

IMPROVING THE SPRAY CYCLE FOR XENON ARC ACCELERATED WEATHERING

Derek Yeager, Olga Kuvshinnikova, James E. Pickett, and Kevin Betts 2023 ASTM Workshop, Denver, Colorado; June 28, 2023

GENERAL BUSINESS USE

MOTIVATION

- Popular 102 min. light / 18 min. light + spray cycle has origins in 1920's and 1930's
 - ASTM G155 Cycle 1; ISO 4892-2
 - See 2022 Atlas -mts.com blog post or Sunspots, 1, 4 (1972) for some history
 - Other common cycles also have short, frequent sprays and sprays during light period
- Inadequate for some classes of coatings → development of ASTM D7869
- Also inadequate for eroding plastics
 - Gentle spray is not like rain
 - Does not adequately wash stable materials, especially carbon black, from surface
 - Very non -predictive for some materials that perform well outdoors
- Previously found more predictive spray/wash protocols, but non -standard and not generally used*
- Can better conditions be found that are a small perturbation on current practices?
 - Different nozzles; less frequent but longer spray periods in dark; regular sponge washes

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THE EXPERIMENTS

Samples

- 20 polycarbonate and PC copolymer samples in colors (9) and in blacks (11)
- Most have good gloss retention outdoors, but poor in xenon arc testing
- All samples with 24 months outdoor exposure at a Miami area test site with monthly washing

Conditions

- Ci4400 xenon arc Weather -ometer ®
- Right Light [®] /CIRA-quartz filters; 0.75 W/m ²/nm at 340 nm
- 23 h light: 63 ° C black panel, 40 °C chamber, 50% RH
- 1 h dark spray: with either standard #3 misting nozzle or fan nozzle * (higher volume; larger drop size)
- Unwashed or washed with sponge and DI water 10 strokes every 1 MJ of exposure (~ 370 h)
- Ci4000 running modified
 ISO 4892 -2 under same conditions with 102 min light / 18 min light + spray cycle
- At different site; unwashed and sponge washed at 1 MJ intervals
- "Mist", "Fan", "ISO" sets, Unwashed or Washed



DATA ANALYSIS: CORRELATION TO FLORIDA

- ➤ Assume 3 MJ/m ²/nm at 340 nm of Right Light -filtered xenon arc ≅ 12 months of Miami, Florida exposure
 - Based on theory as well as empirical data on many aromatic engineering thermoplastics*
- Florida data at 6, 12, 18, and 24 mo. for these materials
- Analysis for 24 mo. predictions gave similar results as using all intermediate times

*References:

- O. Kuvshinnikova, G. Boven, J.E. Pickett, Weathering of aromatic engineering thermoplastics: Comparison of outdoor and xenon arc exposures, *Polym. Deg. Stabil.*, **160**, 177-194 (2019).
- O. Kuvshinnikova, G. Boven, J.E. Pickett, Service life prediction of aromatic engineering thermoplastics: Assessing the quality of predictions from xenon arc exposures, In: C.C. White, M.M. Nichols, and J.E Pickett (Eds.), Service Life Prediction of Polymers and Coatings: Enhanced Methods, Elsevier, Cambridge, Mass., 2020, pp. 163, -182.
- J.E. Pickett, Weathering of Plastics, In: Handbook of Environmental Degradation of Materials (Third Edition), M. Kutz, Ed., Elsevier, 2018, pp. 163-184.



SUMMARY OF FINDINGS

- > 23 h light / 1 h dark spray a little better than 102/18 cycle, on average
- Nozzle type did not make much difference
 - fan spray slightly better than mist, but not much
- ➤ Washing helped considerably, but not so much with 102/18 cycle
 - frequent spray in light seems to "burn in" damage that washing cannot fully mitigate

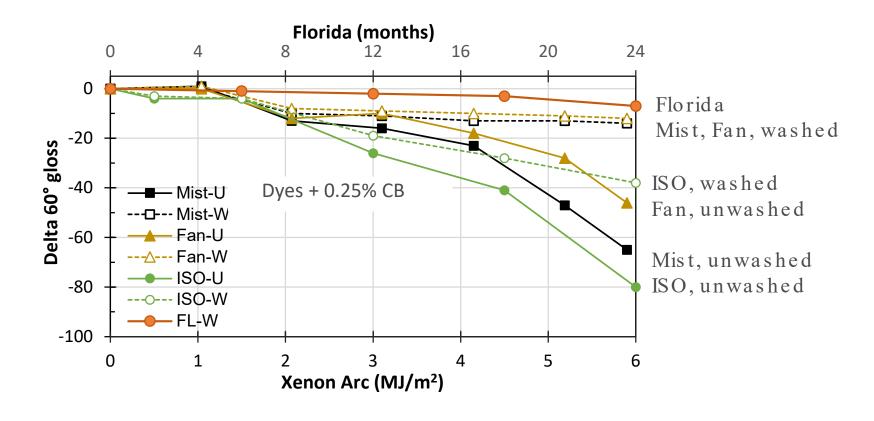


Improved correlation is possible with minor modifications of existing practices!





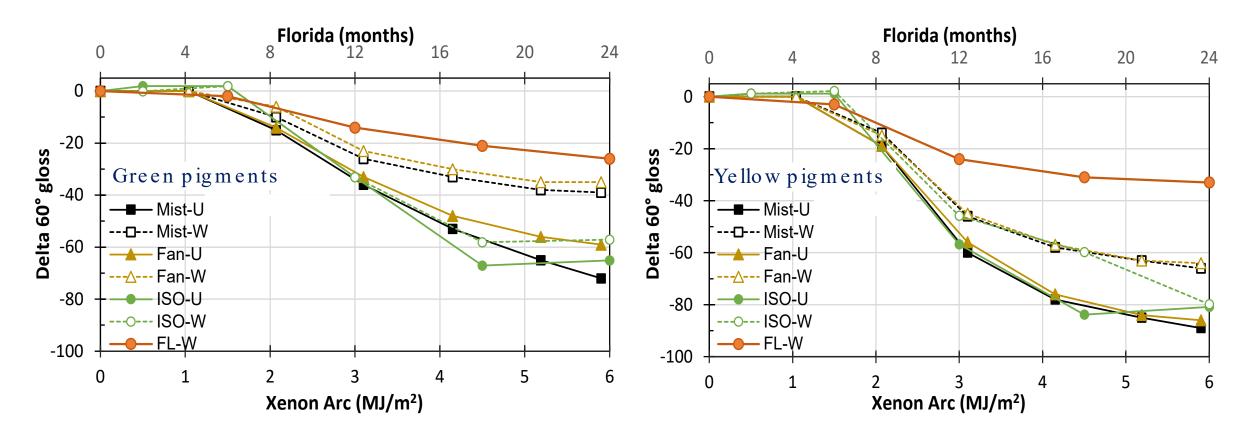
> PC copolymer with dyes + carbon black







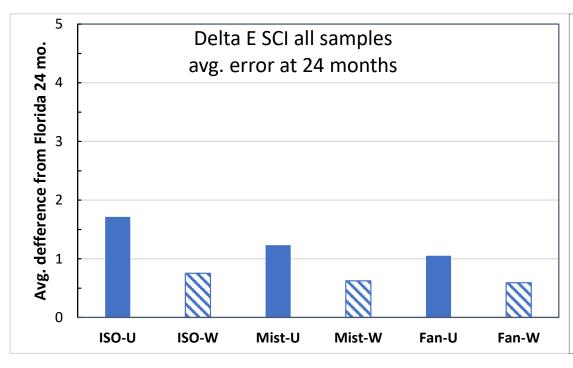
> PC copolymer with pigments: successful and less successful predictions

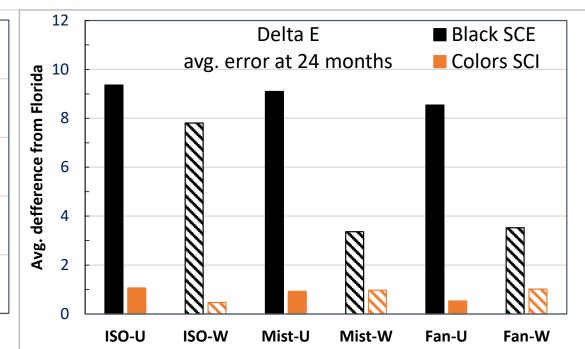




DATA ANALYSIS: COLOR SHIFTS

Compare 6 MJ/m ² xenon arc prediction to 24 mo. Florida data
 analysis including 6, 12, 18 -month data gave very similar results

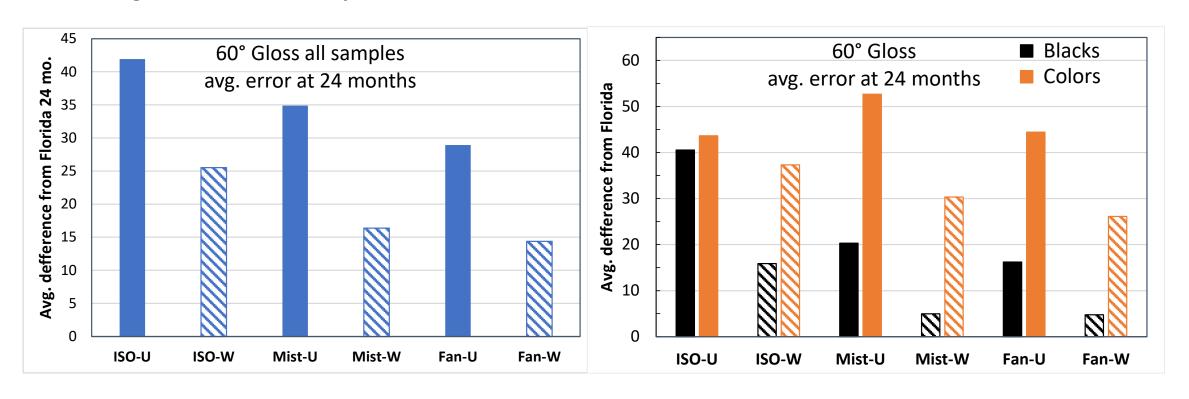






DATA ANALYSIS: GLOSS LOSS

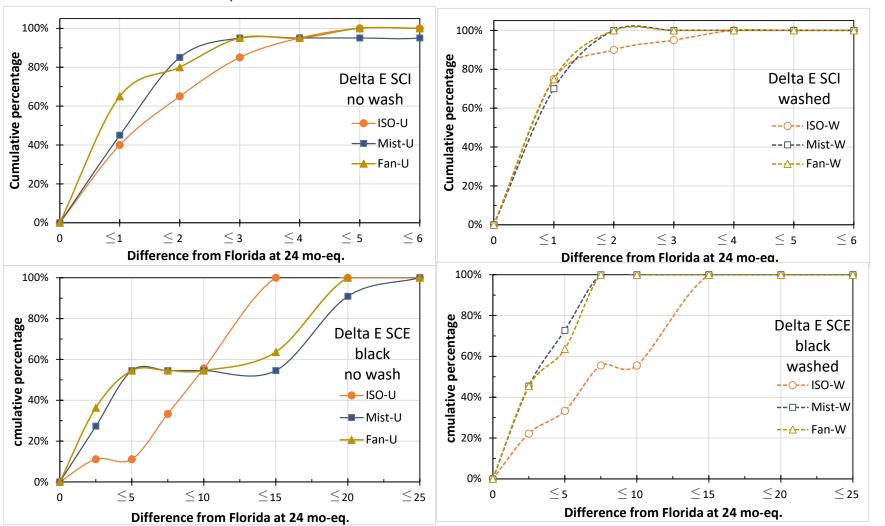
- ➤ Compare 6 MJ/m ² xenon arc prediction to 24 mo. Florida data
 - analysis including 6, 12, 18 -month data gave very similar results
 - 20 ©gloss data showed very similar results





HISTOGRAM OF DIFFERENCES: COLOR SHIFT

➤ Compare 6 MJ/m ² xenon arc prediction to 24 mo. Florida data



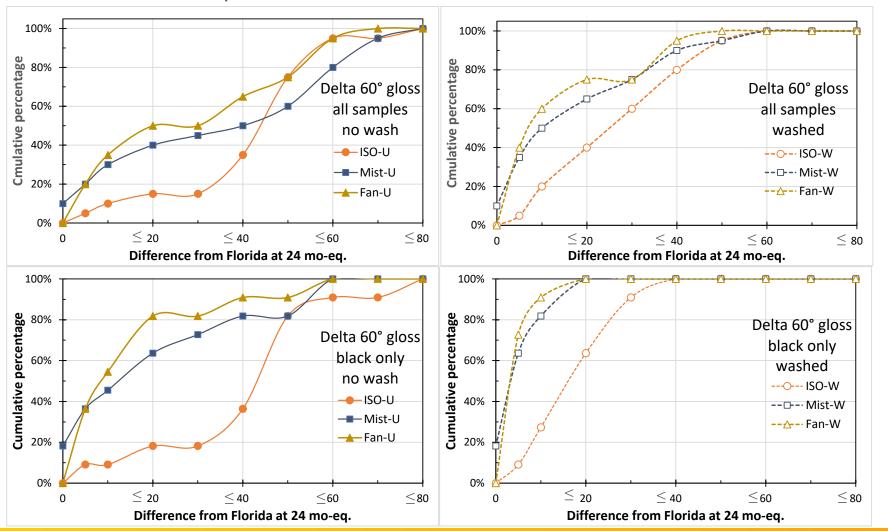
Color shift (SCI) well-predicted under all conditions

Black color shift (SCE) better predicted with new cycle + wash



HISTOGRAM OF DIFFERENCES: 60 DEG GLOSS LOSS

➤ Compare 6 MJ/m ² xenon arc prediction to 24 mo. Florida data



Gloss loss better predicted with new cycle + wash

Black gloss loss much better predicted with new cycle + wash

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CONCLUSIONS

- > Traditional 102/18 cycle is not very predictive for this class of materials, especially for black
- Less frequent/longer duration spray cycle better: 23 h light / 1 h dark + spray
- ➤ More aggressive spray nozzle gives only slight improvement
- ➤ Sponge washing at 1 MJ/m ² intervals (~370 h; ~ 15 days) helps greatly
 - especially for samples with carbon black
 - variable for samples with larger particle size pigments
 → still need some improvement in washing protocol
 - helps less for 102/18 cycle than for modified cycle
- Now checking ASTM D7869 cycle with and without washing
- Washing is now permitted in ASTM G155, with agreement

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ABSTRACT

Many common xenon are accelerated weathering protocols call for frequent water sprays, often during light periods. The traditional 102 min. light / 18 min. light + spray cycle in ASTM G155 Cycle 1 and in ISO 4892-2 has its origins to cycles dating back nearly a hundred years with no real empirical justification. ASTM D7869 was developed with an improved spray cycle specifically for transportation coatings. However, the gentle, low volume sprays are unlike rain, and stable materials such as pigments (especially carbon black) can remain adhered to the surface as a polymer surface erodes during weathering. Data show that such residue does not accumulate under natural weathering conditions, even in the desert, presumably due to the washing effects of rain and perhaps wind. We have investigated cycles with less frequent but longer duration sprays in the dark, standard and more aggressive nozzles, and with approx. biweekly sponge washing on a series of samples that generally perform well outdoors but poorly under standard weathering protocols. Color shift and gloss loss were compared with Florida data assuming a correlation of ~ 3 MJ/m²/nm at 340 nm of Daylight Type Ixenon arc exposure per year of Florida exposure. Color shifts of nonblack colors were well-predicted under all conditions. Changing the spray cycle generally improved the predictability of gloss retention and color shifts of black samples. Adding the washing protocol improved the predictability much more, especially for black samples, but adding washing to the traditional 102/18 cycle resulted in much smaller improvements. The more aggressive spray nozzle was only slightly better than the standard misting spray nozzle. The results suggest that further development of the washing protocol is needed to improve predictability of gloss retention for samples containing large particle size pigments, but that a modified protocol within the scope of ASTM G155-21 and existing equipment can give much more predictive results for materials that undergo erosion during weathering.



Method to Accelerate

UV-Visible Light-Induced

Composite Degradation under Paint

Dr. Karen A. Schultz

ASTM Workshop on Weathering and Durability Testing 2023

June 28, 2023

Photooxidative Degradation of Epoxy-based Composites

- Polymers are susceptible to degradation initiated by ultraviolet (UV) and visible light
 - Complex set of chemical reactions involving the combined effect of light energy and oxygen
 - Chain scissions and/or crosslinking are initiated by bond dissociation upon absorption of radiation
 - Double bonds and aromatic rings are more electron donating; they can more easily take up the energy of any photon → more photosensitive

$$H_{2}\overset{O}{C}-CH-CH_{3}-O + \overset{CH_{3}}{C}-CH-CH_{2} & \overset{C}{O}+CH_{2}-CH-CH_{2} & \overset{O}{O}+CH_{2}-CH-CH_{2} & \overset{O}{C}+CH_{3} & \overset{O}{C}+CH_{2}-CH-CH_{2} & \overset{O}{C}+CH_{3} & \overset{O}{C}+CH_{3} & \overset{O}{C}+CH_{2}-CH-CH_{2} & \overset{O}{C}+CH_{3} & \overset{O}{C}+CH_{3}$$

Strategies to Protect Composites from Light

- Structural property requirements for aerospace applications typically prevent usage of aliphatic backbones (less susceptible to photooxidative degradation)
- UV absorbers can slow the rate of degradation of the resin system, but not change the inherent sensitivity to photochemical reactions

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Light needs to be blocked from reaching composite surfaces

- In manufacturing environments, light sources are chosen to reduce UV wavelengths and parts are covered when possible to limit light exposure.
- In service environments, coatings limit light exposure.

Testing Challenge: How to Accelerate Composite Degradation under Paint

- Coatings are used to protect exterior composite parts from weathering in service
 - Light transmission through aerospace coating systems is very low
 - Long exposures, even at low levels, can lead to composite degradation and coating loss
- How can we ensure a coating system will adequately protect the underlying composite structure from photooxidative degradation?

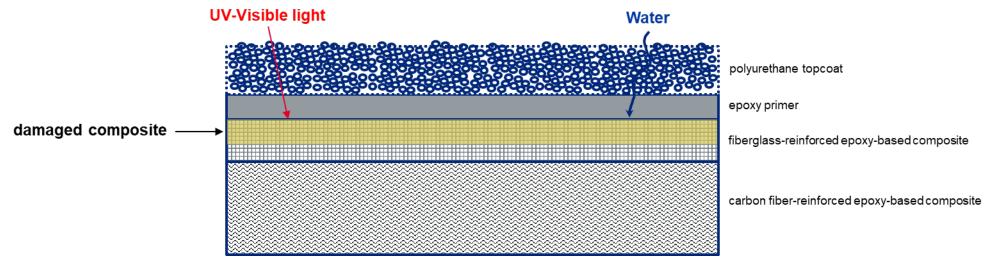
 Real-time outdoor weathering takes too long for efficient development cycles



Approach

- Start with well developed weathering protocol and existing equipment
- Modify parameters to move focus from coating degradation (gloss loss and color shift) to composite degradation under paint
- Light exposure
 - Photooxidative degradation failure mechanism
 - → High intensity, as long as possible

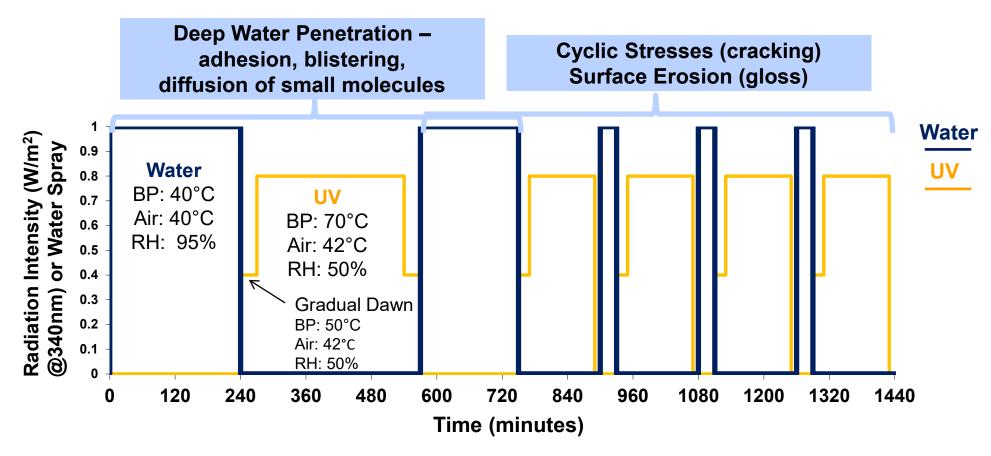
- Moisture exposure
 - What effect does moisture have on failure mechanism and time-to-failure?



