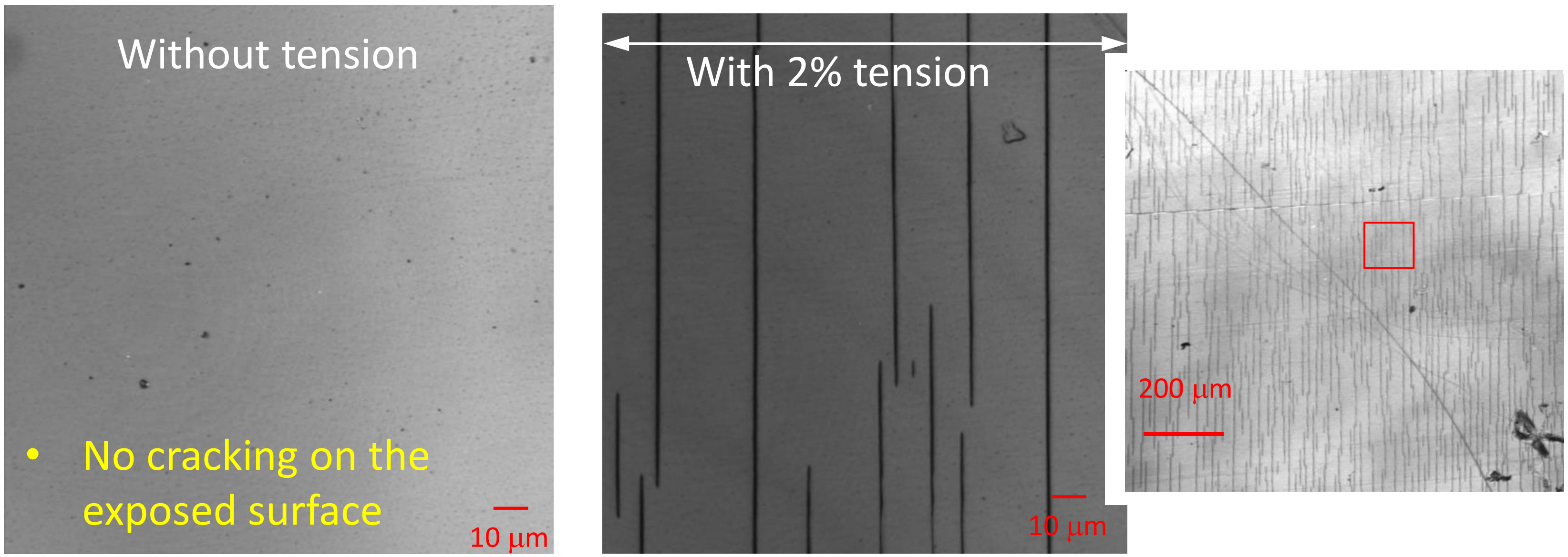
Chain Scission (Ester), UV/RH vs. No UV/RH



- ➢ In damp heat (85 °C/60% RH), PET degradation is not significant, but UV can accelerate the RH effect on PET degradation.

3. Synergistic Effect of UV and Mechanical Loading on Crack Formation of a PET Film

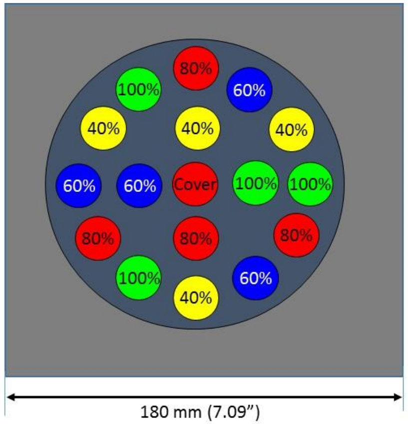
Exposure condition: 170 W/m^2 (300 nm- 400 nm), 24 °C for 20 days (50 % RH)



➢ Simultaneous mechanical loading under UV can accelerate the PET cracking.