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Starting Point: ASTM D7869



Key elements:

- Light filter matches sunlight spectral power distribution (SPD) → correctly reproduces chemical degradation
- ullet High irradiance during light cycle ightarrow accelerated light exposure
- Long water soak for deep water penetration → promotes delamination, blistering, diffusion of small molecules
- \blacksquare Light-moisture cycles \rightarrow promotes cracking, surface erosion, gloss loss

New Protocol Development – Light Exposure

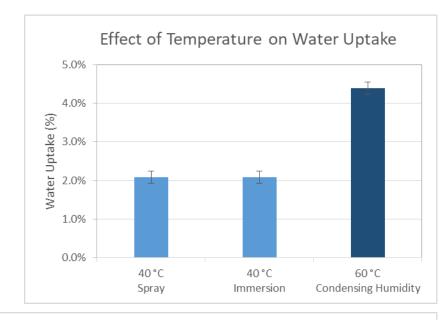
- Simulated sunlight
 - Atlas Ci4000 Weather-Ometer[®]
 - Right Light™ filter
- Irradiance considerations
 - Balance light exposure, lamp power, and temperature
 - 0.7 W/m² irradiance at 340 nm
 - Lamp Power: 3.6-4.0 kW
 - Chamber Temperature: 40-45 °C
 - Black Panel Temperature: 65-75 °C

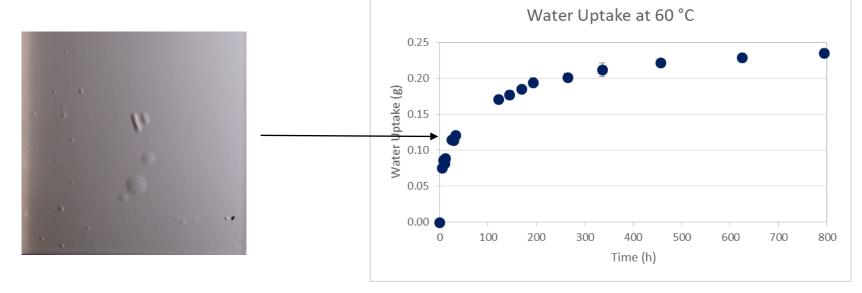


Image courtesy of Atlas

New Protocol Development – Role of Moisture

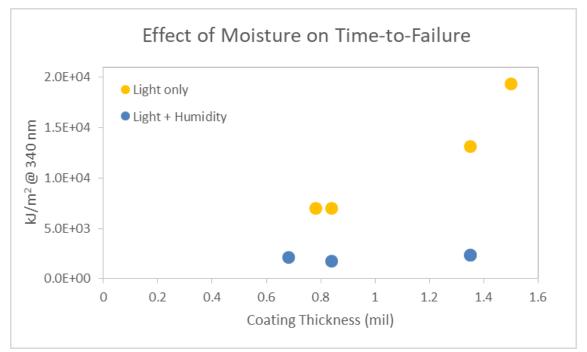
- What temperature?
 - 40 °C (spray or soak) vs. 60 °C (condensing humidity)
- How long does the moisture exposure need to be?
 - Approximately 50% water uptake reached in first 24 hours
 - Blisters formed at composite-coating interface by 24 hours
 - Indication that interface was wetted, though not saturated

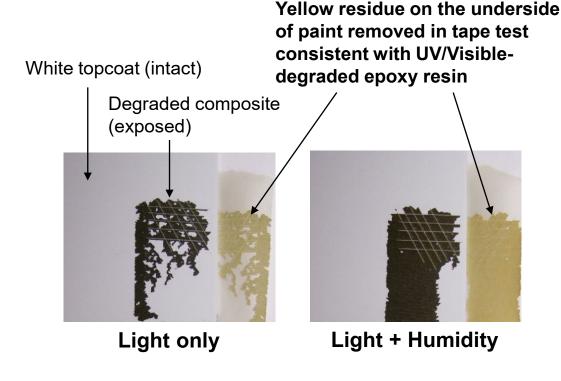




New Protocol Development – Role of Moisture

- Is humidity exposure necessary to produce failures?
 - Same composite degradation and coating loss observed for Light only and Light + Humidity exposures
- Is humidity exposure beneficial to accelerating failures?
 - Time spent in condensing humidity chamber is at expense of light exposure
 - 4-5x faster failure with moisture





Cyclic Exposure Protocol

Balance:

- Light exposure
 - High intensity
 - As much time as possible
- Moisture exposure
 - Elevate temperature to increase amount and rate of water uptake
 - Wet coating-substrate interface as quickly as possible
- Practical lab schedule
 - Test panels moved manually between chambers

Weekly Protocol

- 4 days light exposure (UV + Visible)
- 1 day condensing humidity (60 °C)
- 1 day light exposure (UV + Visible)
- 1 day condensing humidity (60 °C)

Light				Humidity	Light	Humidity
1	2	3	4	5	6	7

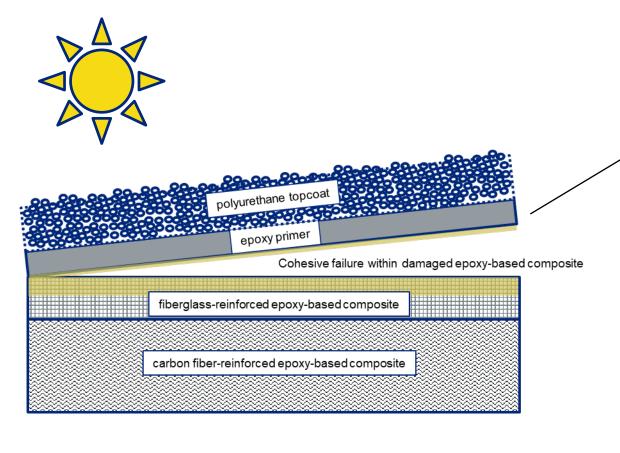


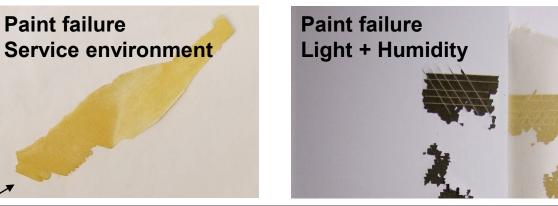
Verification of Failure Mechanism

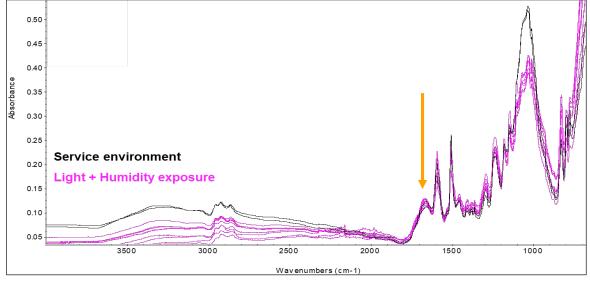
Coating adhesion tested after Light + Humidity cyclic exposure

FTIR confirmed degraded composite resin on both accelerated exposure and

service environment samples







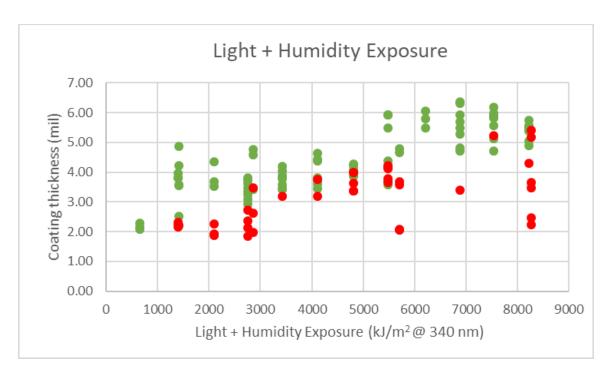
Evaluation of Aerospace Topcoat over Epoxy-based Composite

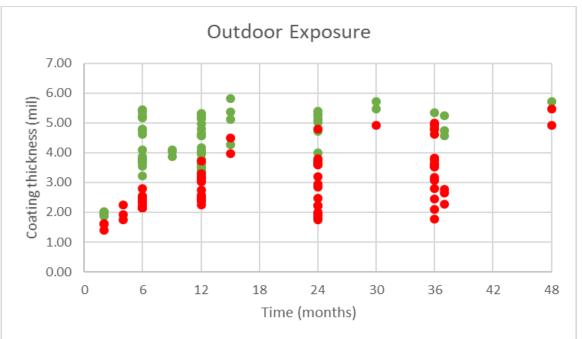
- The extent of composite degradation under paint depends on the light-blocking properties of a coating formulation as well as the applied thickness
 - For a given coating formulation, higher thickness will reduce light transmission and increase time before failure
- One aerospace coating/composite system
 - Three target coating thicknesses
- Light + Humidity cyclic exposure:
 - 12 timepoints (650-8200 kJ/m² @ 340 nm)
- Standard outdoor exposure:
 - 10 timepoints (2-48 months) to date
- Paint adhesion test after exposure to determine level of composite degradation under paint



Results: Light + Humidity vs. Outdoor Exposure

- After exposure, panels were tested and deemed "pass" or "fail" based on level of composite degradation under paint.
- Coating thickness was measured for each test area for better accuracy.
- For both exposures, thinner coatings failed at earlier timepoints.





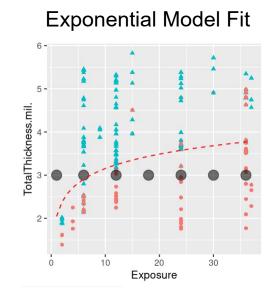
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Acceleration Factor

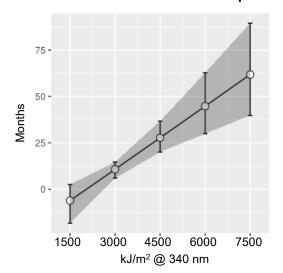
 Linear and exponential models developed using both sets of results

 Calculated conversion between accelerated cyclic and outdoor exposure

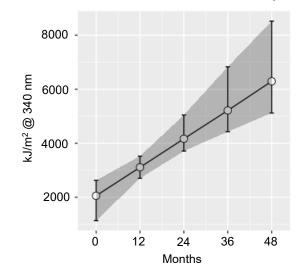
Linear Model Fit



Conversion to outdoor exposure



Conversion to chamber exposure



- ~300 kJ/m² @ 340 nm per week
- Equivalent to 1 year outdoor in ~11 weeks
 in Light + Humidity chamber exposure
- 4-5x acceleration over outdoor weathering

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Summary

- Minute amounts of light transmitted through coatings can degrade composite substrates over time
 - Need test methods that can quickly and accurately predict long-term durability
- New cyclic exposure protocol
 - Utilizes intense simulated sunlight (existing commercially available equipment)
 - Cycles of warm, wet conditions accelerate adhesion failure
 - Reproduces the failure mechanisms observed in outdoor weathering and service environments
 - Acceleration factor 4-5x relative to standard outdoor weathering
- Faster evaluation of UV-visible light protective coating and composite systems

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- Dr. Paul Vahey Associate Technical Fellow
- Dr. Gwen Gross Technical Fellow
- Dr. Nels Olson
- Dr. Ahmad Nahhas

- Geoff House
- James Kirchner
- Maribel Locsin
- Michael Zelinsky

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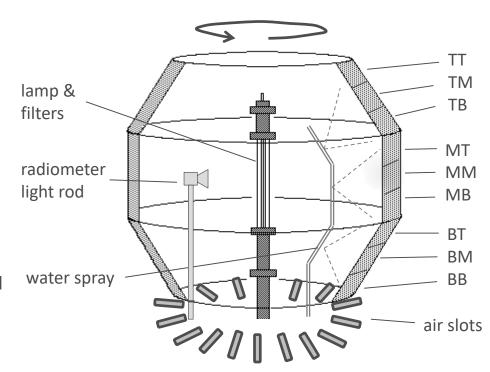
VARIABILITY WITHIN AND AMONG ACCELERATED WEATHERING DEVICES

Derek Yeager, Olga Kuvshinnikova, James E. Pickett, and Kevin Betts 2023 ASTM Workshop, Denver, Colorado; June 28, 2023

GENERAL BUSINESS USE

MOTIVATION

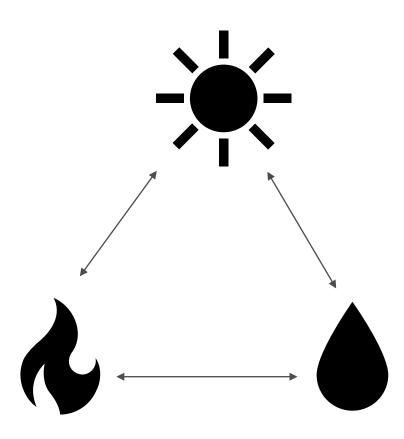
- Consistency of results within and among weathering devices has been a long -standing question
 - Tendency to make a big deal of what might be just experimental variability
 - What at is the expected repeatability and reproducibility under very controlled conditions?
 - Can we avoid wild goose chases?
- Opportunity to investigate with new weathering lab and equipment
 - 3 new Atlas Ci4400 Weather -ometers ®: W4-1, W4-2, W4-3
 - 2 new Atlas Ci5000 Weather -ometers ®: W5-1. W5-2
 - Right Light [®]/quartz filters; 0.75 W/m ²/nm; 40 °C chamber; 63 °C black panel
 - Using 102/18 spray cycle from ISO -4892-2
 - Utilized white and black polycarbonate samples for testing
 - Careful maintenance and rigorous DI water control
 - Samples, operators, and instrumentation consistent



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MOTIVATION (CONT.)

- Accelerated Weathering Variation Factors of Interest
 - Irradiance
 - Temperature
 - Water
- Experiments
 - Color shift and gloss loss study of white and black polycarbonate
 - Irradiance
 - Independent check of irradiance using XenoCal[®]
 - Temperature
 - Temperature mapping in all positions, white and black samples





IRRADIANCE

- Calibrated with all available lamps and checked with XenoCal at set 0.75 W/m²/nm at 340 nm
- Calibrated all Ci4400s with L4-1 and both Ci5000s with L5-1 for subsequent experiments
- > Variability due both to calibration lamps and to systematic device differences, even with good calibration
- Estimated differences by rack position < 4% in Ci4400 and < 2% in Ci5000

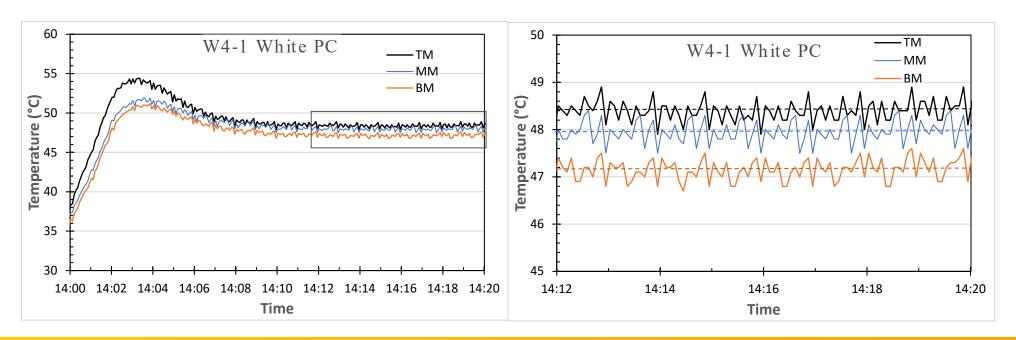
XenoCal measurement for calibration lamp/WoM combinations Normalized to reading for W4-1 with L4-1 (0.755)

		Ci4400s	Ci5000s		
	L4-1	L4-2	L4-3	L5-1	L5-2
W4-1	1.00	1.02	0.95		
W4-2	0.95	0.95	0.91		
W4-3	1.01	0.97	0.95		
W5-1				1.01	
W5-2				1.03	1.05



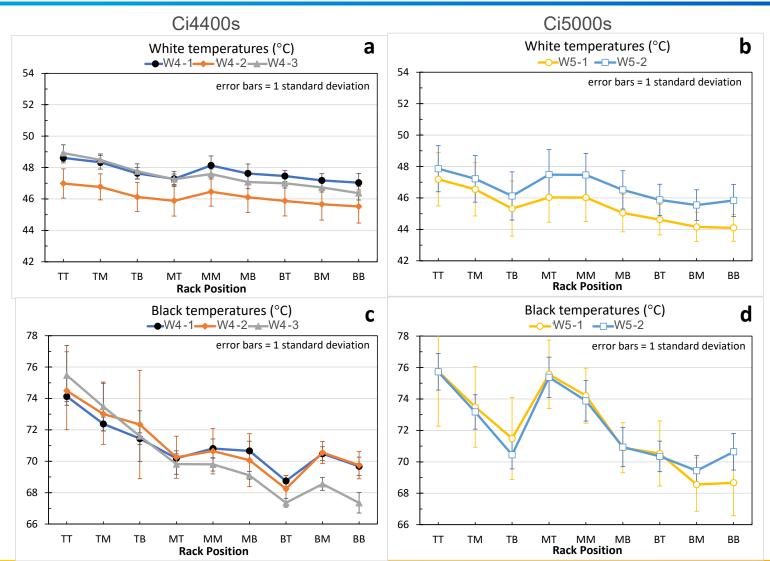
TEMPERATURE MAPPING

- MadgeTech TC101A data loggers with Omega 36-gauge Type Kthermocouples
- Lightly imbedded into surface of white (3% TiO₂) or black (0.15% carbon black) polycarbonate
- Warmed up WoM for at least 20 minutes
- Installed 9 specimens, three per holder, in a line on each of the three tiers. Rack filled with blanks
- > Delay start on recorders, so all began simultaneously after WoM restart with data every 5 seconds
- > Typical data below. Averaged data for last 10-15 minutes during flat period
- > Averaged 3 to 4 independent runs for each color in each WoM









White specimens

Modest top to bottom gradient

Similar for both models

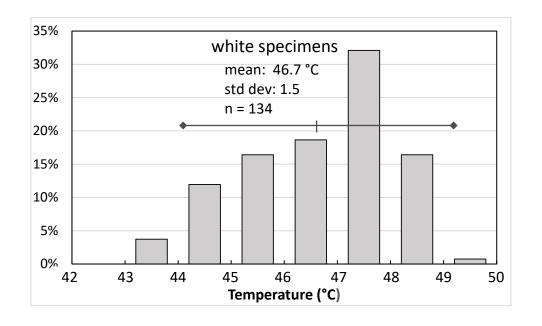
Small systematic variations among WoMs

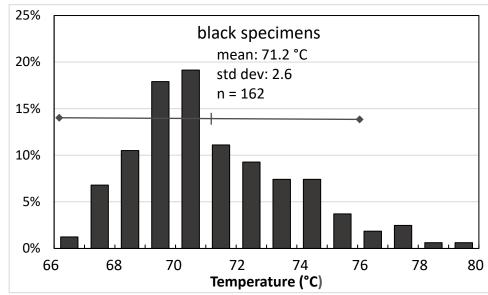
Black specimens
Significant top to bottom gradient
Systematic differences between models
May be different at a different irradiance





	Ci4400s	Ci5000s	combined
white mean (©)	47.1	46.1	46.7
white std dev	1.2	1.6	1.5
black mean (©)	70.6	72.2	71.2
black std dev	2.3	2.8	2.6

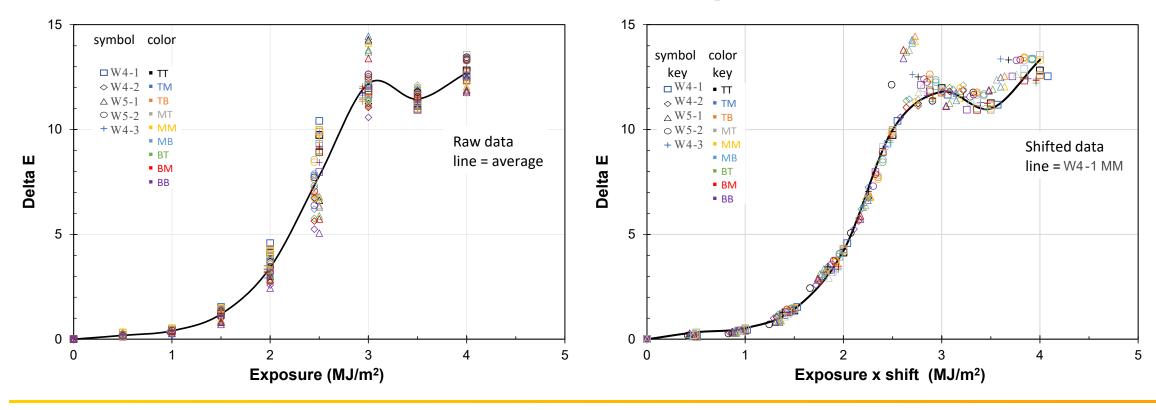






DEGRADATION RATE MEASUREMENTS

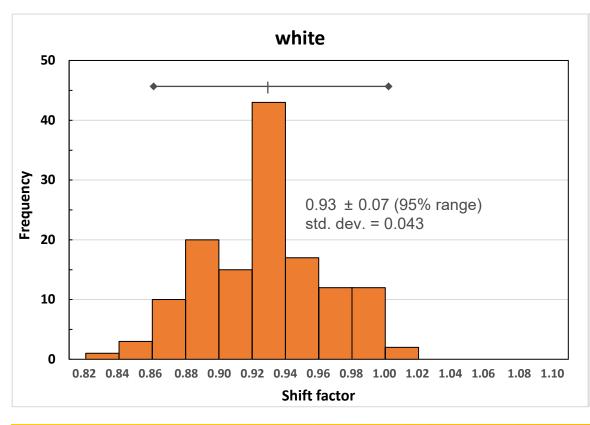
- ➤ White and black polycarbonate; Delta E, Delta 20 ©gloss, Delta 60 ©gloss
- Example: white PC Delta E
- Large variation in raw data—hard to define variation
- ➤ Used shift factors on Exposure axis to superpose data onto a reference set from Ci4400 "W4-1 MM"
- ➤ Shift factors are the rates relative to the reference W4-1 MM samples

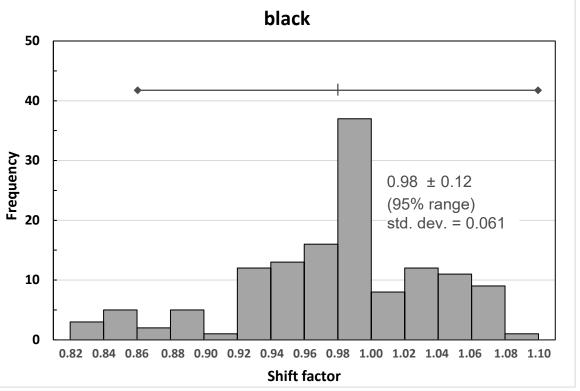




HISTOGRAMS OF RELATIVE RATES

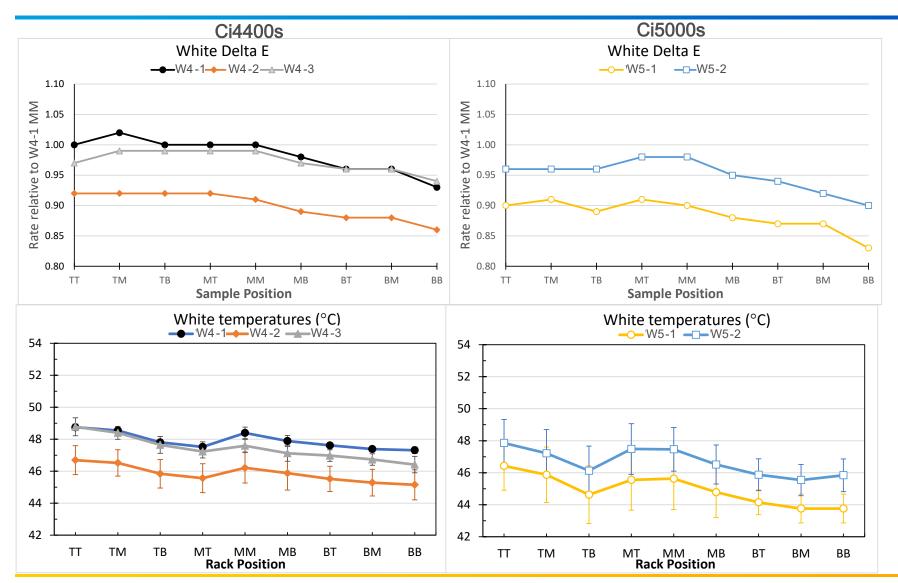
- ➤ All WoMs, all 3 properties. Rates relative to MM position in W4-1
- ➤ White samples well behaved. All specimens within 20% range; 95% of samples within 14% range
- ➤ Black samples have a tail due to gloss loss in two particular WoMs: 95% of samples within 24% range
- > Water spray can affect surface appearance of black materials