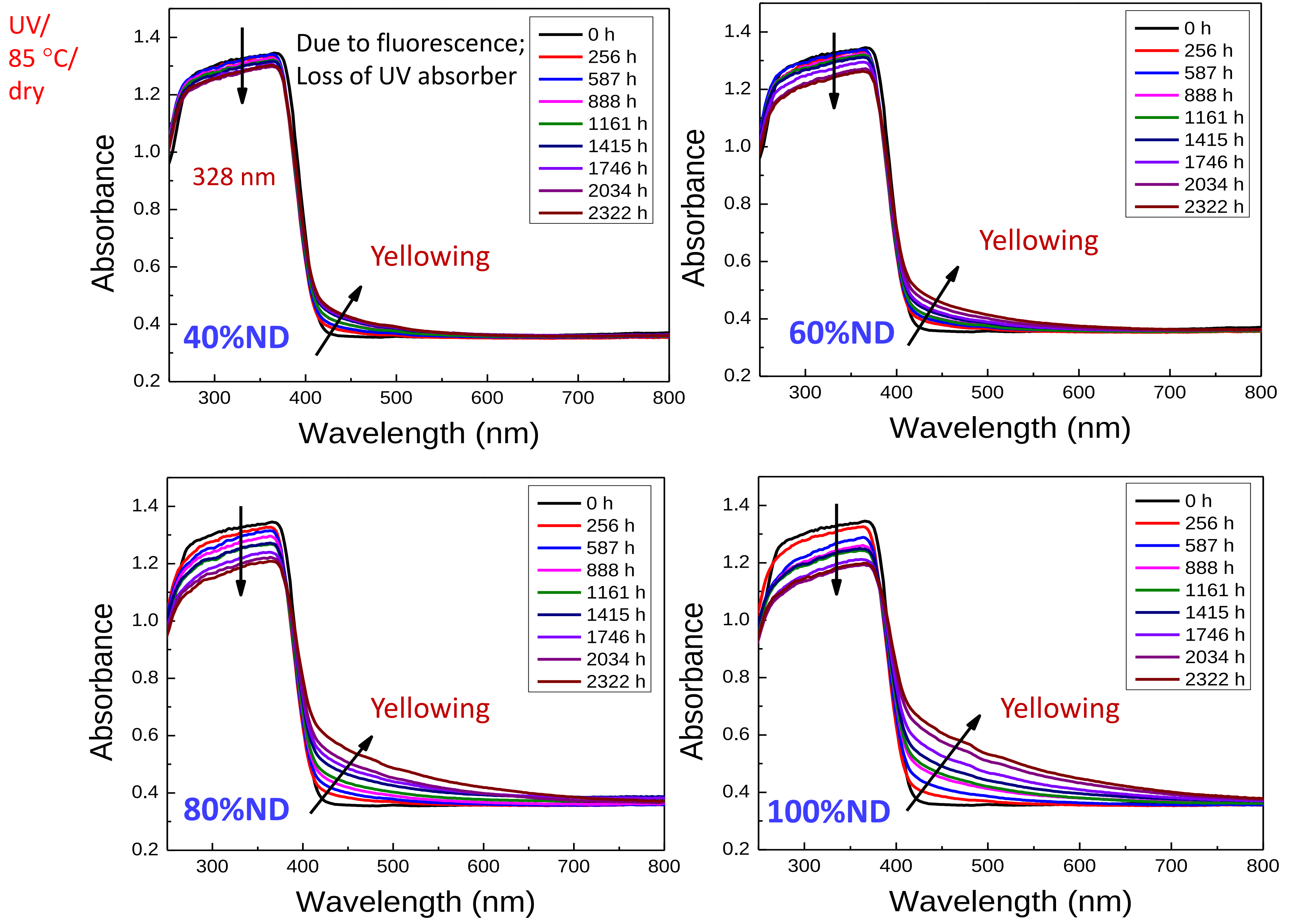
4. Influence of Temperature on Effect of Light Intensity (Reciprocity behavior) on Glass/EVA/PPE Yellowing

- UV/85 °C/dry
- UV/65 °C/dry
- UV/45 °C/dry



- For each temperature, 4 light intensity levels using 40%, 60%, 80%, 100% Neutral Density Filters (300-400 nm)
$\rightarrow$ 68, 102, 136, 170 W/m^2

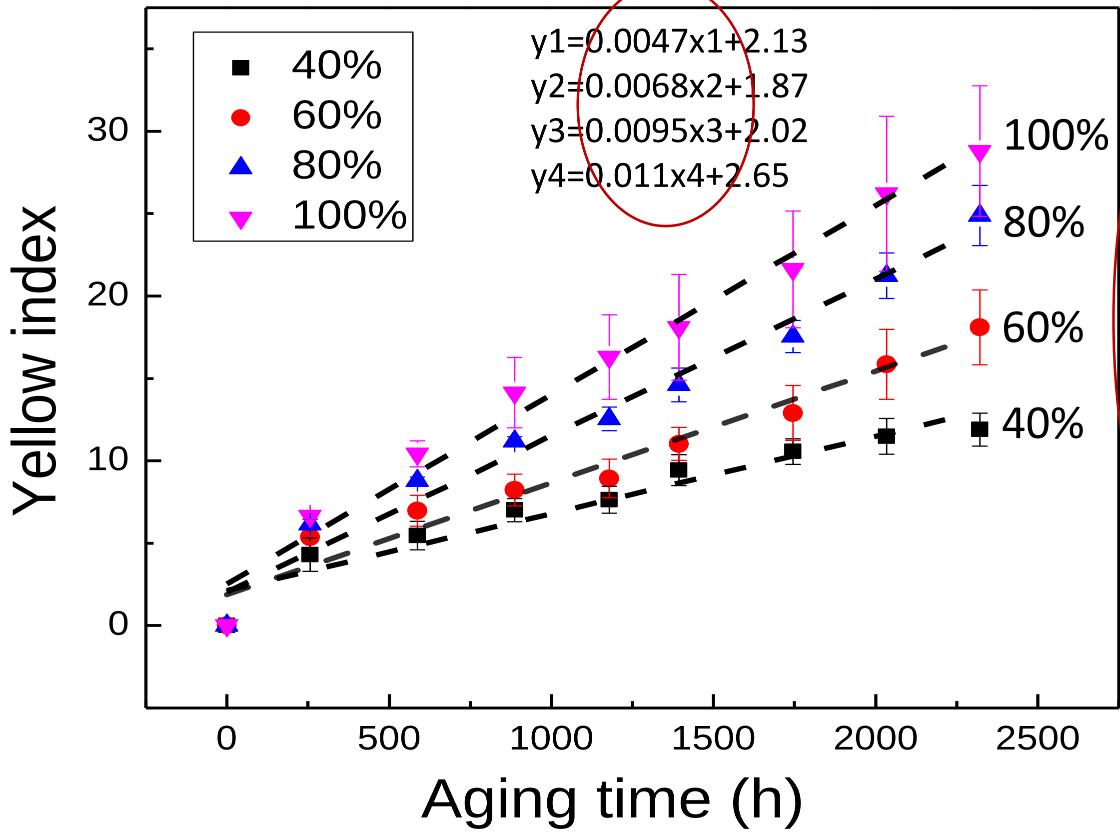
UV-Vis Spectra of Specimens at Different Exposure Time (Different UV Intensities)