WHITE DEGRADATION RELATIVE RATES



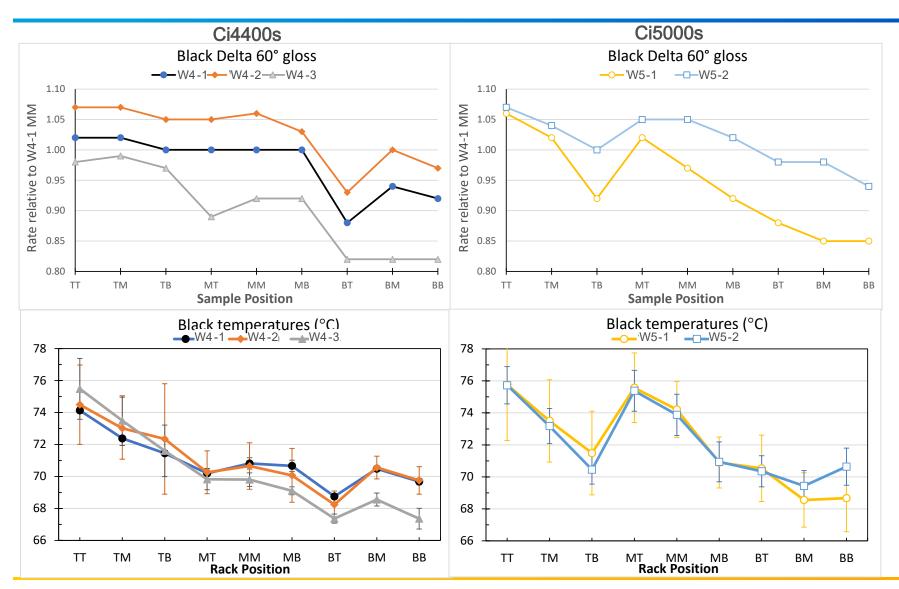
White Delta Erates closely follow temperature profiles

Gloss loss very similar

Delta E (SCE) for black samples similar



BLACK DEGRADATION RATES



Gloss data rates track temperature less well

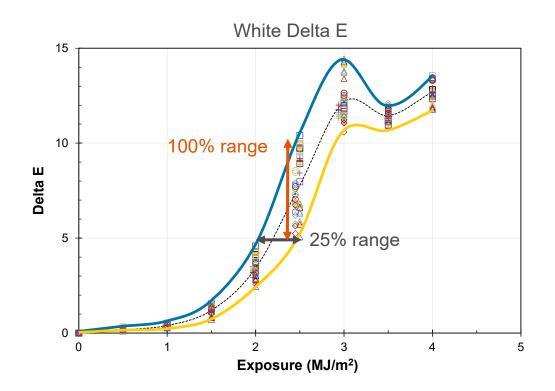
Water spray variations may affect gloss results

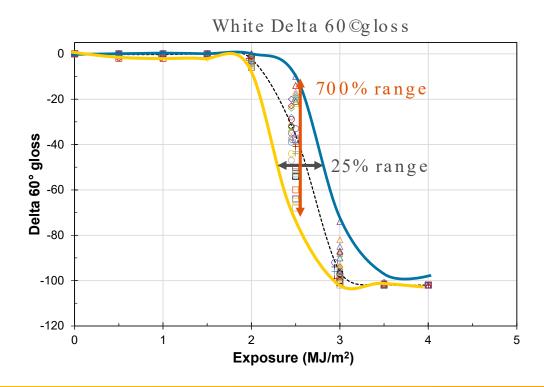
... but these conditions do not accurately predict gloss loss outdoors!



SIGNIFICANCE

- > PC is a challenging test because of abrupt property change
 - Can be large difference in values at steep part of curves (e.g. meeting specs)
 - Much less difference in actual rate of degradation (e.g. service life prediction)
- > Impossible to judge without understanding shape of degradation curve
 - Makes evaluation by value of a property at a single point problematic





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CONCLUSIONS

- > Temperature gradients seen in all instruments
 - Less severe for white specimens: 8 °C range; min 41.1, max 49.3 °C
 - Pronounced for black specimens: 12 °C range; min 66.9, max 79.8 °C
 - Some differences between models, especially for the black specimens
- Temperature variations account for most of degradation rate variations
 - Irradiance variations are fairly small and can be reduced with
 XenoCal calibration
 - Additional factors (probably spray) affect black gloss loss
- > Rate differences of < 20% to 25% cannot be considered significant unless specimens are literally side

-by-side.

- Assuming activation energy of 4
 -5 kcal/mol (15 -20 kJ/mol), typical of aromatic polymers
- Larger rate differences expected for polyolefins, which have higher activation energies
- Need very frequent sample rotation to fully average temperature differences in dark colors
- Significance depends on shape and magnitude of property change curve and method of data analysis
 - Very large differences in property change can occur on the steep part of curves
 - May not be important if change is small or nearly linear
- > Expect flat bed instruments also to have significant variations without sample rotation

سابک عادہ

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ABSTRACT

As part of the commissioning process for five new Atlas xenon arc Weather-ometers®, the degrees of uniformity within each device and consistency among devices were evaluated. We measured the actual specimen surface temperatures of white and black polycarbonate plaques in all the nine sample positions from top to bottom in each device, determined the actual irradiance in each device using an independent radiometer, and determined the relative rates of color shift and gloss loss for white and black polycarbonate plaques in all the nine sample positions from top to bottom in each device. A temperature gradient was found for all devices. This was fairly small for white specimens but larger and more variable for black specimens. Degradation rates generally followed the temperature profiles. For whites, the standard deviation for color shift and gloss loss relative rates for all devices and all positions was 0.042, suggesting that rates for 95% of specimens should be within ± 8% of the mean. Black specimens had a higher overall standard deviation of 0.061 (mostly due to higher ranges for gloss loss), suggesting that rate s for ± 12% of the mean. Therefore, rate differences of 15% 95% of the specimens should be within - 20% in light colors and ~25% in dark colors should not be considered as significant unless samples are exposed literally side -by-side. The values of a property any pull may vary much more if the property is changing rapidly around that time. Evaluations should involve taking enough d ata points to understand the shapes of the degradation curves. Samples should be regularly rotated through the various rack positions to improve uniformity. There is no reason to expect that flat -bed devices provide less variations in temperature and degradation rates.

Cool Things you can do with Black and White Panels

Backyard weathering plus lots of colorful photos and graphs!

Q-Lab Corporation Michael Crewdson



History and Origins

- Previous study by Dick Fischer and Warren Ketola
 - Surface Temperature of Materials in Exterior Exposures and Artificial Accelerated Tests
 - STP 1202: Accelerated and Outdoor Testing of Organic Materials 1994. Eds. Ketola and Grossman
- Compared colored panels on outdoor exposure
 - They calculated a regression analysis to predict temp based on color
- Compared differences outdoors to differences in accelerated weathering



Follow up Questions

- Can other specimens' temperature be predicted?
 - Can we establish a relationship for different materials?
 - Theoretically or using other experimental methods
- Further study on black panel constructions
 - ASTM G179 standard panel versus other panels
- How reliable is Black Panel Temperature as a weathering input?
- What about other exposures: backing, angle, substrate?



Study in Two Parts

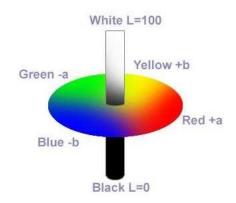
- Part One: Experimental
 - See if the original study could be replicated
 - Compare different black panel constructions
 - Use Heat Box to see if temperatures can be predicted
 - Other exposure types?

- Part Two: Data Mining
 - Used one year of Florida outdoor weather data
 - Compare data already saved
 - Used the 2022 year



Recreate the Study

- 7 "colored" panels*
 - Black, Gray, White, Red, Blue, Green, Yellow



Panel	L*	a*	b*	C*	h°
Black	24.42	0.03	-0.21	0.21	277.70
White	91.74	-1.08	-0.13	1.09	186.94
Gray	45.56	-0.82	-6.12	6.17	262.35
Red	40.69	43.39	23.36	49.28	28.30
Blue	45.88	-8.00	-36.66	37.53	257.69
Green	58.10	-42.78	18.41	46.57	156.72
Yellow	82.67	0.69	62.04	62.05	89.36
G179 Black	25.05	0.02	0.63	0.63	88.50
G179 White	88.94	-0.88	-0.01	0.88	180.69

* Added gray, omitted orange



Gloss Values

Panel	20°	60°	85°
Black	0	1	5
White	1	6	14
Gray	0	3	8
Red	40	87	90
Blue	33	79	77
Green	2	12	15
Yellow	36	82	78
G179 Black	69	89	97
G179 White	80	88	94

Spoiler Alert: Gloss is not a significant factor

- Not concerned about coating durability
- Used readily available paints
- Tried to get all the same "type"
 - Black and white not glossy
 - Gray is primer only
 - Green low gloss (?)



We make testing simple

Making the Panels

Construction

- 4" x 12" x 0.032" steel panels
 - Type T Thermocouples
- Four coats 'primer+paint'
- "Hot Spot" welded T/C to back center
- Trial and error to find correct setting
- G179 Black Panel compared











Other Equipment Used

- Omega 8 channel data logger
 - 1 minute sampling rate
 - All channels verified
 - Eko Instruments TSR Pyranometer
 - Range ~300 3000 nm
 - Approx 7 μV per W/m²
- Aluminum exposure rack
 - Per ASTM G7-69
 - Tilted at ~25 degrees South





Exposure

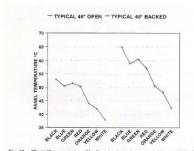
- Exposed 25° South, Florida
 - Lat. 26.5 N, Lon. 82.0 W, Elev. 4 m
- Unbacked and backed options
- Quick disconnect thermocouples
- Exposure from 9 am to approx. 4 pm
 - Only interested in comparison temp rise





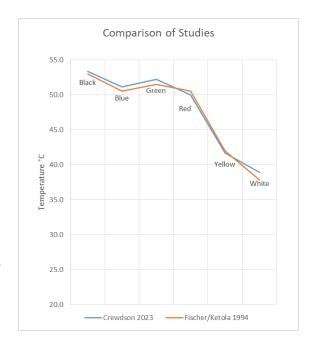
Confirmation of Earlier Data

	Crewdson 2023	Fischer/Ketola 1994
Black	53.3	53.0
Blue	51.1	50.5
Green	52.2	51.5
Red	50.0	50.5
Yellow	41.7	42.0
White	38.9	37.8



Comparison of common colors pretty good

Data expressed in same order and manner prove the techniques in the Fischer/Ketola study and mine, are the same



backed (BPT - 65°C) exposures. (from Minnesota 45° open and Minnesota 30° backed temperature

Black Panel Constructions

- ASTM G179 specified black and white
- Commercially available types
 - ACT Cross
 - Omega Washer Thermocouple
- Mike's gray and white panels
- 4" x 12" aluminum black panel
- ASTM G179 black backed





Top I-r: G179 BP, Mike's White, Mike's Gray, Washer TC, G179 White, Aluminum, ACT Panel, Bottom: G179 BP Backed

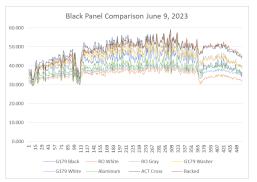


Findings on Constructions

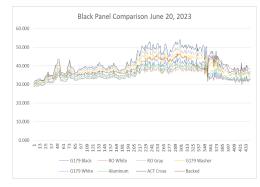
	Maximum Change per Minute °C								
Approx Run Times	G179 Black	RO White	RO Gray	G179 Washer	G179 White	Aluminum	ACT Cross	Backed	
6/9/2023 9am to 4:45 pm	5	3	9	5	3	5	8	5	461 Readings
6/20/2023 9am to 4:15 pm	14	6	13	9	6	10	13	10	443 Readings

	Total Degrees of Change *C								
Approx Run Times	G179 Black	RO White	RO Gray	G179 Washer	G179 White	Aluminum	ACT Cross	Backed	
6/9/2023 9am to 4:45 pm	353	168	369	257	176	296	366	213	461 Readings
6/20/2023 9am to 4:15 pm	725	322	633	476	339	598	565	548	443 Readings

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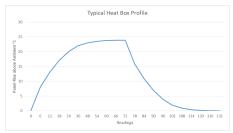






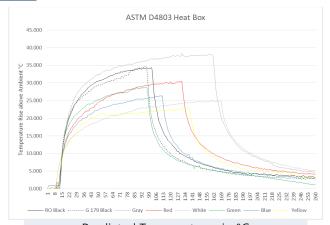
Experimental Prediction

- ASTM D4803 Heat Box for plastic materials
 - Calcium Silicate insulated box
 - GE 250W Heat Lamp
 - Type T Thermocouple
 - Data Logger
- Turn on heat lamp and record the temperature
 - increase
- Stop when temps stop rising
- Compare to a reference panel

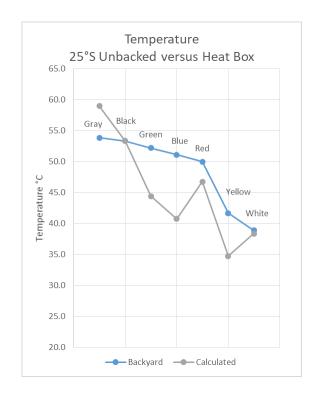




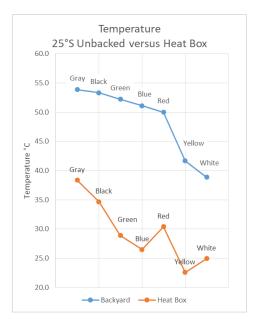
Heat Box D4803 Results



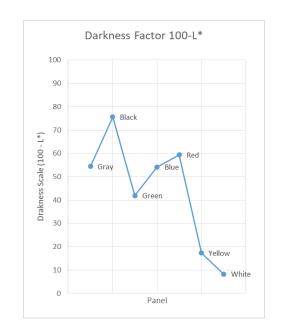
Predicted Temperatures in °C							
Color	25°Rack	Heat Box	Predicted				
Gray	53.89	38.36	58.99				
Black	53.33	34.68	53.33				
Green	52.22	28.90	44.44				
Blue	51.11	26.50	40.75				
Red	50.00	30.42	46.78				
Yellow	41.67	22.60	34.76				
White	38.89	24.95	38.37				



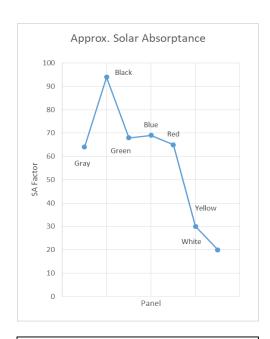
Can we Predict Panel Temperatures?







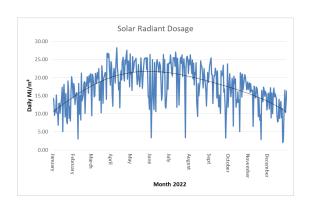
Not with an L* Scale



Not with Solar Absorptance

Weather Data Studies

- Typical daily variability
- Most days are a mixture of sun and clouds
- Much fewer all clear or all cloudy days



Typical summer day in South Florida

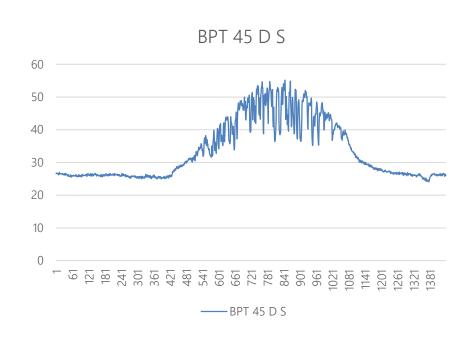


Figure 16: Mike's all-time favorite weathering photo



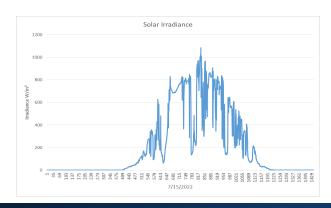
Black Panel Temperature Changes

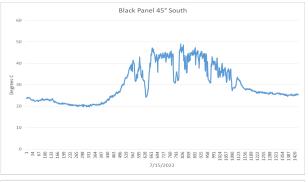
- Typical summer day, no rain
- Example July 2, 2022
- Using minute data
 - Total of 1009 °C of change
 - 81 events > 3 °C per min
 - 24 events > 5 °C per min
 - Maximum 60 second change of 7.4 °C

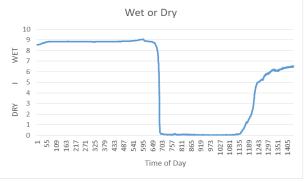


Rapid Responses

- The overall diurnal change is slow
- But there are many rapid fluctuations in a day
- Some fluctuations can be rather large
- Outdoor conditions can change often and quickly too



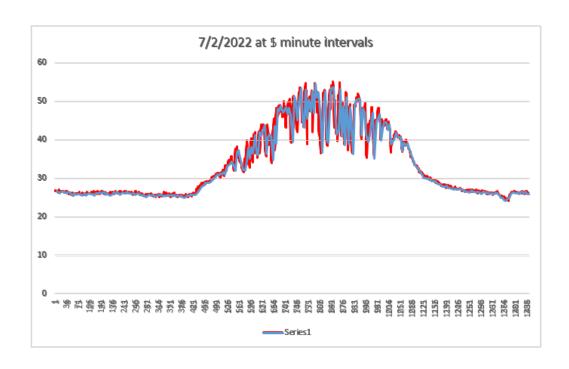






Other Considerations

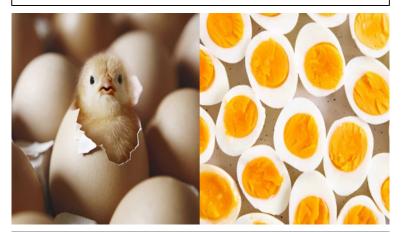
- Rate of change of the specimen
- Sampling rate of data logger must be faster
- 5 minute versus 1 minute
- Charts look the same
- Until compared together
- Highs and lows missed
- Total temp change is less





What it means for Ramp Times

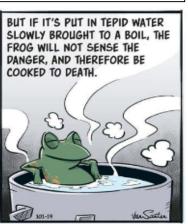
Traditional meme of the importance of temperature



If you increase the temperature you will get faster results

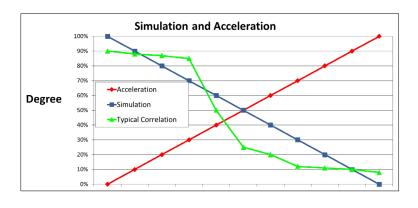
Proposed new meme of the importance of rapid ramping





Rapid changes in weathering factors are more realistic

Can we Improve Correlation?

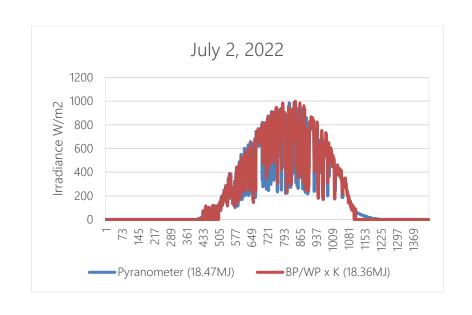


- Even at "perfect" simulation we don't get 100% agreement
- I suggest that the "outdoor operational fluctuation" may be a contributing factor
- The lab testers are not the problem, they're great
- But maybe we can improve the cycles
 - add more fluctuation?
 - faster ramp times?

We make testing simple

Poor Person's Solar Radiometer

- Black and White panels can be used to measure solar irradiance
- The intensity of solar flux is shown by the difference in the temperature between the two panels
- There is a factor K that converts the difference to an irradiance

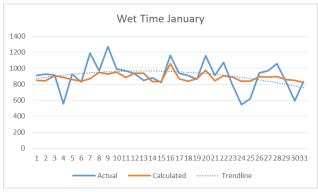


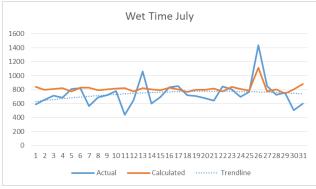
Useful in an emergency!



Possible Wet Time Sensor

- We are always looking for a <u>simple</u> way to derive wet time indirectly
- Condensation assumed when black panel temperature is lower than white panel temperature
 - If there is sufficient humidity?
- Not good for actual day to day
- Further analysis needed
- Matches well to the "trendline"
- Might be good to characterize a site location







Further Investigations

- Compare the G179 black panel to the G151 Black Panel and Black Standard
 - Exactly how realistic is an insulated black panel?
- Evaluate different constructions for the outdoor black panel
 - Easier to make and more rugged, not Type T
 - Improve the instructions in ASTM G179
- Look at relationships in desert exposures (Arizona)
- Compare steel panels to aluminum and plastic black panels
 - Include different sizes and thicknesses



Final Thoughts?

- This is definitive proof for a <u>standardized Outdoor Black Panel</u> (sOBP)
 - Maybe not the same exact construction as now though
- The sOBP is intended as a climate identifier only, like ambient temp
 - Characterize a test site (Climate)
 - Record of each day (Weather)
- The sOBP cannot be used to guess or predict the temperature of other specimens
 - Unless the construction is "identical" (not just close)
 - Cannot cross reference to other materials
- Sometimes the sOBP is not the hottest specimen in the field
- IF this is true for outdoors, then it's probably true for accelerated!



Thank you for your time.

Questions? info@q-lab.com







ASTM WEATHERING AND DURABILITY WORKSHOP 2023

UNCERTAINTY BUDGET OF SERVICE LIFE PREDICTION MODELS BASED ON ACCELERATED WEATHERING

DR. FLORIAN FEIL, MATT MCGREER, OSCAR CORDO

JUNE 28^{TH} , 2023, DENVER







How fast is my test?



How good is the correlation?

How reliable is the prediction?







General model for service life prediction/SLP:

- under defined operating conditions,
- \blacksquare based on mathematical modeling of time to product failure (t_F)
- \blacksquare compared to the accelerated weather factors (E_e , T_s , H_2O),
- calculated using (historic) data (solar radiation, air temperature, RH, etc...)

SLP:
$$P(t_F)-P(0) = \int_0^{t_F} f(E_e, T_s, H_2O) dt$$

According to David M. Burns, Gunther Stollweck. ATCAE 2011.

P(0): original property $P(t_F)$: property at failure

(pass/fail)

t: time

E_e: effective irradianceT_s: surface temperature

H₂O: water in each

modification (liquid, vaporous)

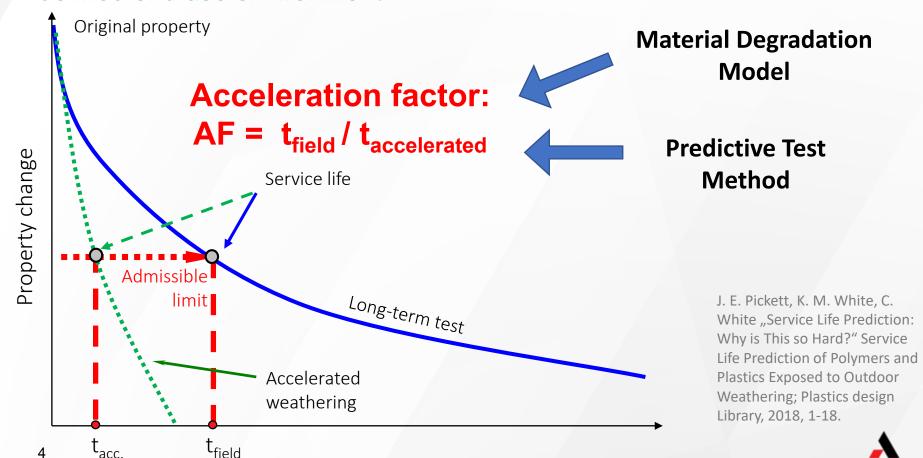


SLP – SIMPLIFIED APPROACH



SLP reduced to continuous ageing processes:

To calculate the (theoretical) acceleration factor based on a material degradation model of a realistic predictive (tailored) test method and a defined end use environment.



OUTLINE: UNCERTAINTY OF SLP



- a. Influence of the material uniformity
- Influence of the evaluation criteria on the uncertainty budget
- Uncertainty budget of parameter control and measurement
- d. Uncertainty budget of accelerated weathering
- e. Influence of the end use environment on the uncertainty budget
- f. Uncertainty of the model
- g. Summary



STATISTICS OF SERVICE LIFE DATA



Weibull Probability Density

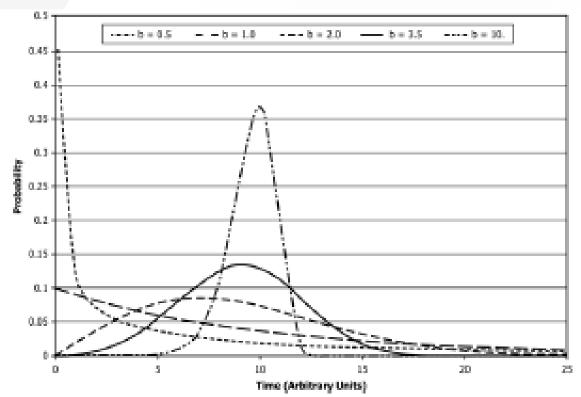


FIG. 1 Effect of the Shape Parameter (b) on the Welbull Probability Density

Guidance exists:

ASTM G166 - Standard Guide for Statistical Analysis of Service Life Data

ASTM G172 - Standard Guide for Statistical Analysis of Accelerated Service Life Data

$$F(t) = 1 - e^{-\left(\frac{t}{c}\right)^b}$$

t = units of time used for service life

c = scale parameter

b = shape parameter

b < 1: decreasing # of failures

Infant mortality

b = 1: constant failure rate

b > 1: increasing failure rate

Here we look at continuous ageing effects and not at infant mortality.

Assumption: 100% uniform and identical material





OUTLINE: UNCERTAINTY OF SLP



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UNCERTAINTY OF EVALUATION CRITERIA



The uncertainty budget depends on the evaluation criteria for the relevant property change

- To minimize uncertainties, it is important to follow established standards and guidelines
 - Color according to ASTM E1331, ASTM E308, ASTM D2244
 Influence on uncertainty: color, color depth, surface pattern, dirt, ...
 - Gloss according to ASTM D523, ISO 2810
 Influence on uncertainty: reflectance, surface pattern, sample curvature, dirt, ...
 - Chemical degradation (FTIR) according to ISO 10640;
 Influence on uncertainty: resolution, thickness, depth profile
 - Others (Haze, delamination, ...)

Estimated "average"
uncertainty:
Evaluation criteria: ± 10%



OUTLINE: UNCERTAINTY OF SLP



- Influence of the evaluation criteria on the uncertainty budget
- b. Influence of the material uniformity
- c. Uncertainty budget of parameter control and measurement
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UNCERTAINTY BUDGET OF CONTROL PARAMETERS



The uncertainty budget of **UV and Temperature** measurement depends on the following:

- Instrument Calibration
- Measurement equipment (type, spectral range, sensitivity, linearity, and stability of the instrument) and technique (probe or integrating sphere-based measurements)
- Environmental Conditions during calibration and measurement (ambient temperature, humidity, and atmospheric pressure, ...).
- Operator Skill and Training
- The size of the uncertainty budget can vary depending on the accuracy requirements of the measurement, the quality of the equipment, and the calibration standards used.
- To minimize uncertainties, it is important to follow established standards and guidelines
 - Measurement of surface temperature according to EN 16795
 - Calibration of surface temperature sensors according to EN 16465
 - Instrumental measurement of irradiance according to ISO 9370

Estimated uncertainty:

Irradiance: ± 10%

Temperature: ± 5 K



OUTLINE: UNCERTAINTY OF SLP



- a. Influence of the material uniformity
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- Uncertainty budget of parameter control and measurement
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UNCERTAINTY OF ACCELERATED WEATHERING



Sources of uncertainty

- Instrument type and geometry
- Lamp type and age
- Spectral irradiance distribution
- Irradiance and temperature calibration

Control tolerances

- Irradiance (300 nm 400 nm): ± 5 W/m² (ASTM G151, ISO 4892-1)
- BPT/BST: ± 3 K (below 70 °C)/ ± 4 K (above 70 °C) (ASTM G151, ISO 4892-1)

Exposure uniformity

- Irradiance: at least 90% of maximum (ASTM G151)
- BPT/BST: ± 5 K (bellow 70 °C)/ ± 7 K (above 70 °C) (ISO 4892-1)

Estimated uncertainty:

Irradiance: ± 12%

Temperature: ± 8 K - 11 K

OUTLINE: UNCERTAINTY OF SLP

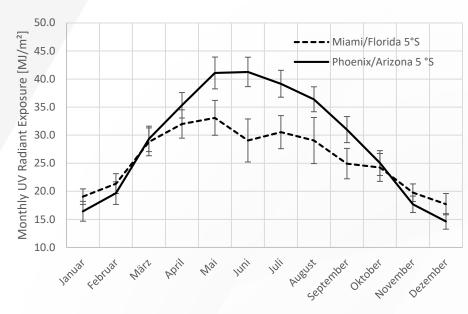


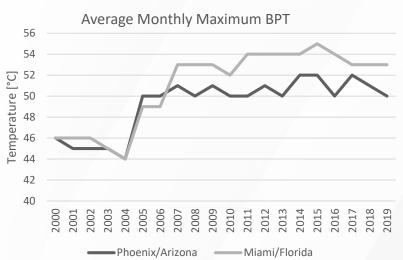
- a. Influence of the material uniformity
- Influence of the evaluation criteria on the uncertainty budget
- Uncertainty budget of parameter control and measurement
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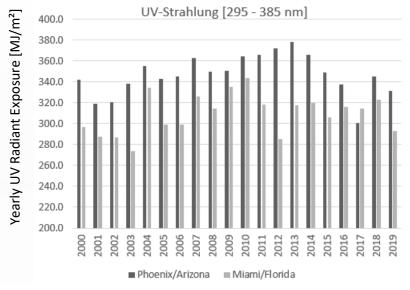


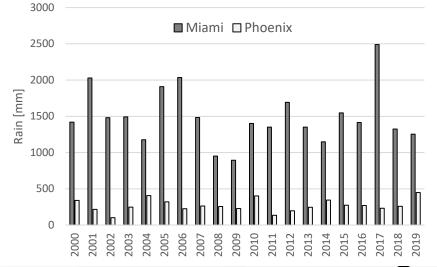
CLIMATIC VARIATION













VARIABILITY OF END-USE ENVIRONMENT



Location (Period)	Exposure angle	Tota	al	UV	
		295 - 24	50 nm	295 - 385 nm	
(i cilou)		MJ/m²	Rel. Std.	MJ/m²	Rel. Std.
Miami, Florida/US (2000 - 2022)	5° S	6337	2.6%	309	5.8%
	26° S	6675	2.7%	313	7.5%
	45° S	6313	3.1%	288	7.7%
Phoenix, Arizona/US (2000 - 2022)	5° S	7553	2.0%	348	5.3%
	32° S	8377	2.2%	351	6.8%
	45° S	8235	2.8%	333	6.6%
Sanary-sur-Mer, France (2006 - 2022)	0°	5774	3.5%	232	7.8%
	45° S	6896	3.4%	257	8.8%
Hoek van Holland, NL	0°	3963	6.1%	-	-
(2007 - 2022)	45 ° S	4587	5.4%	-	-
Chennai, India (2008 - 2022)	5° S	6876	4.0%	304	11.4%

- Location: Miami and Phoenix have the lowest year-to-year variation
- Spectral Range: UV year-to-year variability is higher than total radiation variability
- <u>Orientation</u>: Annual radiant exposure varies between maximum (often at latitude) and minimum (e.g. in the shadow, only diffuse) radiation
- Assumption: Orientation and end use environment of product is known

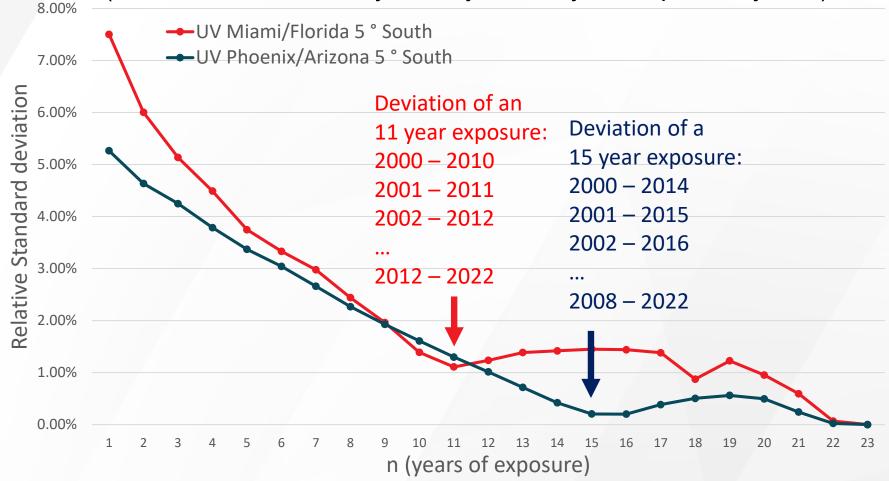
Estimated uncertainty: Irradiance: ± 10% Temperature: ± 2 K







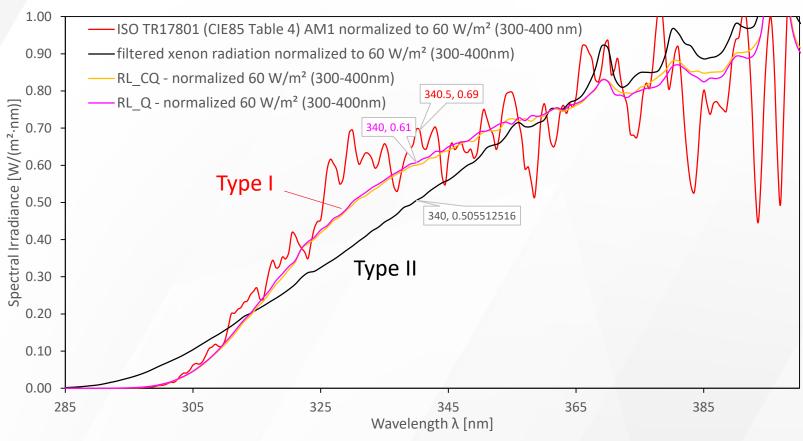
UV Irradiance deviation of a **n** year exposure in South Florida and Arizona (from 2000 to 2022; 1 year, 2 years, 3 years up to 23 years):





COMPARISON TO XENON



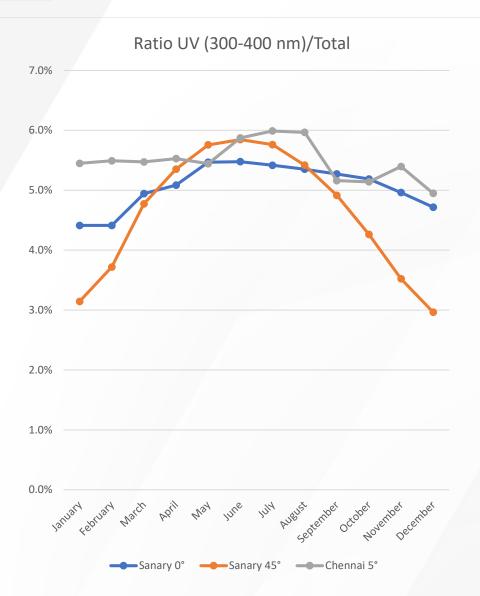


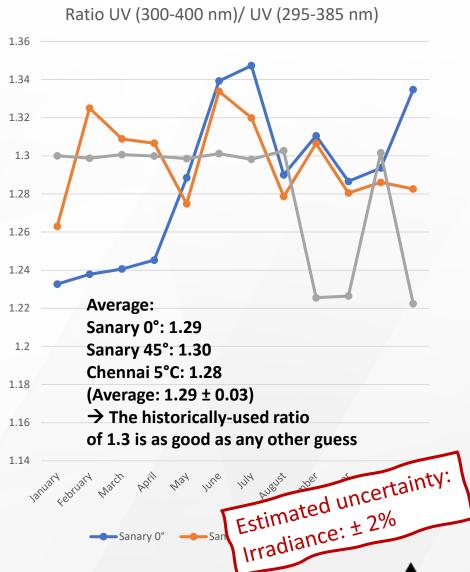
- → Comparison of Xenon-arc with narrowband (340 nm) and natural solar radiation (TR17801 or CIE 241) for Xenon-arc instruments does not make sense
- → Better to use broadband (300 nm 400 nm) for SLP, provided the effective irradiance is the same



RATIOS OF UV







CALCULATION OF UV FROM TOTAL



Often UV data are not available but total solar data is
Often the ratio **6.8%** from CIE 85/CIE 241 is used for conversion.....but:

Measured ratios (based on 2000 - 2022 averages)	UV (300 nm - 400 nm)/Total, direct									
	Miami*			Phoenix*			Sanary**		Chennai**	
	5°	26°	45°	5°	34°	45°	0°	45°	5°	
January	5.9%	5.2%	4.8%	5.5%	4.4%	4.0%	4.4%	3.1%	5.4%	
February	6.1%	5.6%	5.4%	5.7%	4.9%	4.6%	4.4%	3.7%	5.5%	
March	6.3%	6.1%	5.9%	5.9%	5.4%	5.1%	4.9%	4.8%	5.5%	
April	6.4%	6.5%	6.3%	6.0%	5.8%	5.6%	5.1%	5.4%	5.5%	
May	6.5%	6.7%	6.8%	6.1%	6.0%	6.1%	5.5%	5.8%	5.4%	
June	6.7%	7.0%	7.2%	6.1%	6.2%	6.3%	5.5%	5.8%	5.9%	
July	6.7%	6.9%	7.1%	6.3%	6.2%	6.5%	5.4%	5.8%	6.0%	
August	6.5%	6.6%	6.6%	6.4%	6.1%	6.2%	5.4%	5.4%	6.0%	
September	6.6%	6.3%	6.3%	6.2%	5.7%	5.5%	5.3%	4.9%	5.2%	
October	6.3%	5.7%	5.6%	6.0%	5.1%	4.9%	5.2%	4.3%	5.1%	
November	6.1%	5.2%	5.1%	5.7%	4.6%	4.2%	5.0%	3.5%	5.4%	
December	6.0%	5.1%	4.8%	5.5%	4.2%	3.8%	4.7%	3.0%	4.9%	
Average	6.3%	6.1%	6.0%	6.0%	5.4%	5.2%	5.1%	4.6%	5.5%	
rel. Stdev.	4.4%	11.3%	14.2%	5.3%	13.5%	18.1%	7.5%	23.0%	5.9%	

^{*)} values calculated based on H(300 - 400 nm) $^{\sim}$ 1.3 x H(295-385 nm); **) measured

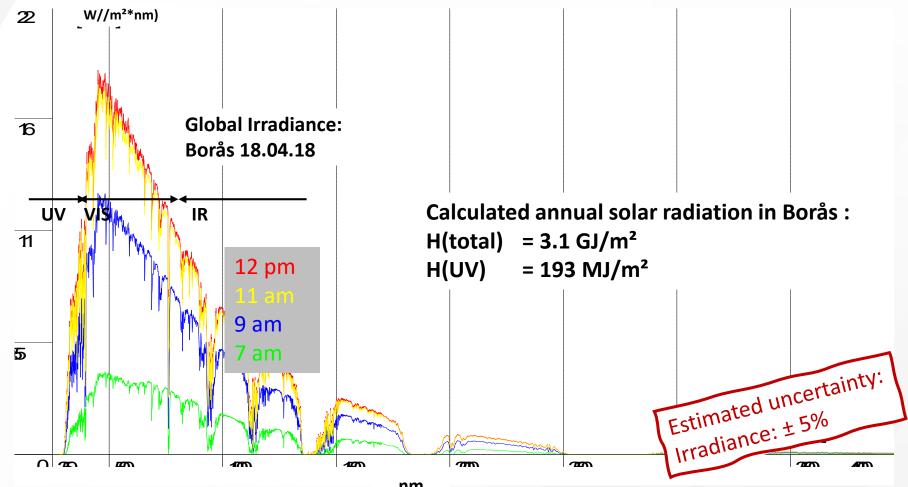
- → UV/Total ratio depends on atmosphere (local, diurnal, seasonal variation)
- → For most climates, the estimate of 6.8% is too high (average 5.6% ± 0.7%)
- → Additional information required on average UV/Total ratio of end-use environment
- → Not recommended to use total radiation to estimate UV
- →₁₉ ASTM G222 provides guidance on estimation of UV irradiance



LIMITATIONS OF ARTIFICIAL LIGHT SOURCES



Even the best optical filter system represents only one specific spectral irradiance distribution





OUTLINE: UNCERTAINTY OF SLP



- a. Influence of the material uniformity
- Influence of the evaluation criteria on the uncertainty budget
- Uncertainty budget of parameter control and measurement
- d. Uncertainty budget of accelerated weathering
- e. Influence of the end use environment on the uncertainty budget
- f. Uncertainty of the model
- g. Summary



MATERIAL DEGRADATION MODELS



Depending on material, different degradation pathways are possible with different influencing factors:

Property Change:

$$\Delta P(H,T,H_2O) = \sum \Delta P_{Thermal}(T) + \sum \Delta P_{UV}(H,T,H_2O) + \sum \Delta P_{Hydrolysis}(T,H_2O)$$

Luis E. Pimentel Real, 5th EWS 2006

Sum of all reactions that produce the same property change:

- thermal ΔP_{Thermal}(T_s)
- photochemical $\Delta P_{UV}(H_e,T_s,H_2O)$
- hydrolytic processes ΔP_{Hydrolysis}(T_s, H₂O)



Degradation rate:

$$\Delta P(H,T,H_2O) \sim E_{eff}^{\alpha} \cdot e^{-\frac{E_A}{RT}} \cdot [H_2O]^n$$

Law of Reciprocity (H) Arrhenius Concept (T)



RECIPROCITY LAW



Reciprocity is given

if the same amount of radiant exposure (H)
causes the same amount of photochemical
damage (or property change) no matter over
which time (t) it is applied.

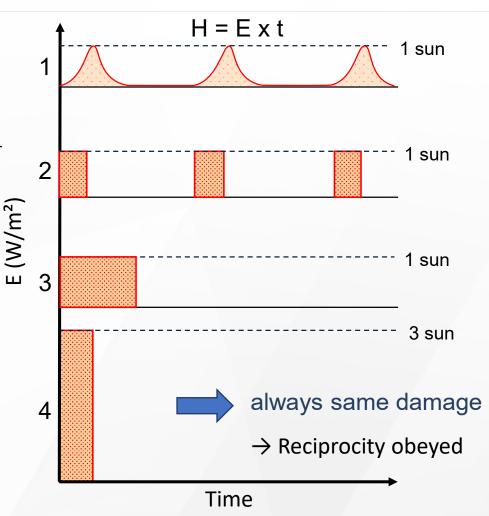
Reciprocity Law (Bunsen, Roscoe 1859):

 $E \times t = constant$

for a given photoresponse

Limitations of Reciprocity

- All other reaction parameters (temperature, humidity...) must be the same.
- Other reaction steps (like oxygen diffusion)
 must not be a factor in determining rate





SCHWARZSCHILD'S EQUATION



$k \sim A \cdot E^{\alpha}$

- Where k is the rate of reaction, A is a proportionality constant, E is irradianceand α is the experimentally derived Schwarzschild coefficient (slope of line of log(k) v. log(l) plot.
- When $\alpha = 1$, reciprocity is linear, i.e., strictly observed. Therefore, "reciprocity" is a special subset when Schwarschild's $\alpha = 1$
- For α < 1, the rate of property change increases less than expected from the increased light intensity. For low levels of α , degradation is underestimated, and lifetime is overstated (often between 0.5 and 1).
- However, even if reciprocity is not linear the effect may be repeatable for a given material. In such cases, high irradiance testing may still be used for materials with high α values, provided an equation can be fit to the data. As α values decreases, the correlation goes down and the test acceleration factor decreases, limiting its usefulness.