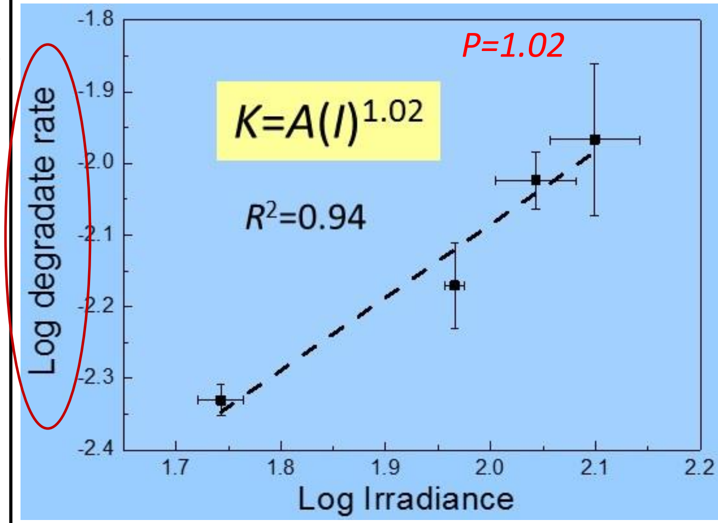


An increase in yellow index was observed with a higher light intensity.

Effect of Light Intensity on Yellowing Index vs. Exposure Time

UV/85 °C/dry



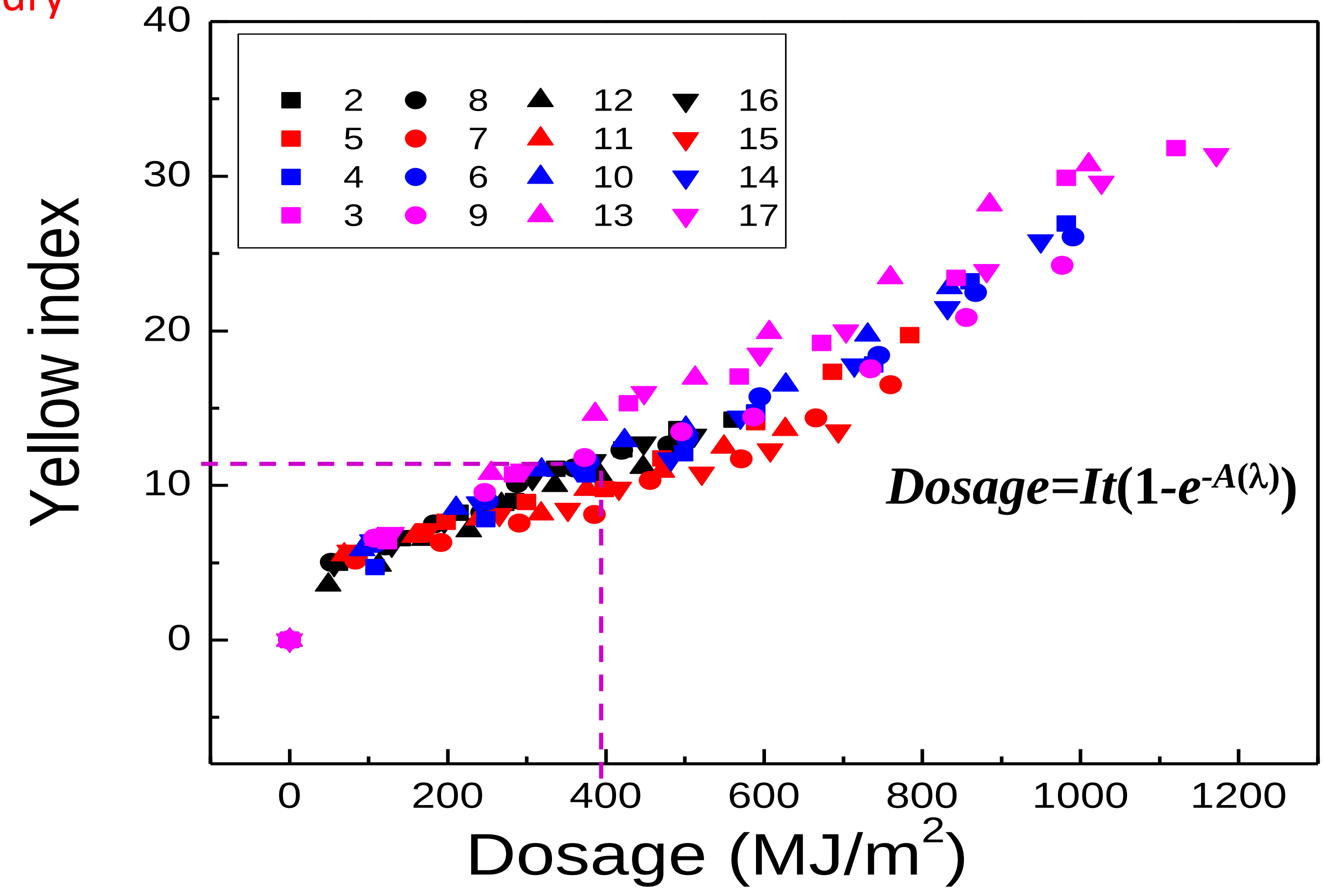


Reciprocity Law appeared to be obeyed.

1) A higher light intensity led to a faster yellowing growth.
2) A quasi-linear relationship was observed for the yellow index-aging time plot.

Yellowing Index as a Function of Dosage under Different UV Intensities

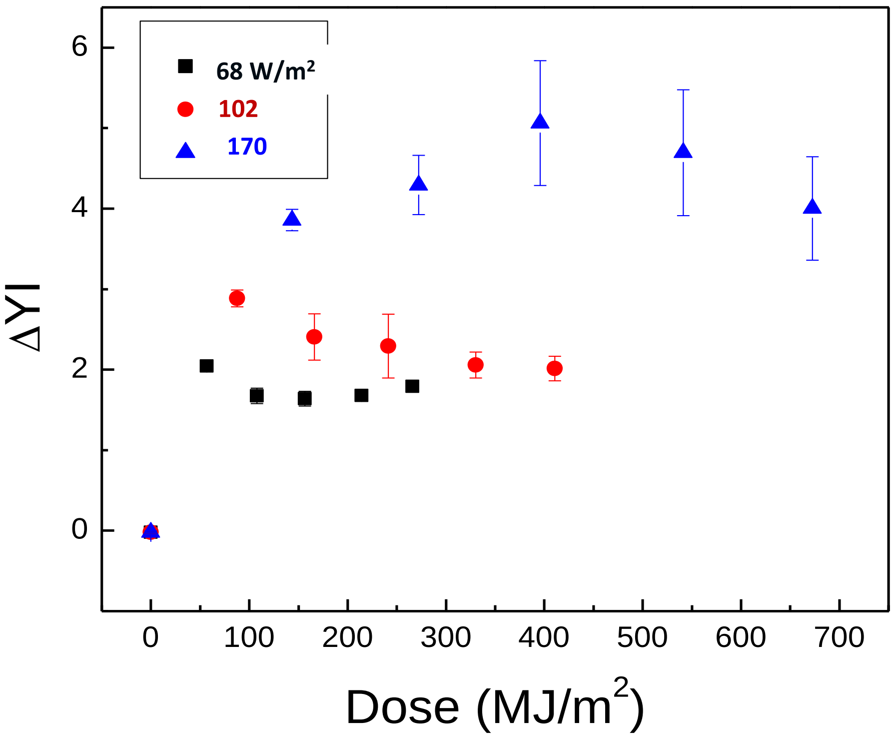
UV/85 °C/dry



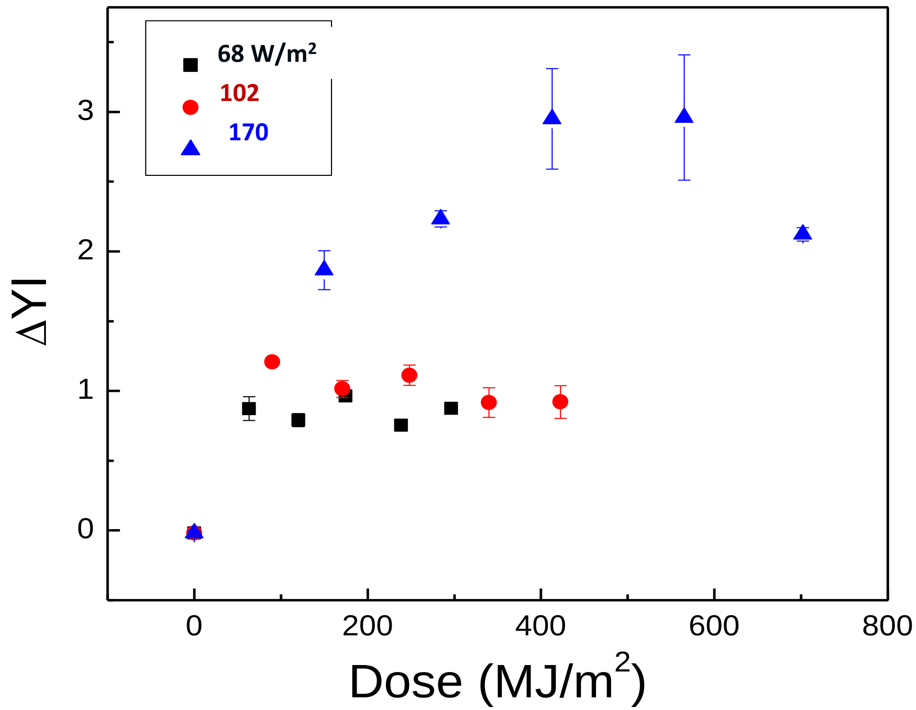
For a given damage, the required dosage is similar at different UV intensities.
– Reciprocity Law seems to be obeyed.