ARRHENIUS EQUATION FOR PHOTOCHEMICAL REACTIONS

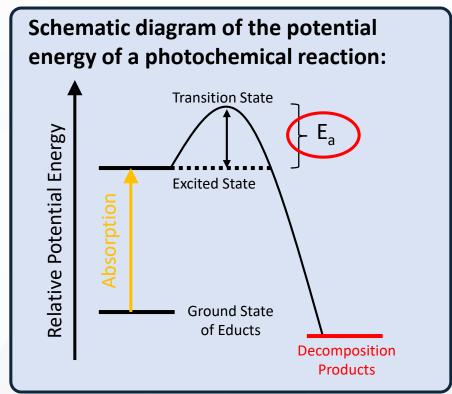


The modified Arrhenius equation describes the influence of temperature on the reaction rate of photochemical degradation processes:

$$k = A \cdot E^{\alpha} \cdot e^{Ea}$$

where

k is the reaction rate of the process A is an Arrhenius pre-exponential factor E_a is the apparent activation energy (in J.mol⁻¹) R is the gas constant (8.314 J.mol⁻¹·K⁻¹) T is the absolute temperature (in K) α is a material specific coefficient E is the effective irradiance (in W·m⁻²)



The reaction rate depends on effective irradiance and temperature



ACCELERATION FACTOR FOR SLP



- Acceleration factor AF:

$$AF = rac{t_u}{t_a}$$

where t is time to failure in a: accelerated environment u: use environment

Modified Arrhenius:

$$k = A \cdot E^{\alpha} \cdot e^{-\frac{E_a}{RT}}$$

■ The (theoretical) acceleration of temperature dependent photochemical degradation resulting from an increase in temperature and/or irradiance can be described by the ratio of the reaction rates:

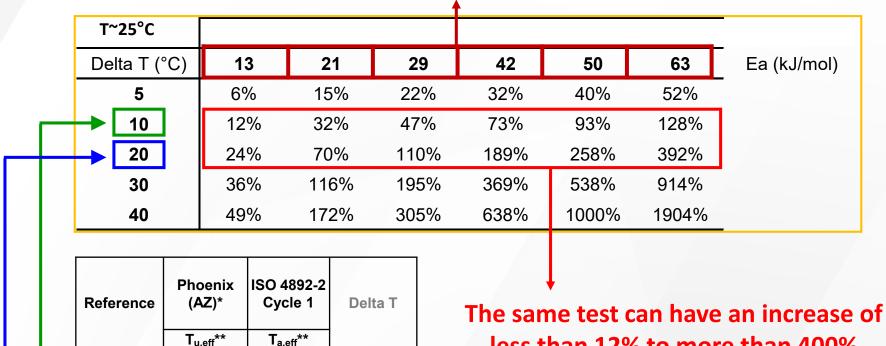
$$\frac{k_a}{k_u} = \left(\frac{E_a}{E_u}\right)^{\alpha} \cdot e^{\frac{E_A}{R}\left(\frac{1}{T_u} - \frac{1}{T_a}\right)} = AF_R \cdot AF_T = AF$$







Different reactions will have different acceleration rates due to increased temperatures



38

48

47

69

9

21

WST

BST

less than 12% to more than 400%

→ for SLP it is essential to know E_a and the surface temperature



^{**)} Effective average temperatures (T_{eff}) are calculated based on the cummulative damage model (J.E. Pickett, J.R. Sargent, Polym. Degrad. Stab., 94 (2009) 189.)





$$\frac{k_{ac}}{k_u} = \left(\frac{E_{ac}}{E_u}\right)^{\alpha} \cdot e^{\frac{E_a}{R} \cdot \frac{1}{T_u} \cdot \frac{1}{T_a}} = AF_R \cdot AF_T = AF$$

Irradiance

Photo-degradation Process

Surface Temperature

Depending on

- Test conditions
 (the SPD has to be similar or the effective irradiance has to be considered)
- Same for all materials (if reciprocity applies)

Depending on:

- Material
- Critical property
- Same for all tests
 (realistic test conditions,
 i.e. SPD, RH etc.)

Depending on:

- Ambient conditions
- Surface temperature
- Absorbance (specimen color)
- Mounting (specimen Insulation)



MINIMIZING UNCERTAINTIES



$$\frac{k_{ac}}{k_u} = \left(\frac{E_{ac}}{E_u}\right)^{\alpha} \cdot e^{\frac{E_a}{R} \left(\frac{1}{T_u} - \frac{1}{T_a}\right)} = AF_R \cdot AF_T = AF$$

Irradiance

Photo-degradation Process

Surface Temperature

ISO/TS 19022

Plastics –
Controlled acceleration
of laboratory weathering
by increased irradiance

ISO 23706

Plastics — Determination of apparent activation energies of property changes in standard weathering test methods

EN 16795

Plastics - Method for estimating heat build up of flat surfaces by simulated solar radiation

Estimated uncertainty:

Irradiance: ± 10%

Temperature: ± 5 K



OUTLINE: UNCERTAINTY OF SLP



- a. Influence of the material uniformity
- Uncertainty budget of parameter control and measurement
- c. Uncertainty budget of accelerated weathering
- d. Influence of the end use environment on the uncertainty budget
- e. Influence of the evaluation criteria on the uncertainty budget
- f. Uncertainty of the model
- g. Summary



NEXT STEP: SIMPLIFICATION



- Assumption 1: We have the perfect model
- Assumption 2: We have the perfect accelerated test with same effective irradiance and perfect water concentration
- Assumption 3: Uniform material and uniform ageing processes
- Assumption 4: Orientation of product and end-use environment is known

Degradation rate:
$$\Delta P(H,T,H_2O) \sim E_{eff}^{\alpha} \cdot e^{-\frac{E_A}{RT}} \cdot [H_2O]^n$$



SUMMARY: TOTAL UNCERTAINTY



Uncertainies of perfect model:

Irradiance: ± 10%

Temperature: ± 5 K

Uncertainies of defined end-use environment:

Irradiance: ± 17%

Temperature: ± 2 K

$$\Delta P(H,T,H_2O) \sim E_{eff}^{\alpha} \cdot e^{-\frac{E_A}{RT}} \cdot [H_2O]^n$$

Uncertainies of **Evaluation criteria:**

± 10%

Uncertainies of accelerated testing:

Irradiance: ± 27%

Temperature: ± 13 K to 16 K

Summarized total uncertainty for perfect model and perfect test method:

Irradiance: \pm 54%; Temperature: \pm 20 - 23 K; Evaluation criteria: \pm 10%

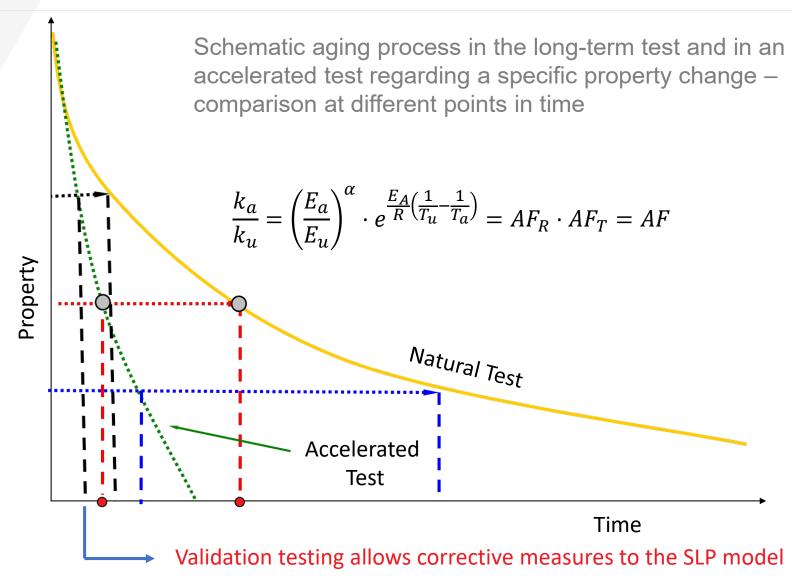
→ Total uncertainty easily in the 100% range





SLP BASED ON STRESS FACTORS (E,T)







IN CONCLUSION...



- Even with the perfect model and with the perfect test method there will be high uncertainties
- Does SLP make sense at all? Yes!!! By considering realistic test methods, optimizing the model and measuring the test parameters the uncertainties can be minimized
- SLP does not substitute validation
- ...but what is the right term:
 - Service Life Prediction?
 - Service Life Estimation?
 - Service Life Guess-timation?





THANK YOU!







Durability and Service Life Prediction in Building Envelope Materials: Knowledge Gaps and Path Forward

Marzieh Riahinezhad; PhD, PEng, PMP

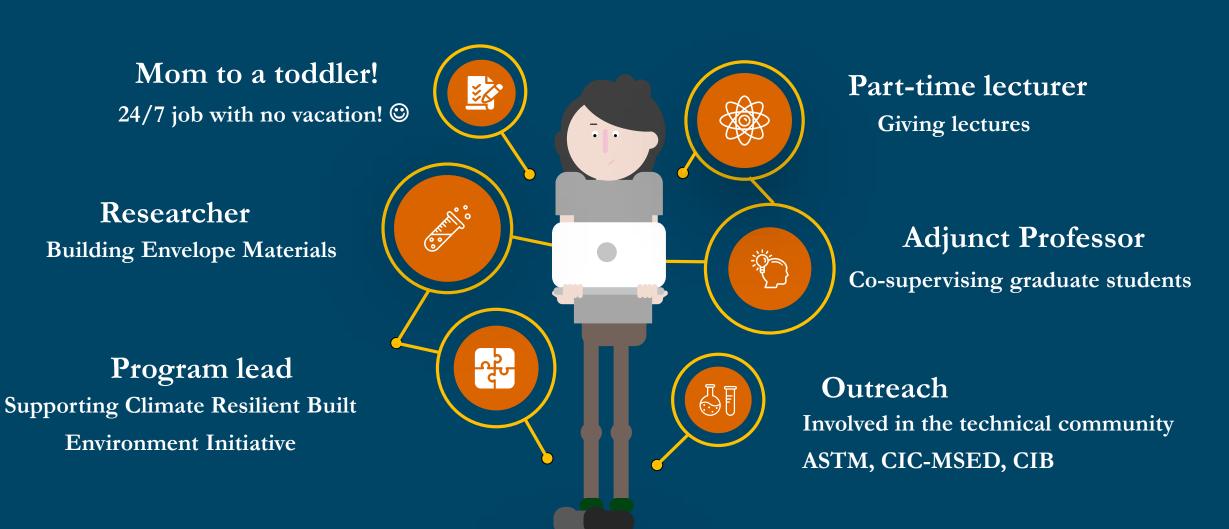
National Research Council of Canada

Workshop on Weathering and Durability Testing 2023





— Me at a Glance! —



National Research Council of Canada

BUSINESS INNOVATION

POLICY FOR GOVERNMENT

ADVANCING KNOWLEDGE 4,000

SCIENTISTS, ENGINEERS, TECHNICIANS, AND OTHER SPECIALISTS

179

BUILDINGS MANAGED in 22 locations

— National Research Council Canada

Digital Technologies

Emerging Technologies

Engineering

Life sciences

Transportation & Manufacturing

Energy, mining, & Environment

Ocean, Coastal, & River Engineering

CONSTRUCTION



Construction Research Centre



Building Envelope & Materials



Fire Safety



Civil Engineering & Infrastructure



Building Regulations



Intelligent Building
Operations



Technical & Testing
Services

Construction Material Evaluation and Durability Assessment Lab



Our core expertise:

- Durability assessment
- Accelerated laboratory aging
- Service-life prediction
- Product development and formulation
- Failure analysis
- Physico-chemical characterization









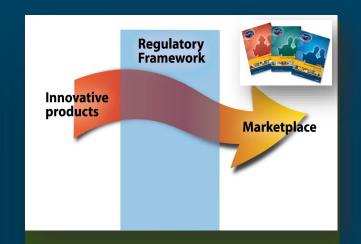




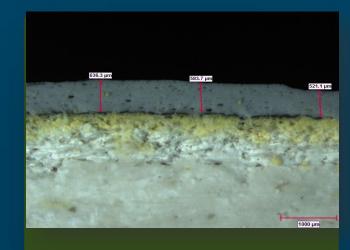
INNOVATIVE — CONSTRUCTION PRODUCTS

Qualification,
Performance
assessment &
Quality control

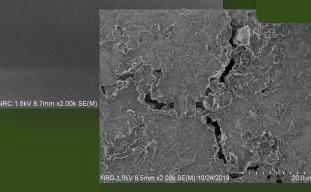
Undergoing projects with industry



Qualification
Canadian Construction
Materials Centre
(CCMC)



Quality Control



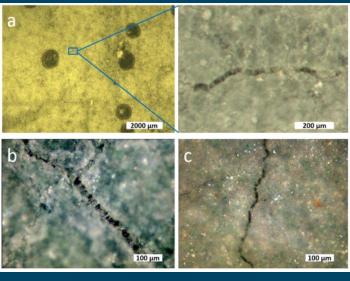
Performance
Assessment including
durability



DURABILITY
ASSESSMENT &
SERVICE LIFE
PREDICTION
(SLP)

Undergoing internal research projects





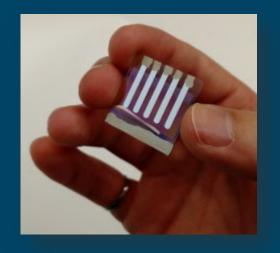
Field (natural) aging of sealants

Accelerated aging of sealants

SLP method is most accurate when used in conjunction.



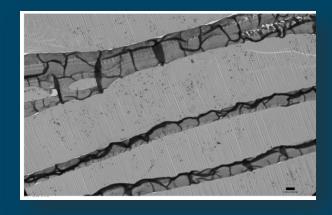
DURABILITY
ASSESSMENT
& SERVICE
LIFE
PREDICTION
(SLP)



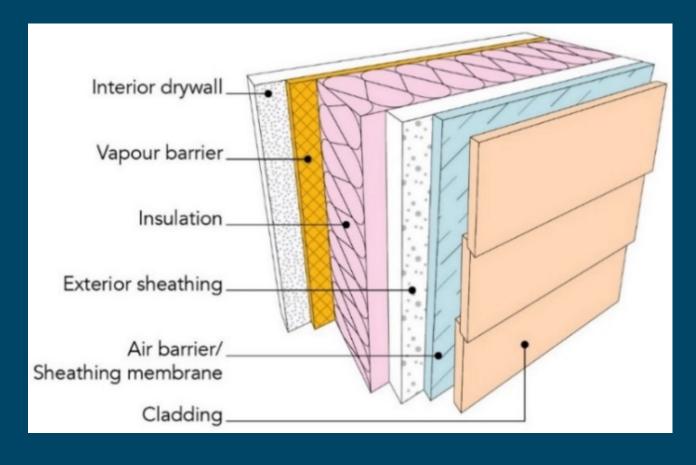
Weather resistance organic photovoltaics (collaboration with University of Toronto)

Undergoing projects with academia

Long-term performance of silicon dioxide reinforced wood substrates (collaboration with University of Ottawa)



Durability and Service Life Prediction in Building Envelope Materials (BEMs)



Typical multi-layer building envelope

— Degradation factors for BEMs

- Mechanical: vibrations, gravitation, deformations, impact
- Thermal: high & low temperatures and cyclic fluctuations
- Chemical: water, solvent. Oxidisers, acids, bases
- Electromagnetic: solar radiation, eclectic current
- Biological: fungi, microbial growth, animal-related erosion

— Environmental loads

	Cladding	Vapour/Air Barrier	Insulation	Sealants	Fenestration	Gaskets	
UV Radiation	Sunlight (I,S)	Sunlight (I)	Sunlight (I)	Sunlight (I,S)	Sunlight (I,S)	Sunlight (I,S)	
Moisture	Rain, snow, ground water, dew. (I,S)	AB: rain, snow, dew VB: showers, tap (I,S)	Rain, snow, ground water, dew, taps (I,S)	Rain, snow, ground water, dew (I,S)	Rain, snow, ground water, dew (I,S)	Rain, snow, ground water, dew (I,S)	
Thermal	Hot/cold weather, heating/cooling (I,S)	Hot/cold weather, heating/cooling (I,S)	Hot/cold weather, heating /cooling (I,S)	Hot/cold weather, heating /cooling (I,S)	Hot/cold weather, heating /cooling (I,S)	Hot/cold weather, heating/cooling (I,S)	
Mechanical	Wind, expansion/ contraction (I,S)	AB: Wind, Air Pressure, (I,S)	Thermal cycles and gradients (I,S)	wind, expansion/ contraction (I,S)	wind, expansion/ contraction (I,S)	Wind, expansion /contraction (I,S)	
Biological	Fungus, rodent (I,S)	N/A	Fungus (S)	Fungus, rodent(I,S)	Fungus (I,S)	N/A	
Combined	A11	All except biological	A11	A 11	A 11	All except biological	

I: Installation

S: Service

Service Life Prediction (SLP) in BEMs



Field aging

- Field exposure sites
- Experimental buildings
- Building inspections



Lab accelerated aging



Modelling



— Durability of BEMs

Assumed lifespan of some plastics

Type	Assumed Lifespan (Years)	Building Components		
Butyl Rubber	2 to 35	Gaskets/Sealants		
Polyethylene, Polypropylene	2 to 15	Air/Vapour Barriers, Cladding		
Polyurethane	7 to 10	Cladding, Insulation Air/Vapour Barrier,		
Polyvinyl Chloride	8 to 30	Cladding		
Silicone	14 to 50	Air/Vapour Barrier, Sealants		

Design Service Life (DSL) for buildings

DSL Category	Range of DSL (Years)
Short life	Up to 10
	10 to 25
Medium life	25 to 50
	25 to 99
Long life	50 to 99
Permanent	100 to 300

Ref.: Berge, B. The Ecology of Building Materials, Architectural Press & Elsevier: 2009

Ref.: CSA S478:19 Durability in Buildings; 2019

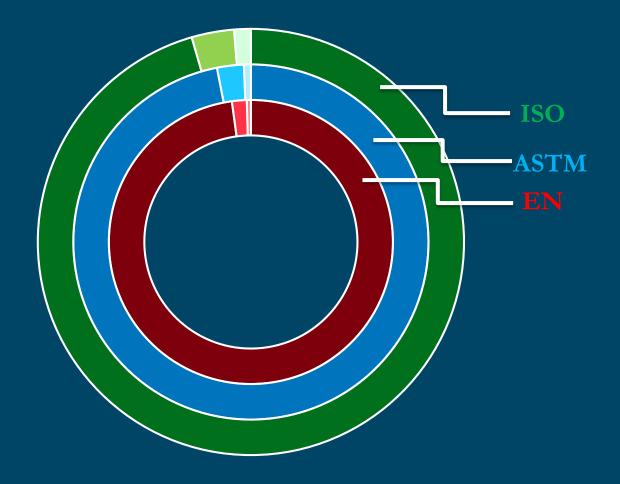
— Reference Service Life

- Sparce information on Reference Service Life (RSL) values. Development a database of RSLV for BEMs:
 - Historical performance as reported by building professionals
 - Experimental work involving accelerated aging, e.g., ASTM C1850-17
 - Historical environmental load assessment and future climate predictions
 - Assessments of degradation rates and mechanisms
 - SLP modelling to establish a RSLV and validate the models



—Review of Existing Standards on Durability & SLP

Standard body	# Standards on construction (ICS 91)	Standards on durability, SL, accelerated ageing	Standards on durability, SL, accelerated ageing of BEMs
EN	5351	2%	0.5%
ASTM	3101	2%	1%
ISO	1748	3%	1%



CONCLUDING REMARKS Developing performance based standards Standards Including all BEMs in SLP PSY Stp Chinate change Developing a database of RSL for Considering climate

change

BEMs

Thank You

Marzieh Riahinezhad Researcher and Adjunct Professor

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Developing Methodology and Online Tools for Service Life Prediction of Polymers used in Photovoltaics and Infrastructure



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Infrastructure Materials Group NIST Engineering Laboratory Gaithersburg, Maryland

**Department of Statistics Virginia Tech Blacksburg, VA

Workshop on Weathering and Durability Testing 2023 June 28, 2023 **Denver, Colorado, USA**

*Email address: xiaohong.qu@nist.gov

NIST 2m SPHERE



NIST 6-Port SPHERE



Outline



- Brief introduction of the reliability-based methodology for service life prediction (SLP) of polymers
- An example of SLP based on a pigmented UV-stabilized PET material used for PV backsheets
 - Accelerated laboratory exposure (SPHERE)
 - Outdoor exposure (FL, AZ, MD)
 - Statistical modeling for linking laboratory and outdoor exposure results towards SLP
- Introduction of an online SLP tool based on Python Shiny (SLPS) (under development, by NIST and Virginia Tech)

Methodology for Service Life Prediction of Polymers NUST

Reliability-based Methodology

Laboratory Exposure

Effects of key environmental factors on polymer degradation (UV (I, λ), T, RH, σ/ϵ , ...)

Failure mechanism

Outdoor Exposure

(UV Spectral irradiance, T, RH recording)



To build physical-based statistical predictive model

Quantitative linkage based on modeling

To validate the predictive models using outdoor conditions as input

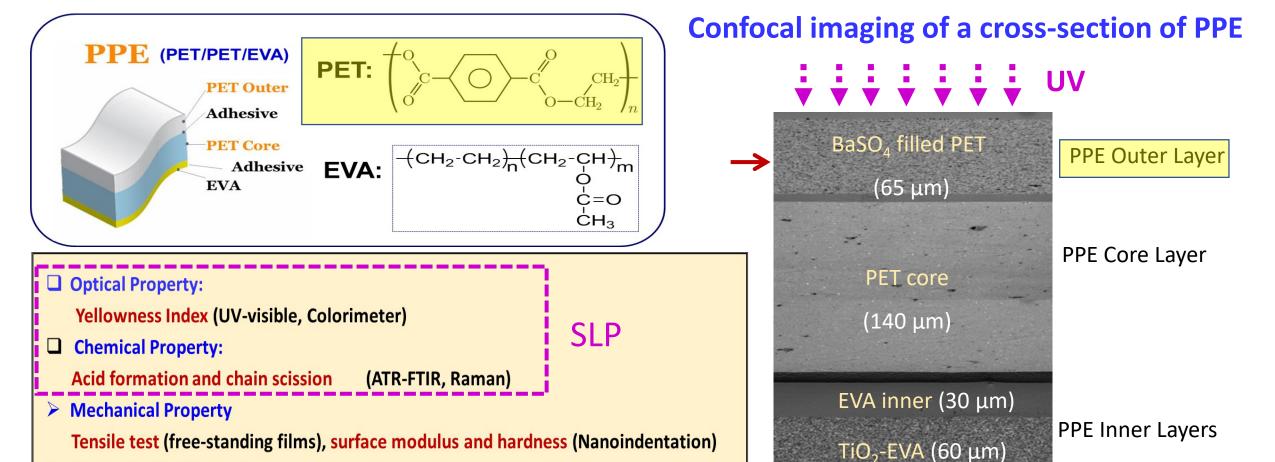
- Based on Effective Dosage Model and Cumulative Damage Model
- The parameters of predictive models are estimated solely from laboratory experiments

 Predicted outdoor results are used to verify the models using field exposure conditions as input.