Effect of Light Intensity on Glass/EVA/PPE Yellowing at UV/65 °C/Dry and UV/45 °C/Dry

Δ YI vs. Dose, UV/65 °C



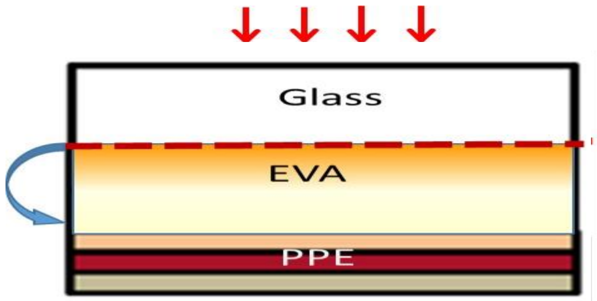
Δ YI vs. Dose, UV/45 °C

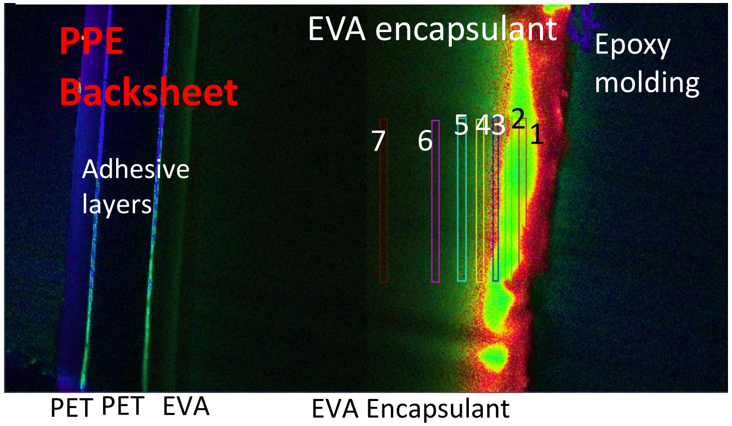


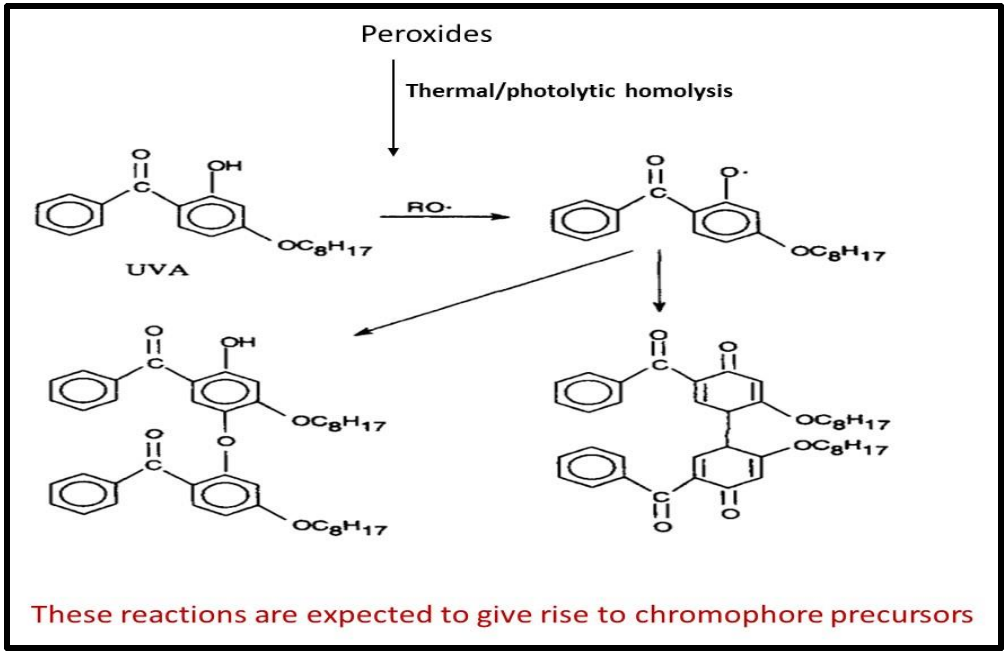
- **Reciprocity failure observed** for YI change under 65°C and 45 °C at UV/dry due to the dominant photobleaching.
- At lower temperature (< 65 °C), a higher light intensity can lead to an unrealistic yellowing that a lower light intensity can't achieve even the accumulated doses are the same.
- The intensity effect is influenced by the temperature, so the model for intensity effect should be adjusted by the temperature. $[F(\lambda)]^{p \cdot \exp(p_t \cdot \mathrm{TEMP})}$

Possible Discoloration Reactions of UV-exposed Glass/EVA/PPE

1. Additives reactions (UV absorbers, curing peroxide or other antioxidants) in the encapsulant Layer (primary reason)







2. Backsheet E-layer yellowing (little)
3. Photodegradation of EVA (little)
4. Photobleaching (depending on O_2 availability and long wavelength light)