To predict the service lives of polymers

A Pigmented PET Material used for PV Backsheet - PPE (PET/PET/EVA)





EVA outer (30 μm)

Solar Energy Materials and Solar Cells (2016); Prog Photovolt Res Appl. (2018); IEEE PVSC Proceedings (2019, 2020, 2021)

Cross-sectional Chemical, Optical, Mechanical Characterization (Failure Mode Analysis)

Morphological Property

(AFM, Confocal Microscopy, SEM)

Laboratory Exposure of PPE: NIST SPHERE with Simultaneous UV/T/RH



Air side (PET outer layer)

NIST 2m SPHERE



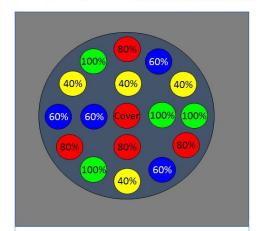
- Simulated Photodegradation via High Energy Radiant Exposure (SPHERE)
- High power metal halide lamps
- Low wavelength < 295 nm removed, and most visible and infrared radiation removed
- Individually controlled environmental chamber (UV, RH,T)

SPHERE Exposure Condition

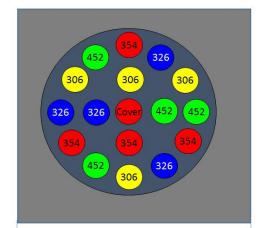
RH Temp	0 %	30 %	60 %
45 °C	X R		
65 °C	X R, W	X (not yet)	
75 °C	X R, W		
85 °C	X R, W	X (not yet)	X R, W

- > UV Intensities (I): 0, 40, 60, 80 and 100 % (ND filters) Reciprocity
- ➤ Wavelengths (WL): 306, 326, 354, 452 nm (band pass filters) Wavelength

1) Reciprocity Study: Neutral Density filters



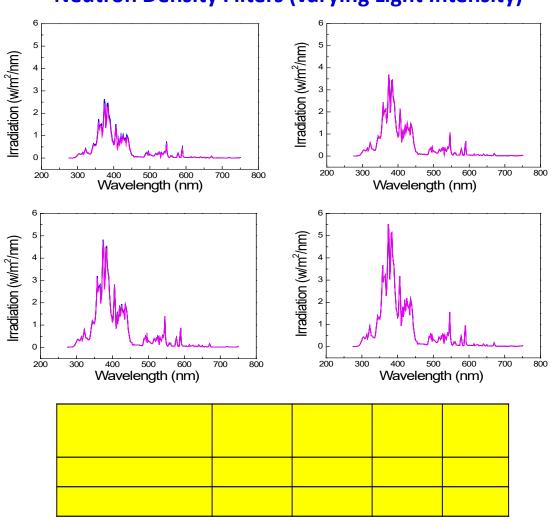
2) Wavelength Study: Band pass filters



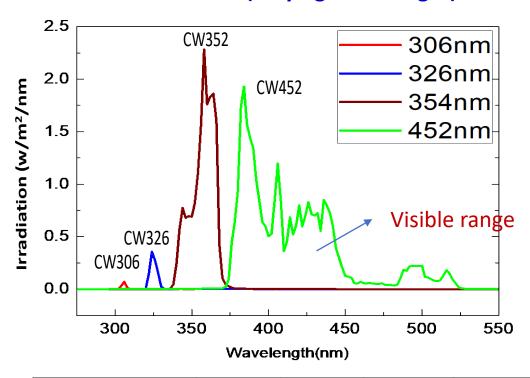
<u>Spectral Irradiance</u> through Filters on PPE Samples (Laboratory Exposure)



Neutron Density Filters (varying Light Intensity)



Band Pass Filters (varying Wavelength)

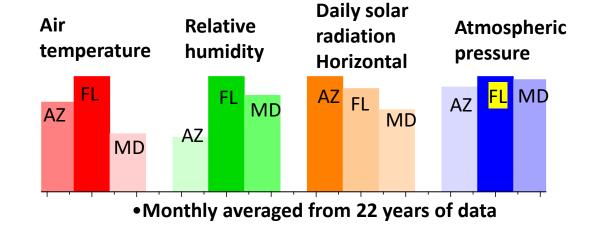


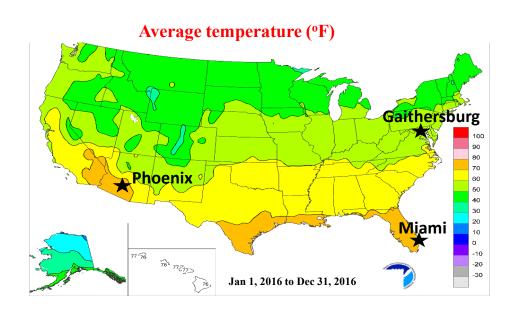
Irradiance through BP Filters (W/m²)	306 ± 3	326 ± 6	354 ± 19	452 ± 80
	(nm)	(nm)	(nm)	(nm)
295-385 nm	0.33	2.25	38.2	36.8

Outdoor Exposure of PPE: 3 Different Climates (AZ, FL, MD)



- > Arizona hot, arid
- > Florida hot, humid
- Maryland humid continental
 - Open rack mount
 - Closed box mount





Rack exposure site in Florida NIST rooftop rack exposure in MD





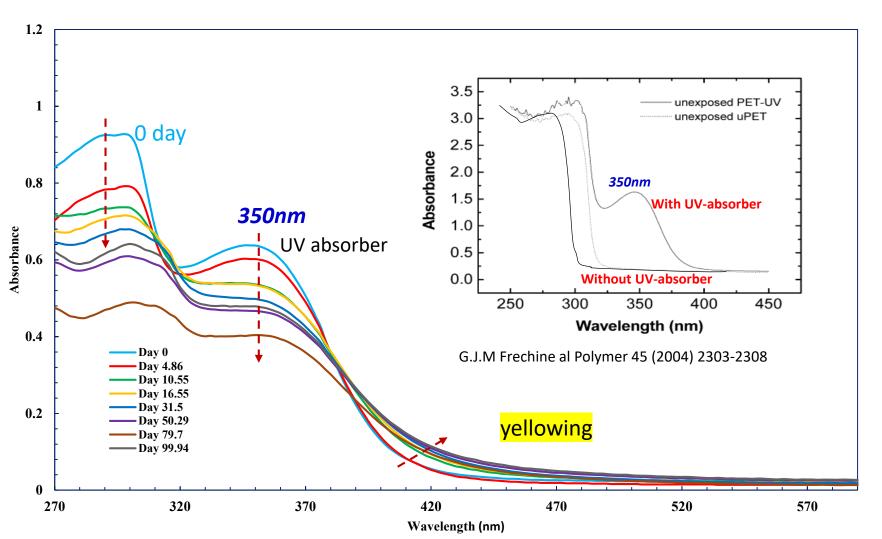
Tilt at latitude angle

http://www.cpc.ncep.noaa.gov/products/analysis_monitoring/regional_monitoring/us_12-month_avgt.shtml

https://eosweb.larc.nasa.gov/cgi-bin/sse/retscreen.cgi?&email=rets@nrcan.gc.ca&step=1&p=&lat=33.4484&submit=Submit&lon=-112.0740

UV-Vis Spectra of PET Outer Layer Exposed to UV/85 °C/ 0% RH at Different Times



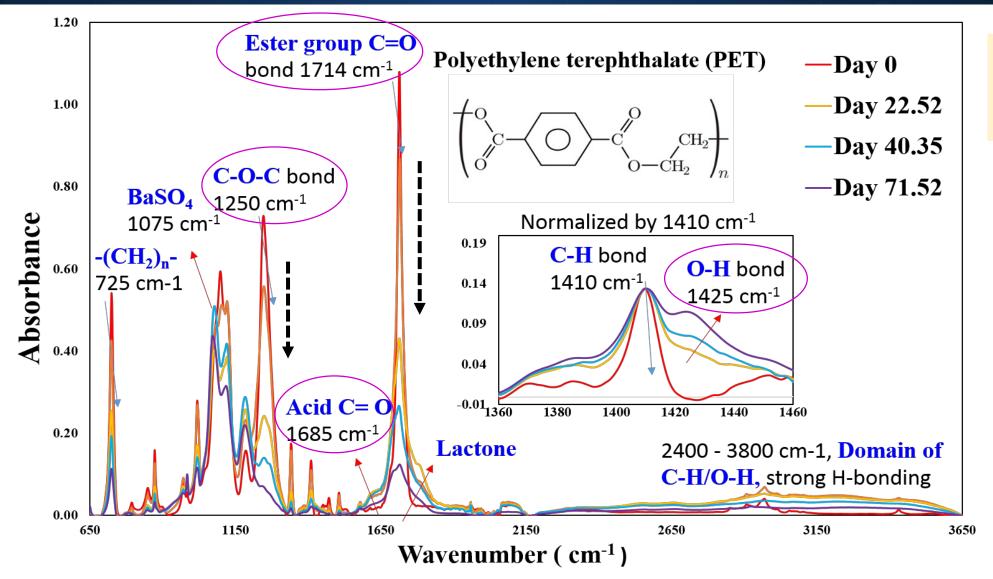


UV absorbance at 350 nm for unexposed PPE indicates the presence of UVA.

However, this peak can't be used to monitor UVA amount due to the influence of fluorescence after degradation.

ATR-FTIR Spectra of PET Outer Layer Exposed to UV/85 °C/0% RH at Different Times



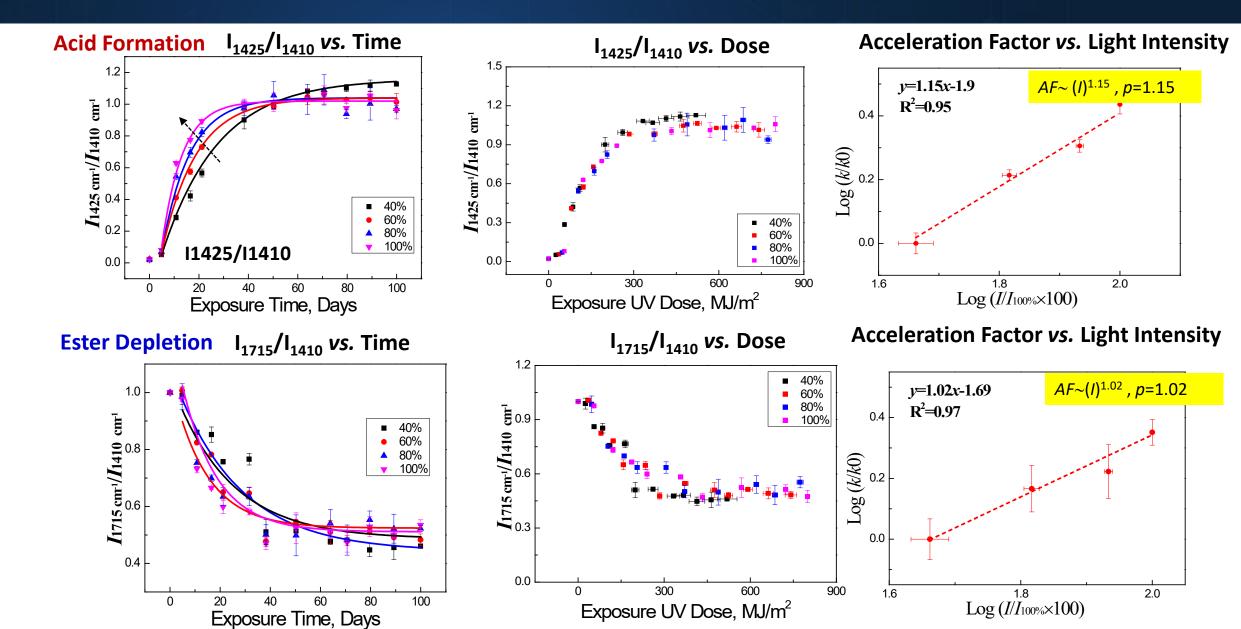


Ester Depletion
I₁₇₁₄(C=O) /I₁₄₁₀ (C-H)

Acid Formation
I₁₄₂₅ (O-H) /I₁₄₁₀ (C-H)

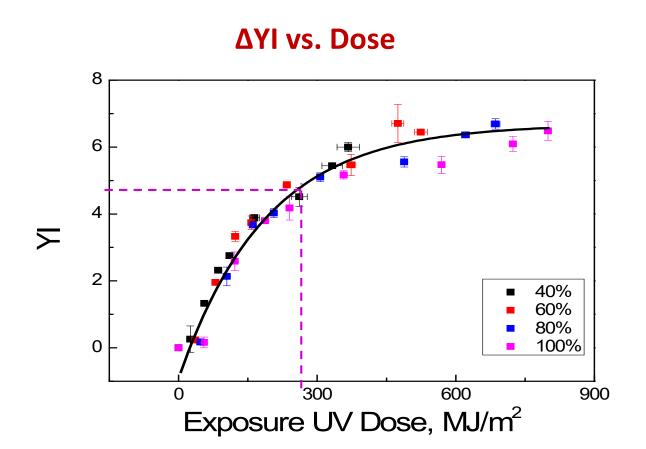
Effect of Light Intensity on Chemical Changes at UV/85 °C/0%RH



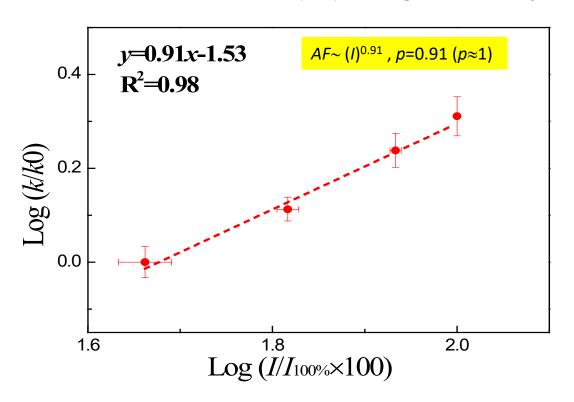


Effect of Light Intensity on ΔYI at UV/85 °C/0%RH





Quantitative Validation of Reciprocity Law for YI Acceleration Factor (AF) vs. Light Intensity

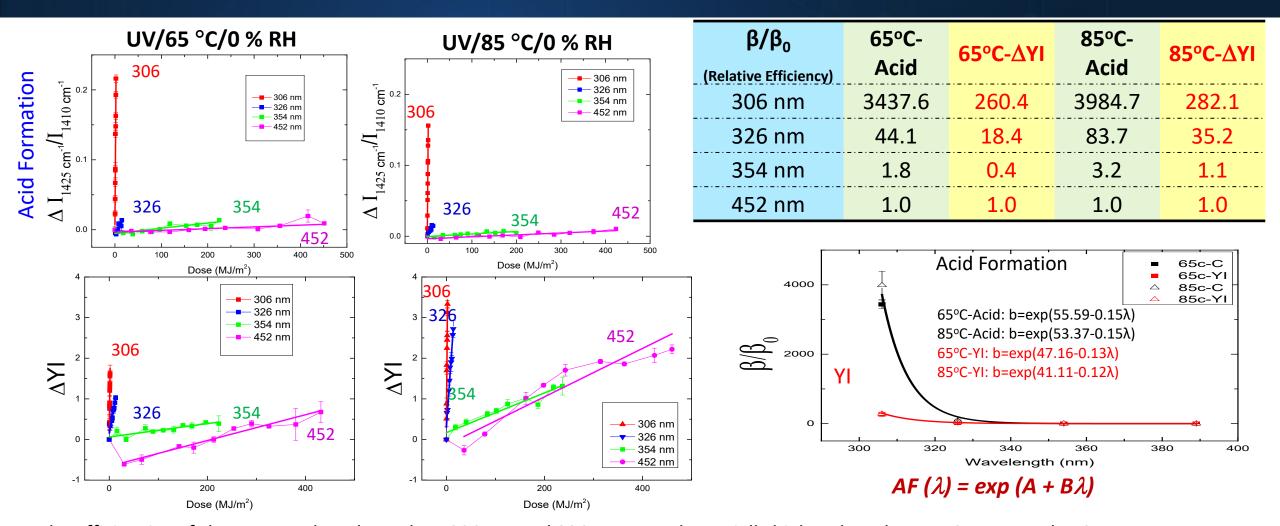


To achieve the same damage, the required dosage is similar at different UV intensities.

ho Reciprocity Law is generally obeyed for YI and chemical changes of PPE outer layer under UV/85 lpha/0 lpha RH.

Effect of Wavelength on PPE Chemical and optical Degradation

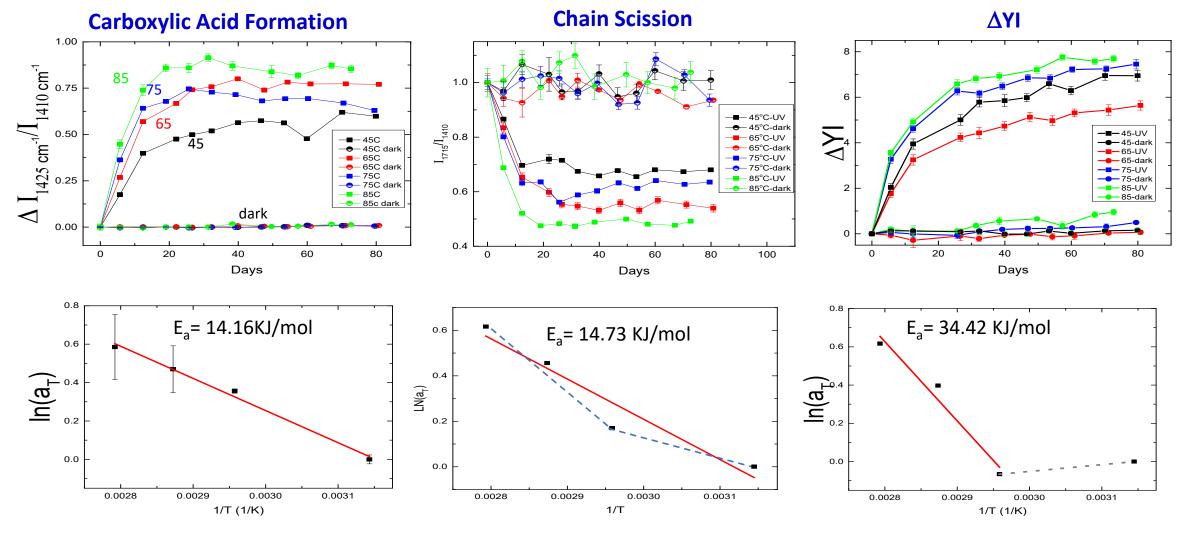




- The efficiencies of shorter wavelengths such as 306 nm and 326 nm are substantially higher than those at 354 nm and 452 nm.
- Photobleaching has been observed at both 65°C and 85°C for 452 nm.
- The exponential dependence between efficiency and wavelength appears to be common for optical and chemical degradation of PET outer layer. Different degradation modes have different function parameters.

Effect of Temperature on PPE Chemical and Optical Degradation

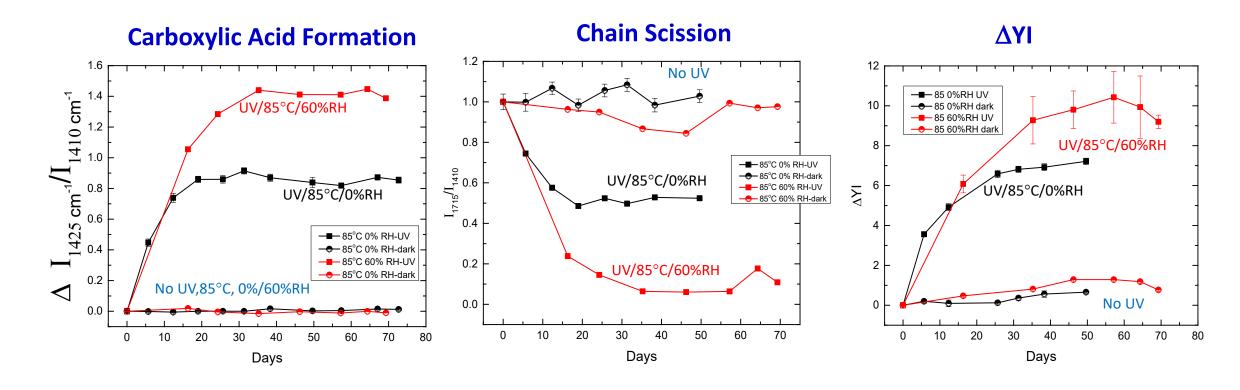




The temperature dependence for the chemical degradation generally follows the Arrhenius Law, but yellowness index doesn't appear to follow.

Effect of RH on PPE Chemical and Optical Degradation



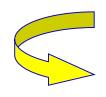


- High RH accelerates both chemical and optical degradation.
- No obvious degradation has been observed in dark condition.
- Synergistic effect has been observed between UV and moisture.

Results from NIST SPHERE Exposure



- Effect of light intensity (40%, 60%, 80%, 100%)
 - Reciprocity study
- Effect of UV wavelength (306nm, 326nm, 354nm, 452nm)
 - Action Spectra
- **Effect of temperature** (45 °C, 65 °C, 75 °C, 85 °C)
- Effect of relative humidity (RH) on PPE degradation (0 % RH vs. 65 % RH)



Statistical Predictive Models

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

How to Use Laboratory Accelerated Exposure Results to Predict Outdoor Performance?