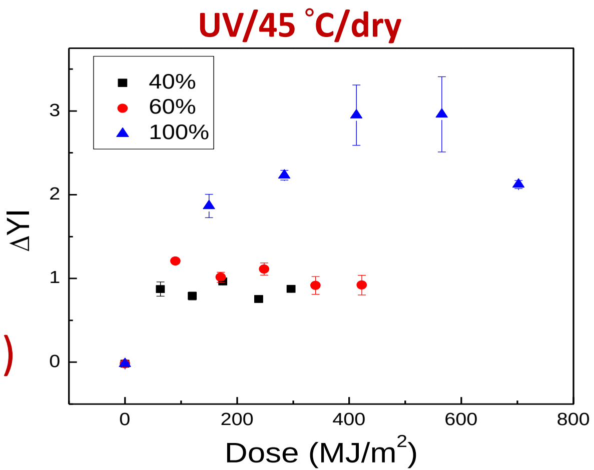


Photo-yellowing Vs. Photo-bleaching

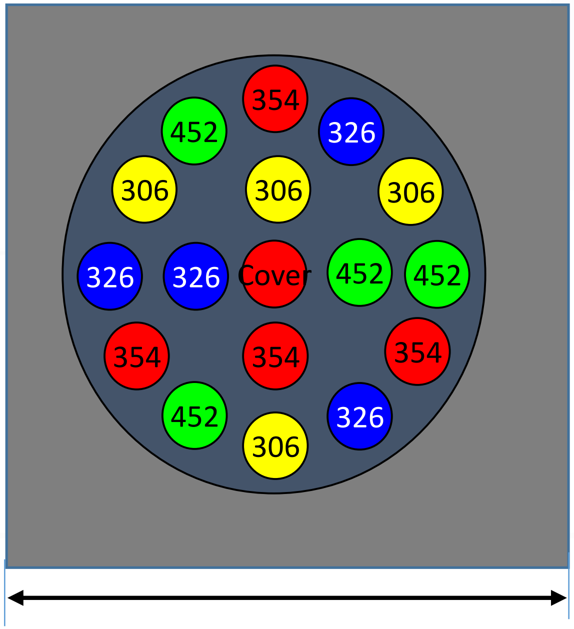
At lower temperatures (<65°C), photobleaching seems to be competitive with photoyellowing.

At higher temperatures, photoyellowing is more dominant.

4. Influence of Temperature on Effect of Wavelength on Glass/EVA/PPE and PPE Degradation

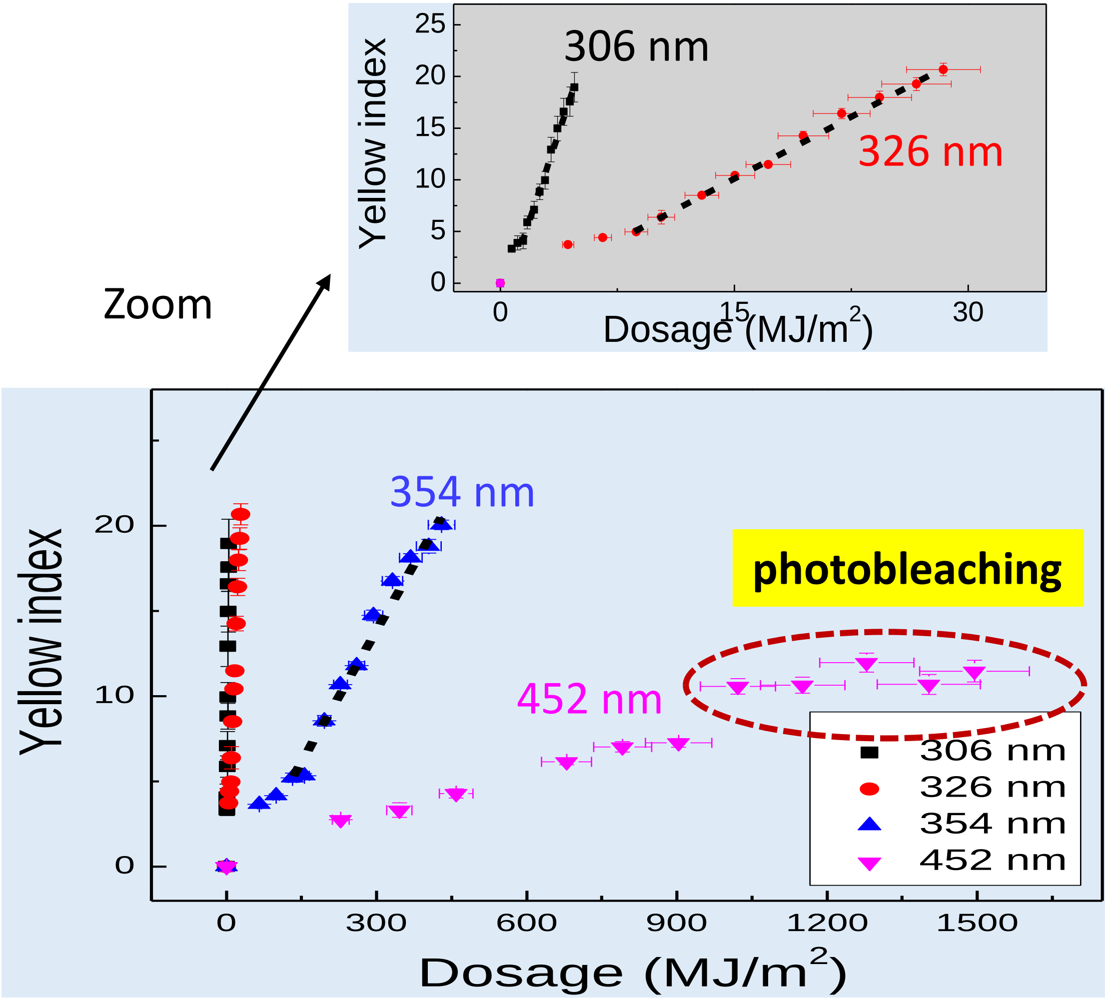
- UV/85 °C/dry
- UV/65 °C/dry
- UV/45 °C/dry

- **4 Wavelength ranges:**
  (306 ± 3) nm
  (326 ± 6) nm
  (354 ± 19) nm
  (452 ± 80) nm



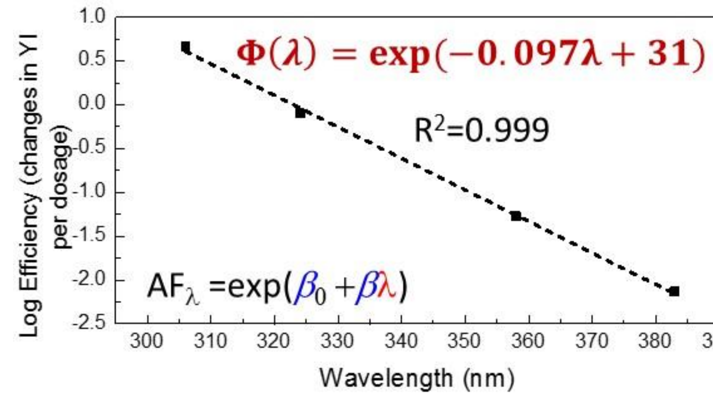
Averaged Yellow Index vs. Dose For Glass/EVA/PPE at Different Wavelengths

At UV/85°C/dry



Wavelength (nm)	Pseudo-rate constant (m^2/MJ)	Relative Efficiency 1
306	4.56	580.9
326 (324)	0.80	101.9
354 (358)	0.053	6.7
452 (383)	0.0079	1