➤ Building Predictive Models based on SPHERE Exposure Results

EVD ID	TEMD	DII	DEN	CAMPLE	DD	CAMPLE	EVD ID	TEMD	DII	DEM	CAMBLE	DD	CAMPLE
EXP_ID		_	DEN	SAMPLE	BP	SAMPLE	EXP_ID	TEMP	RH	DEN	SAMPLE	BP	SAMPLE
45_0_R	45	0	40%	8, 11, 14, 17	-	-	75_0_W	75	0	100%	2, 3, 4, 5	-	-
	45	0	60%	7, 10, 13, 16	-	-		75	0	-	-		8, 12, 16
	45	0	80%	6, 9, 12, 15	-	-		75	0	-	-	326	9, 13, 17
	45	0	100%	2, 3, 4, 5	-	-		75	0	-	-	354	6, 10, 14
								75	0	-	-	389	7, 11, 15
65_0_R	65	0	40%	2, 8, 12, 16	-	-							
	65	0	60%	3, 9, 13, 17	-	-	85_0_W	85	0	100%	2, 3, 4, 5	-	-
	65	0	80%	4, 6, 10, 14	-	-		85	0	-	-	306	8, 12, 16
	65	0	100%	5, 7, 11, 15	-	-		85	0	-	-	326	9, 13, 17
								85	0	-	-	354	6, 10, 14
65_0_W	65	0	100%	2, 3, 4, 5	-	-				-	-	389	7, 11, 15
	65	0	-	-	306	8, 12, 16							
	65	0	-	-	326	9, 13, 17	85_60_R	85	60	40%	2, 8, 12, 10	-	-
	65	0	-	-	354	6, 10, 14		85	60	60%	3, 9, 13, 17	-	-
	65	0	-	-	389	7, 11, 15		85	60	80%	4, 6, 10, 14	-	-
								85	60	100%	5, 7, 11, 13	-	-
75_0_R	75	0	40%	8, 11, 14, 17	-	-							
	75	0	60%	7, 10, 13, 16	-	-							
	75	0	80%	6, 9, 12, 15	-	-							
	75	0	100%	2, 3, 4, 5	-	-							



The total samples are

- 112 under 7 conditions
- 98 used for model training
- 14 used for model testing (marked in red)

Building Physics-based Predictive Model - Effective Dosage Models



- The wavelength-specific intensity is $I(\lambda) = E(\lambda) \times F(\lambda)$.
- To incorporate the effect of wavelength and intensity, we introduce the idea of effective dosage.
- The usual dosage is computed as $d(t) = \int_0^t \int_{\lambda} I(\lambda) d\lambda d\tau$.
- The effective dosage is modeled as

$$s(t) = \int_0^t \int_{\lambda} [I(\lambda)]^p \phi(\lambda) d\lambda d\tau = t \times \int_{\lambda} [I(\lambda)]^p \phi(\lambda) d\lambda$$

- The effect of wavelength is $\phi(\lambda) = \exp[\beta(\lambda 354)]$, log-linear relationship. Here, we use the 354nm as the baseline. That is the acceleration factor at 354nm is one.
- The effect of intensity is $[I(\lambda)]^p$, power law relationship.

Statistical Model for Degradation Path



The model for degradation measurements is

$$y_{ij} = D(t_{ij}) + \epsilon_{ij}.$$

- $\epsilon_{ij} \sim N(0, \sigma_{\epsilon}^2)$ is the error that can not be captured by $D(t_{ij})$.
- The model used for an increasing degradation path is

$D(t) = \frac{A}{1 + \exp\left\{-\frac{\log[s(t)] - \mu - \beta_t \frac{11605}{\text{TempC} + 273.15} - \beta_r \log(1 + \text{RH})}{\sigma}\right\}}$ $\textit{Effective dosage} \quad s(t) = t \times \int_{\lambda} [I(\lambda)]^p \underline{\phi(\lambda)} d\lambda$ $\textit{Wavelength effect} \quad \phi(\lambda) = \exp[\beta(\lambda - 354)]$

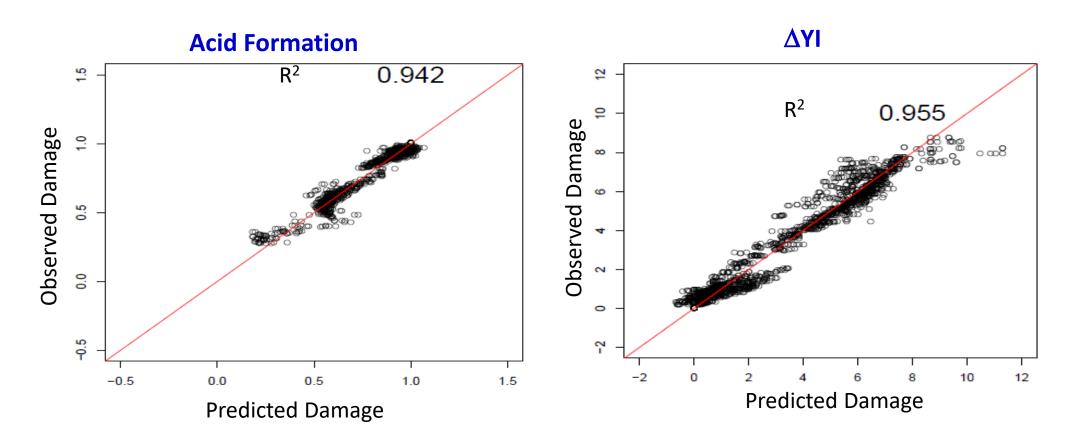
Parameter Estimation for ΔYI

(estimated solely from SPHERE results)

Parameter	Interpretation	Value
Α	ultimate degradation	16.297
$\eta = \exp(\mu)$	half-degradation dose	310.132
$\gamma = 1/\sigma$	steepness	0.458
β	wavelength effect	-0.147
р	intensity effect	1.03
eta_{t}	temperature effect	0.192
β_{r}	RH effect	-1.724
σ_{ϵ}^2	error variance	0.390

Comparing <u>Predicted Damage</u> with <u>Observed Damage</u> for All Laboratory Data





The predictive model fits reasonably well with degradation of PPE exposed on the SPHERE at different environmental conditions.

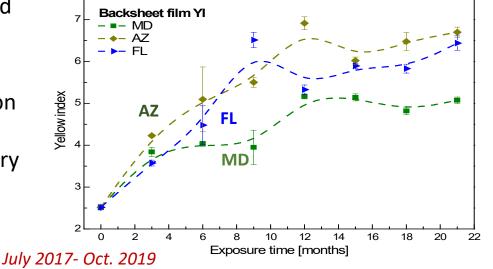
Using Outdoor Results to Validate the Predictive Models



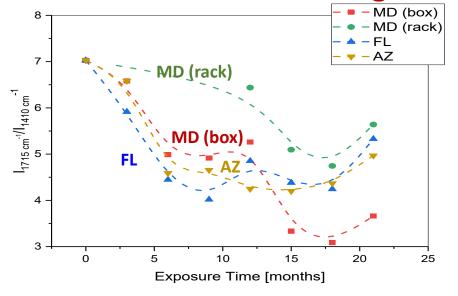
Sample measured every 3 months

 Similar degradation modes observed between laboratory and outdoor

Outdoor Yellowness Index

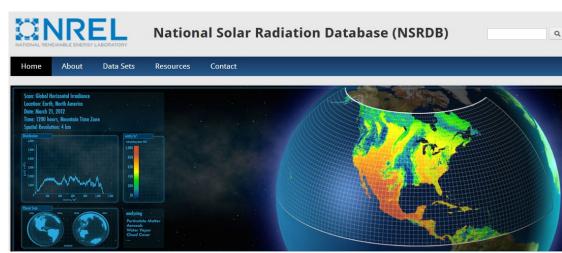


Outdoor Chemical Changes



☐ Outdoor Exposure Conditions

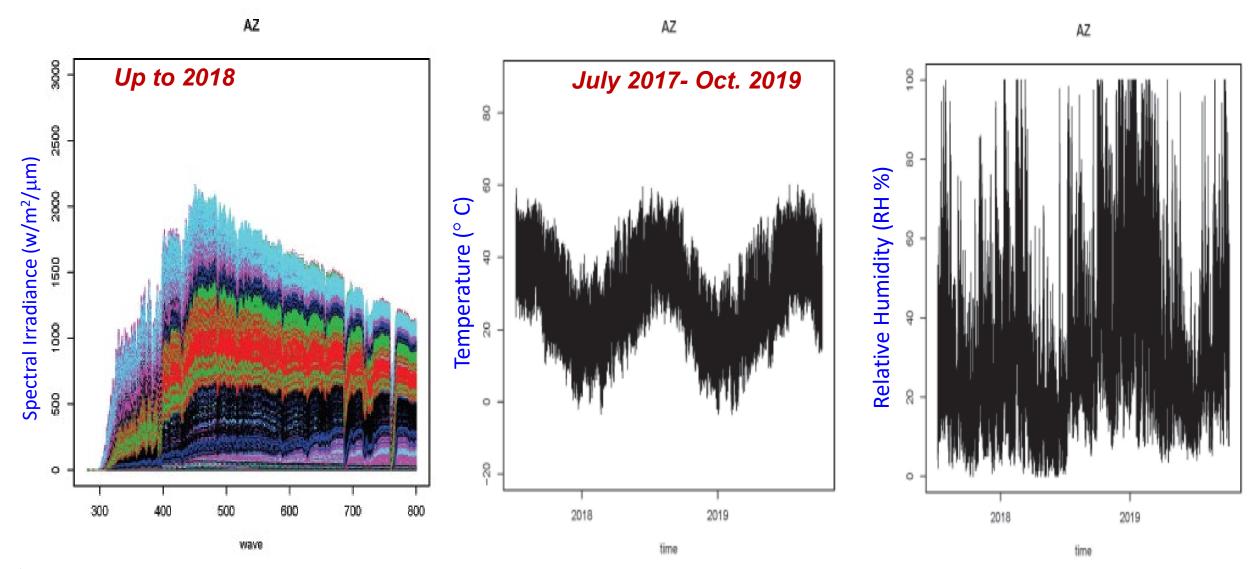
- Specimen T, RH: measured by panel-mounted iButtons
- Spectral UV Irradiance: <u>EKO</u> (226 Roof, limited data); <u>NREL National Solar Radiation Database</u> (Global spectral irradiance for tilted surfaces, only up to 2018 available; With Aron Habte's help)



Time-Varying Spectral Irradiance, Temperature, RH for Outdoor Samples



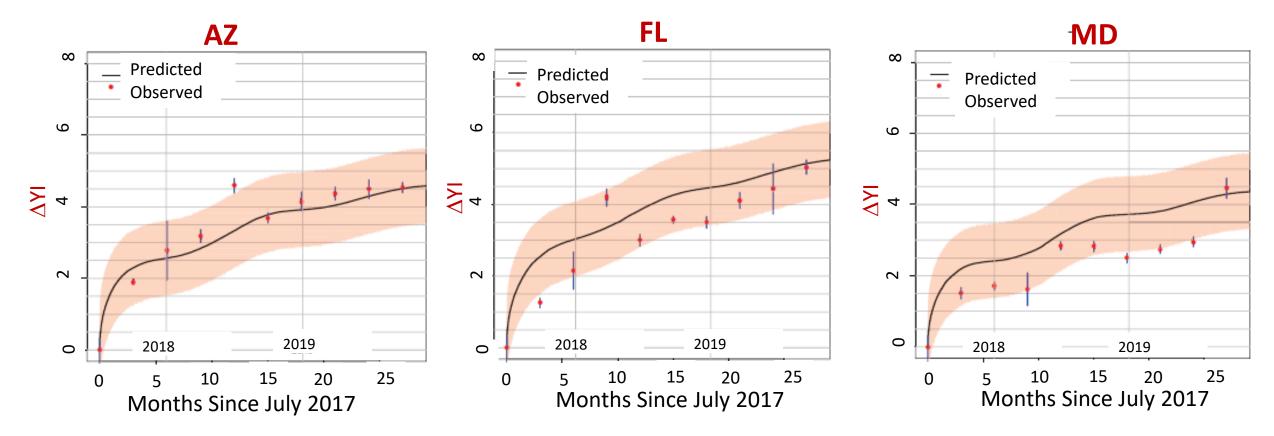
(as input for predictive models to estimate outdoor damage)



Time-varying outdoor damage prediction: Using cumulative damage model and predictive models built on laboratory exposure data.

Predicted Damage (YI) vs. Observed Damage as a Function of Time for Outdoor Exposed PPE





- The shaded area shows the 90% statistical interval for uncertainty quantification.
- Gap between predicted and observed damage could be due to
 - 1) incomplete data for considering RH effect (0%, 60%) on modeling
 - 2) effects of rain, condensed water and physical erosion lacking in the laboratory conditions

Wtothyation of Ars Pplinkish Tool (SLP Shiny, SLPS)

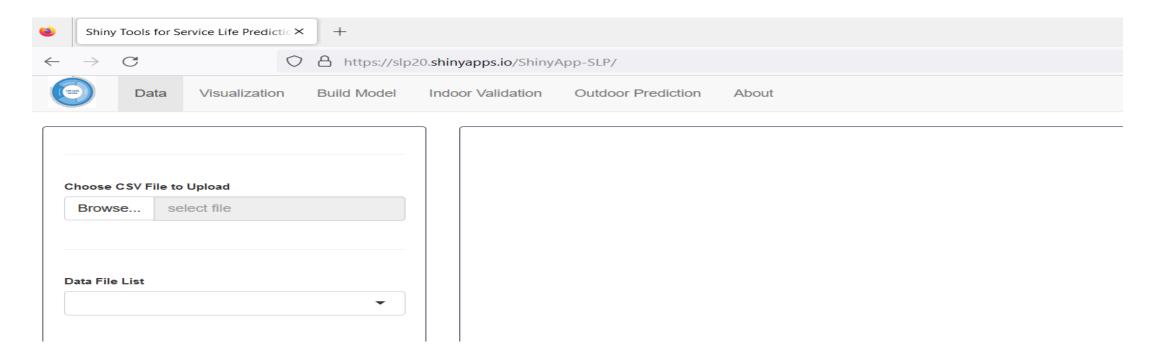


- Over the years, NIST has worked with Virginia Tech (Prof. Yili Hong, previously Dr. meeker from Iowa State U.) to develop reliability-based methodology for SLP of polymers by linking the accelerated laboratory and outdoor results using statistically mathematical models.
- Those methods are implemented in **R**, (https://www.r-project.org/), which is computing language used by many statisticians and machine learning researchers.
- To use R, one needs to write program code, which can be inconvenient for researchers and engineers working on SLP.
- Shiny is a package that makes it easy to build interactive web apps straight from R and Python.
- Thus, we are motivated to build a web-based software tool for SLP, which we call it service life prediction with shiny (SLPS).

SLPS Test Version



- An alpha test version for SLPS has been developed for about two years.
- It can be assessed at https://slp20.shinyapps.io/ShinyApp-SLP/
- The test version shows the possibility of using shiny for SLP.
- A screenshot for the interface is shown below.

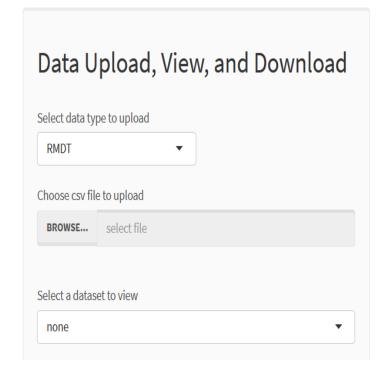


SLP Shiny (SLPS) Current Development



- A more comprehensive version of SLPS is currently under development.
- It contains five main function tabs:
- Data data operations
- Figure interactive visualization
- Analysis modeling and prediction
- Report automatic reporting
- Help examples and manual

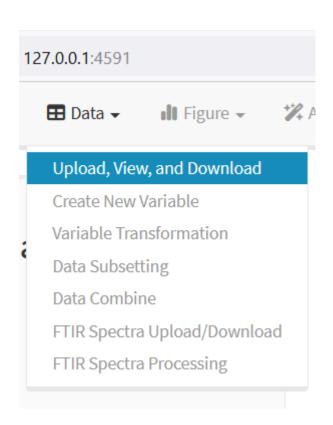


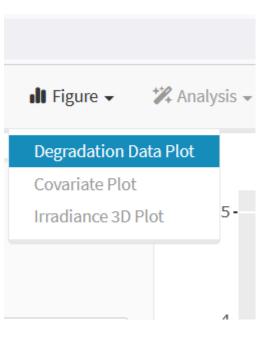


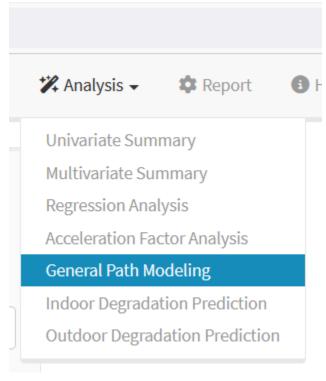
Overview of Functionalities

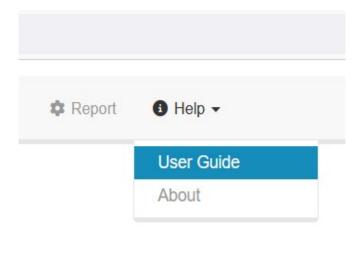
Under each tab, there are menu items







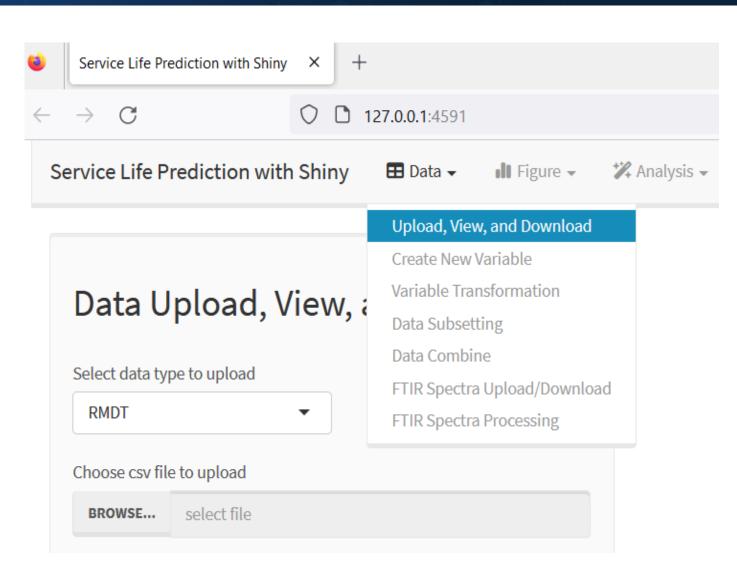




Data Tab



- The data tab provides functions for data upload, download, and view.
- It also has functions for basic data operations for new variable, variable transformations, data subsetting, and merge
- With combination of those basic functions, most data preparation can be done for the subsequent analysis.



FTIR Data Preprocessing



• The FTIR menu can process the FTIR spectra in batch, with interactive plots.

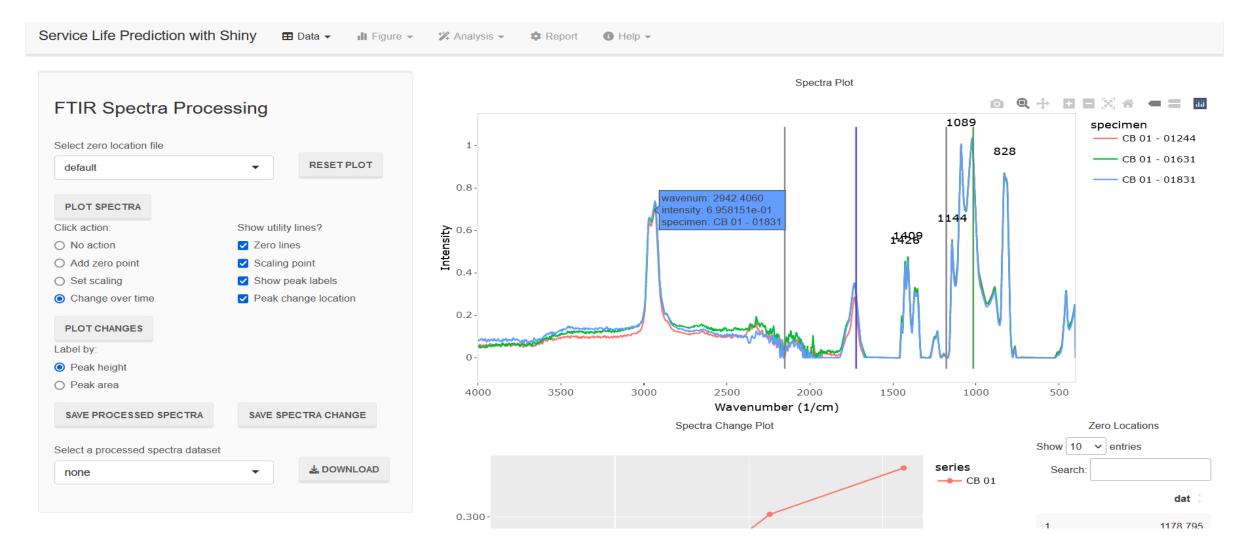


Figure Tab – Degradation Data Plot



Interactive plot for degradation data visualization.

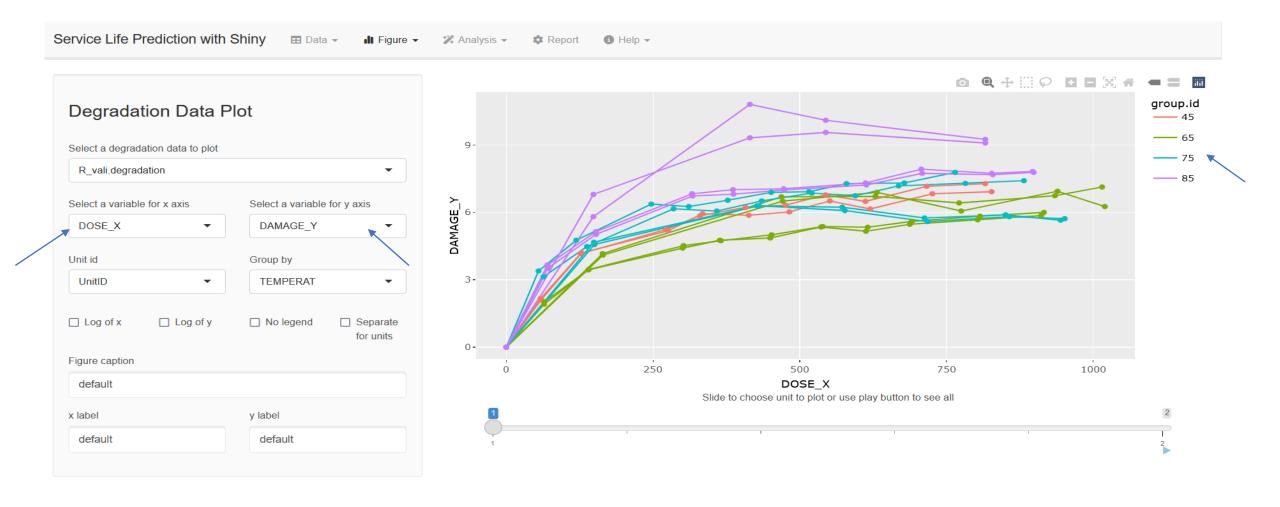


Figure Tab – Covariate Plot



• Interactive plot for covariate data visualization.