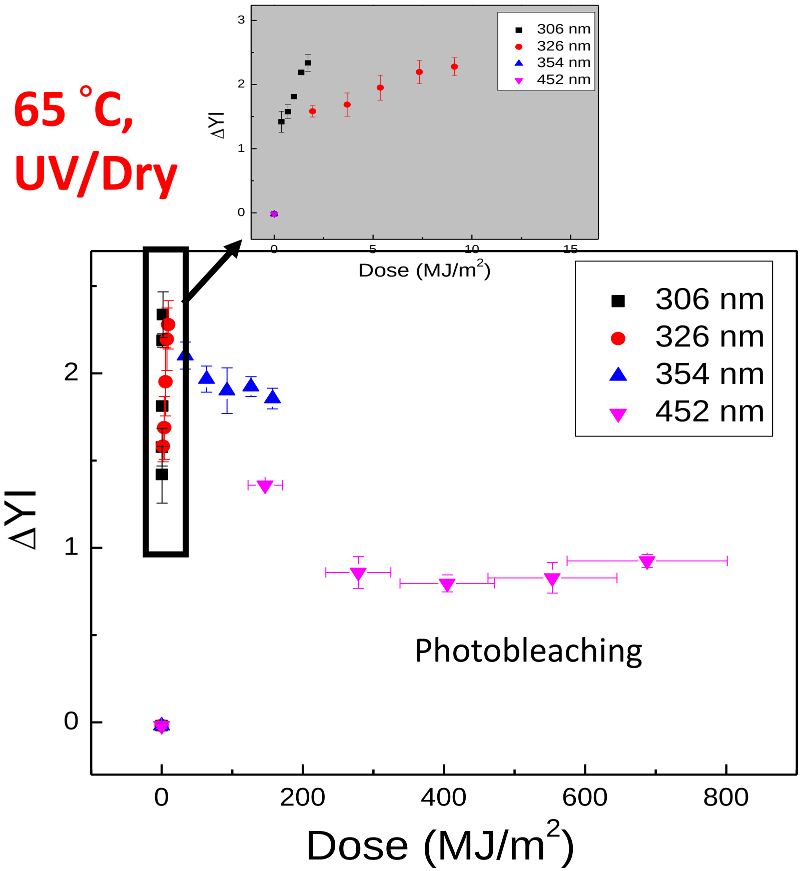
Action Spectrum for increase in YI

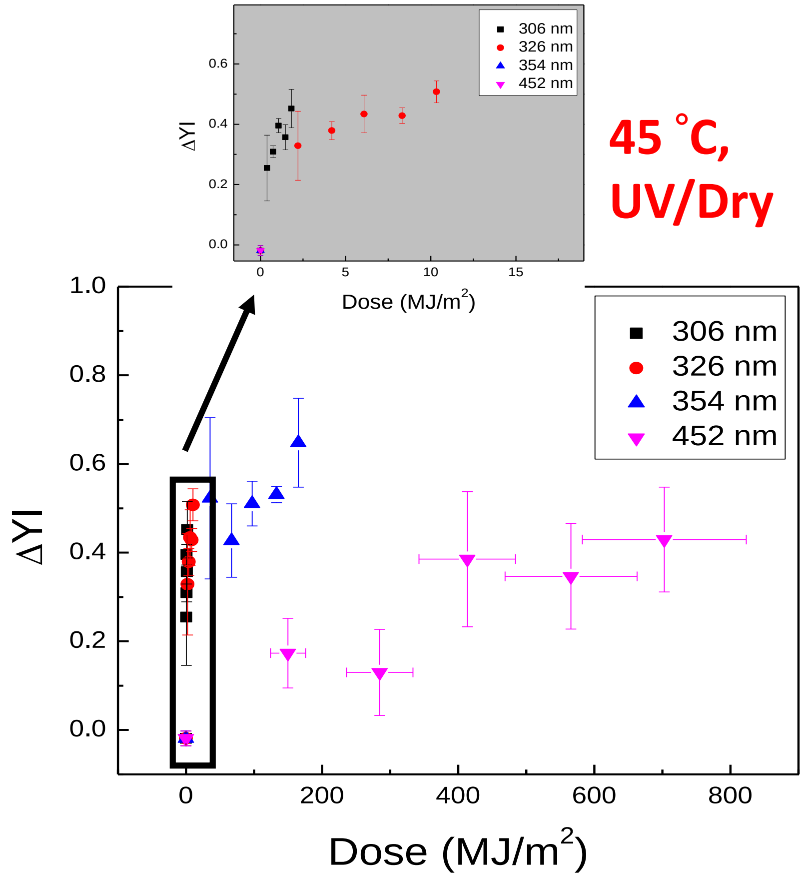


- ✓ Shorter wavelength led to a higher yellow growth at same dosage.
- ✓ Increase of YI under 452 nm slowed down and then fluctuated at late stage, indicating **photobleaching appeared at longer wavelength (372 nm - 532 nm).**
- ➢ An exponential dependence between yellowing and wavelength was obtained.

Effect of Wavelength on Yellowing Index of Glass/EVA/PPE at Lower Temperatures

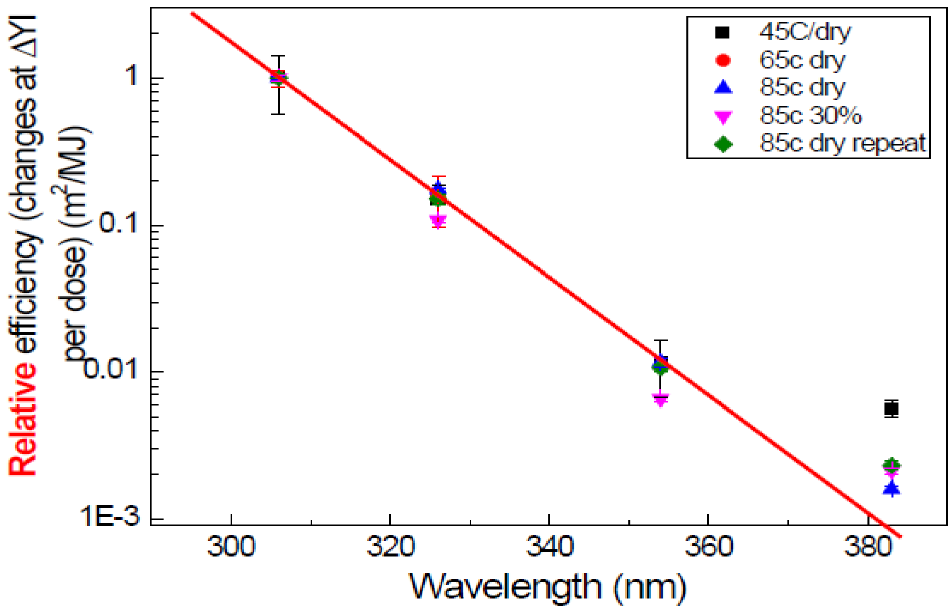
65 °C, UV/Dry





- Monotonous increase in YI was observed under 306 nm and 326 nm, while **photobleaching effects were observed under 354 nm and 452 nm.**
- The yellowing efficiencies for 306 nm and 326 nm became much lower at 65 °C and 45 °C than those at 85 °C, leading to low photoyellowing due to competitive photobleaching at lower temperatures.

Effect of Wavelength on Yellowing Index of Glass/EVA/PPE at Different Temperatures including Longer Wavelengths



- Wavelength effect is modeled by log linear plus some adjustment for **photobleaching effect**

$$\phi(\lambda) = \exp[\beta(\lambda - 354)] + \exp(\beta_0 + \beta_{0t} \cdot \mathrm{TEMP})$$

**Yili Hong, "Statistical Modeling for SLP of PV Materials and Laminates", 4th Atlas-NIST Workshop on PV Materials Durability, Dec. 6, 2017*

Summary

- The multiple environmental stresses can have interdependent effects on PV polymer degradation. Therefore, simultaneous/combined tests with appropriate conditions that don't alter degradation mechanisms compared to field should be considered when designing accelerated laboratory testing.

- The SLP modeling also needs to consider the complex multi-stress effects, e.g., the influences of temperature on other stress effects above or below phase transition temperature could be different.