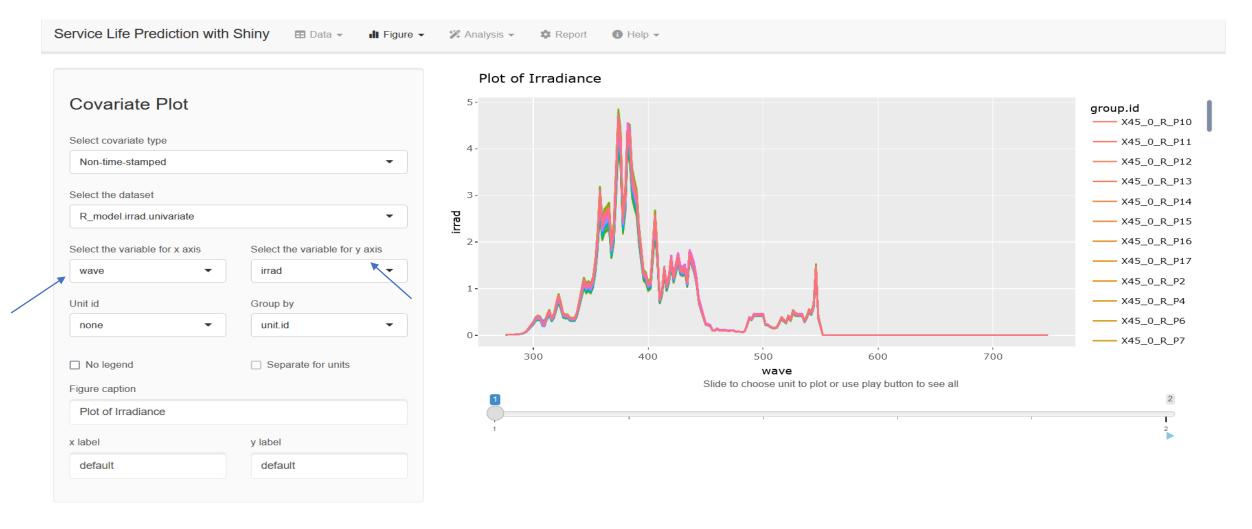
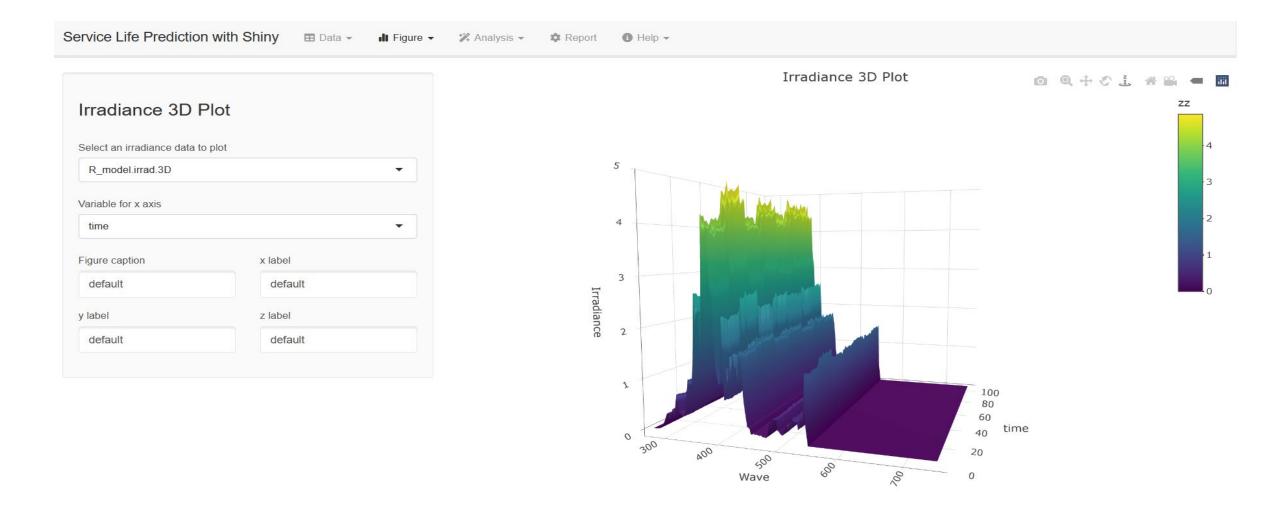


Figure Tab – 3D Plot

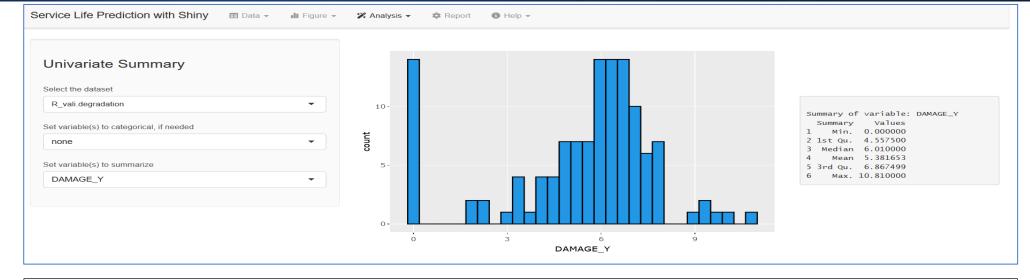


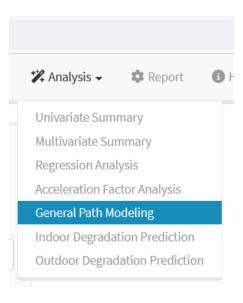
• 3D plot for irradiance as a function of time and wavenumber.

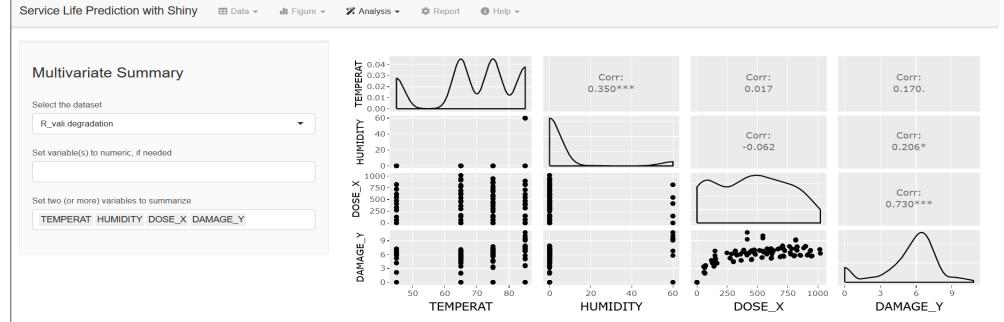


Analysis Tab: Basic Statistical Analysis Tools

 Summary of a variable and correlation of multiple variables.



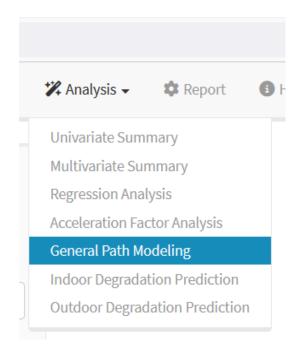


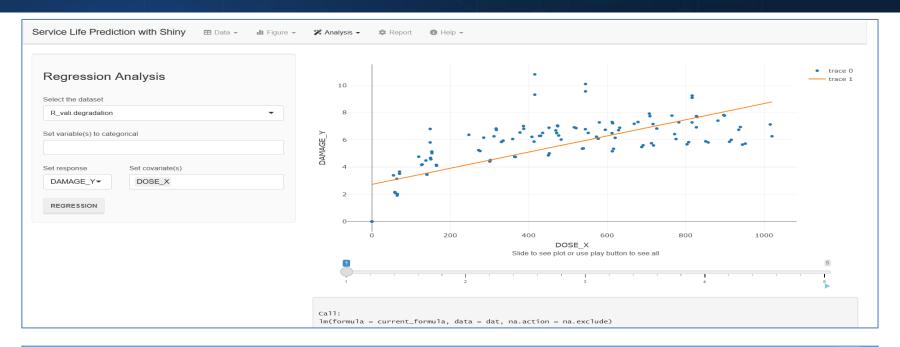


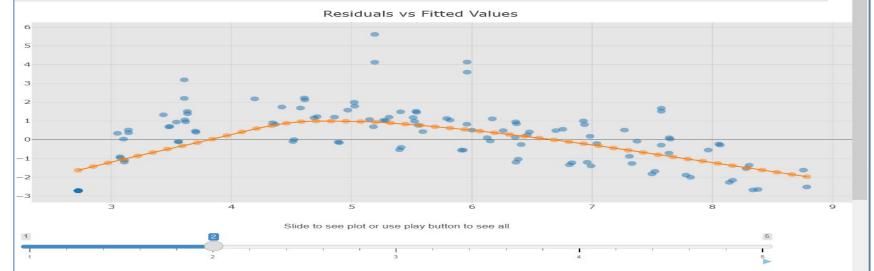
Analysis Tab: Linear Regression



 Ordinary linear regression and visualization of the results.

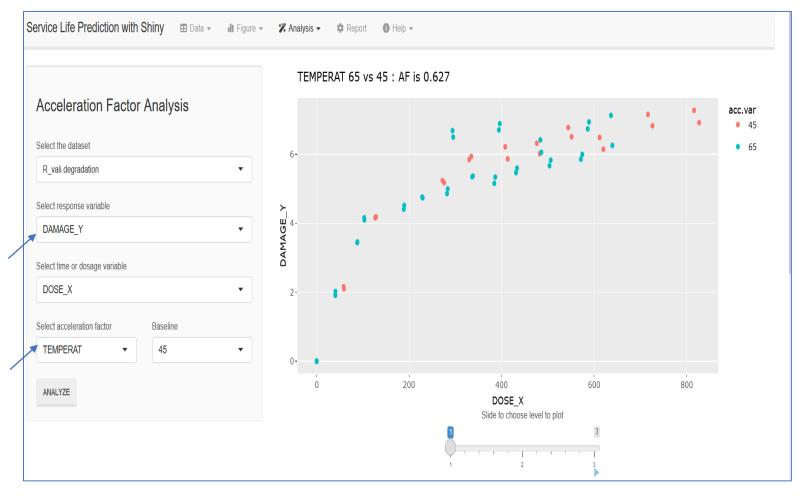


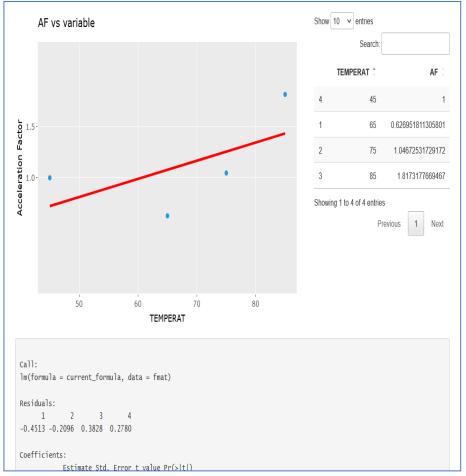




Analysis Tab: Acceleration Factor Analysis NUST

Acceleration factor estimated by super-imposing.

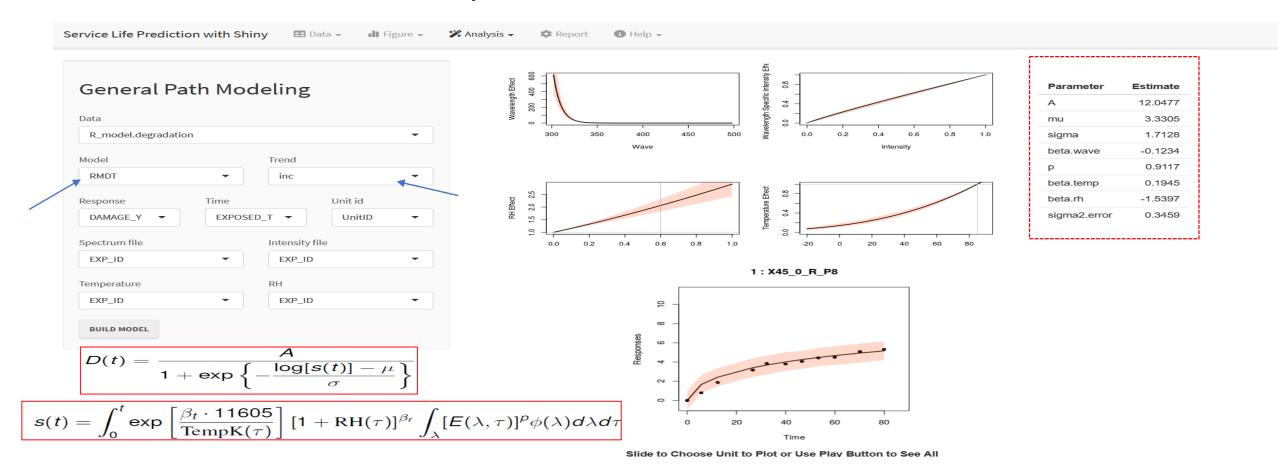




Analysis Tab: General Path Modeling (under development)



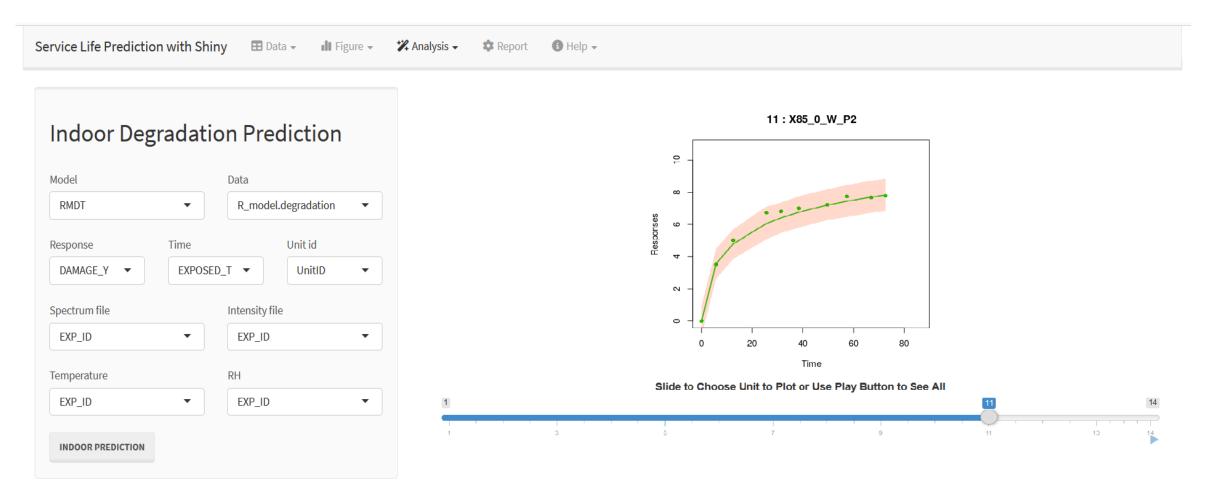
• Fit a general path model to different kinds of degradation data, such repeated measures data, destructive data, with or without covariates. It can handle irradiance data or dose only data.



Analysis Tab: Indoor Prediction



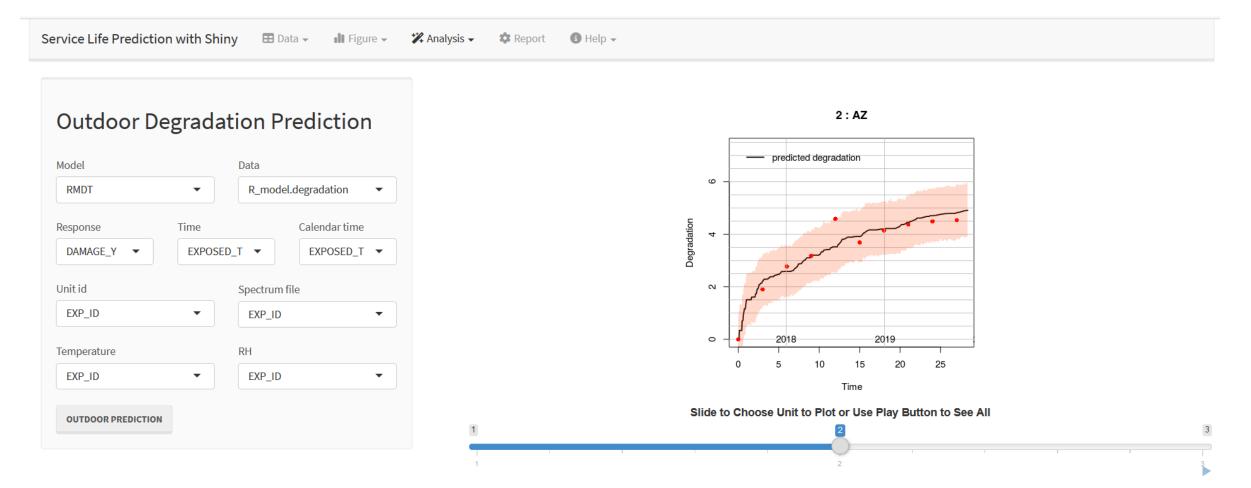
 Based on the model fitted, we can generate prediction for other indoor units with different conditions.



Analysis: Outdoor Prediction



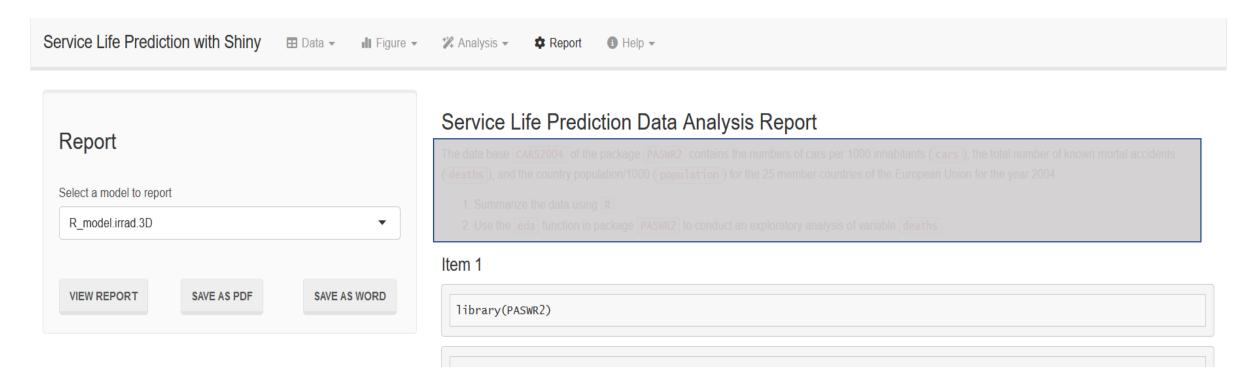
• Based on the model fitted, we can generate prediction for outdoor units with time-varying environmental conditions.



Report Tab (under development)



- The report function can automatically summarize the data analysis.
- It can give summary of the data, model equations, parameter estimates, visualization of the data in figures and tables.
- It can be saved as in pdf or word format.



Help Tab



The help tab gives examples and manual for the software.

Service Life Prediction with Shiny

Data ▼

Figure ▼

✓ Analysis
✓

Repo

Help ▼

User Guide for SLPS

Dataset Preparations

Degradation Data

The indoor degradation data should contain a column for the relative humidity (RH). The time unit is days.

Irradiance Data

The irradiance data is a matrix. The first column is named "wave", which gives the wavenumber for the irradiance. The second column is "dwave", which gives the difference between two consecutive wavenumbers. The rest columns gives the irradiance information for the testing units. Each column is named by the unit ID.

Filter Data

The structure of the filter data is the same as the irradiance data. The value of filter is between 0 and 1. If no filter is used, it can be represented by a vector of ones.

Outdoor Data

The outdoor data consists of several components. The outdoor degradation data should contain an additional column for the calendar time. The temperature and RH are time varying. They are provided in separate files. Each file contains a column for unit ID, a column for time in calendar scale, and a third column for the measurements. The irradiance for outdoor units is also time varying. Each row represents the irradiance for a specific unit at a specific time. The first column for the irradiance file is unit ID, and the second column is the time stamp. The rest columns give the irradiance readings. Note that the column name for irradiance readings should be formatted as "w"+wavenumber (e.g., "w350").

Remarks on SLPS



- SLPS can be easily used with minimal training. No programming is needed and operations can be done by clicking.
- Users need to provide the data in a specific format.
- SLPS is web-based and it can be easily accessed.
- Data are uploaded by the users and are deleted once the session is closed.
- SLPS is open-source and the code is available to public once the app is online.
- SLPS can be expanded by adding more tabs for other functions.

Disclaimer: this App is provided for free use. It is understood by the user that the authors assume no liability for any errors contained in the App.

Summary and Future Work



- > SPHERE exposure results show
 - Reciprocity law is generally obeyed for chemical changes and yellowness index of PET, but not for the mechanical degradation (elongation at break)
 - An exponential dependence has been observed between degradation and wavelength for a pigmented PET. Photobleaching is observed under 452 nm.
 - High temperature and high relative humidity can accelerate chemical degradation and yellowing of a pigmented PET.
- The statistical models developed from the SPHERE exposure can predict the outdoor damage reasonably well.
- The open-source online SLP tool (SLP Shiny) is under development. Need more data to refine and validate the models, and to improve the software.
- ☐ Please contact us if you want to test the tool or work with us to develop a better tool.

Thank you for your attention!