ADVANCEMENTS IN ACCELERATED WEATHERING TESTS AND MATERIALS FOR MOLD-IN-COLOR SHEET MOLDING COMPOUNDS

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Abstract

The use of mold-in-color sheet molding compounds (SMC) for pickup-truck beds and stowage features has grown substantially since the introduction of the 2017 Honda Ridgeline. The advantages of mold-in- color SMC for these applications are substantial: reduced environmental and monetary cost from the elimination of a paint line, ability to fabricate geometrically complex designs, and superior impact/scratch/mar/corrosion resistance. Still, the continuing adoption of these compounds is not without challenges, chiefly the exposure testing necessary to validate their weathering resistance performance. To help understand the challenges, INEOS Composites has completed two-year South Florida exposure testing by OEM-specified SAE 1976 of various mold-in color composites. In parallel, the same compounds were tested by SAE J2020 (QUV) and SAE J2527 (xenon arc) exposure. Herein, the findings are presented, the relationship between the three test standards is discussed, and performance results for next-generation material systems are summarized.
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Background

Weatherable Composites and Testing

Mold-in color SMC is a burgeoning application for the composites industry. An example of its successful application was demonstrated by the Honda Ridgeline (model year 2017-present) truck-bed and stowage tub. One of the initial concerns for the adoption of a mold-in color solution for the Ridgeline was the traditionally suboptimal weathering resistance of unsaturated polyester and vinyl ester resins (1). This concern was alleviated through a unique resin formulation developed by Ashland LLC (now INEOS Composites) and Chromaflo Technologies.

Proving that this formulation could meet the weatherability needs of automotive OEMs was not without challenge. The automotive industry generally requires two-year outdoor exposure testing, which is conducted in Arizona or Florida, before adoption of a plastic for an application that has an exposure requirement (2). A need to shorten the development cycle for products that must undergo exposure testing led to the creation of two accelerated methods: SAE J2527 for xenon arc testing and SAE J2020 for QUV. These methods can measure the weathering resistance of a composite in the span of a few thousand hours, as opposed to two years. An overview of each accelerated method and outdoor exposure is given below.

The formulation created for the Ridgeline was qualified by Florida exposure and developed using xenon arc exposure. Still the question remains: what is the relationship between these two methods? It is still not widely known in the composite industry whether the results from these two tests will agree with each other, and if they do, to what degree. Also, their relationship to the QUV exposure method is not widely known either. The intent of the research conducted in this paper was to answer if these accelerated methods can be used to predict outdoor exposure testing, and if so, which of the two tests would be better. For each method, three mold-in color SMC formulations were tested compared.

Overview of Test Methods

A brief overview of each exposure method is given in the following subsections:

South Florida Exposure by SAE 1976

Outdoor exposure testing consists of uniformly directing specimens in a rack or box designed for that application. The method used for the testing outlined in this report placed the specimens to be tested in an unheated black box at a fixed angle of five degrees from the horizontal facing the equator.

These specimens were washed monthly after the morning dew cycle had been completed. The amount of sunlight exposure for this method depends on real world weather conditions. Therefore, it would not be surprising to see variations in results depending on the period that the data was collected.

QUV Testing by SAE J2020

QUV irradiance differs from xenon arc irradiance by the wavelength spectrum of the light that irradiates the samples. QUV provides a shorter range of wavelengths when compared to xenon arc. For the work outlined in this report, a UVB lamp with a peak emission of 313 nm was utilized. This lamp provides an irradiance spectrum that is concentrated around lower wavelengths of light when compared to sunlight.

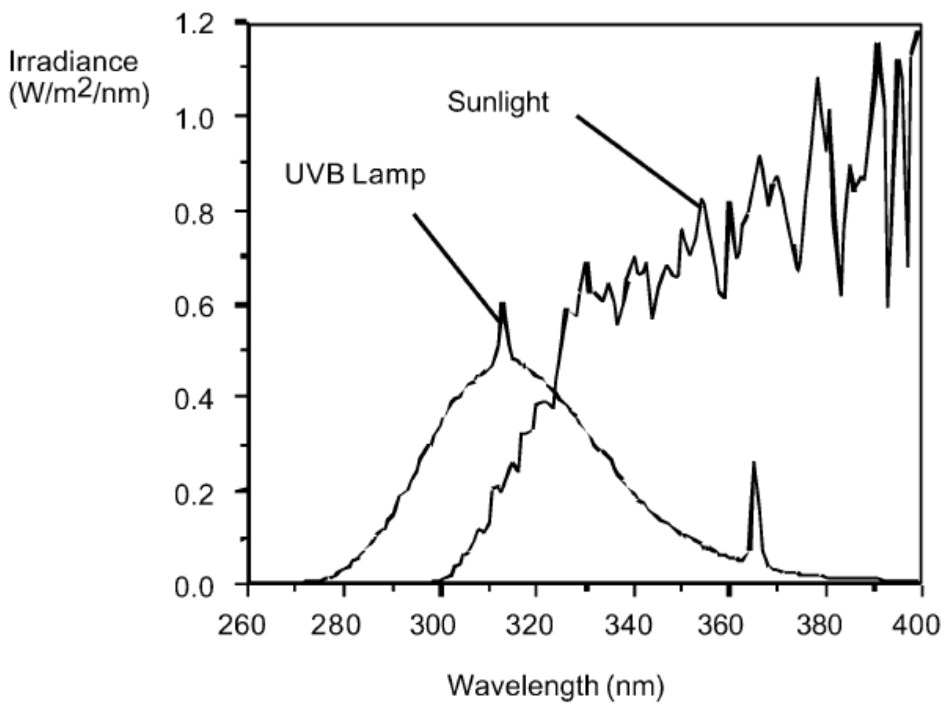


Figure 1: Irradiance spectrum provided by a UVB lamp against natural sunlight (3)

Testing was performed continuously until 500 hours of exposure was reached, at which time the samples were measured, and testing was continued. The test conditions alternated between the following:

- 8 hours of UV light exposure at 70 °C
- 4 hours of condensation exposure at 50 °C. Condensation was formed by heating of a water pan located under the specimens.

Xenon Arc Testing by SAE J2527

Xenon arc testing is performed using a xenon arc lamp to expose samples to a broader spectrum of light that more closely matches natural light than QUV. Specimen exposure is broken into four segments:

1. No light exposure with a front and back water spray for 60 minutes at 38 °C. The target relative humidly is 95 %.
2. 40 minutes of irradiance at a power of 1.32 kJ•m-2•nm-1 with no water spray. The target chamber temperature is 47 °C and the panel target temperature is 70 °C.
3. 20 minutes of irradiance at a power of .66 kJ•m-2•nm-1 with front water spray only. The target chamber temperature is 47 °C and the panel target temperature is 70 °C. The target relative humidity is 50 %.
4. 60 minutes of irradiance at a power of 1.98 kJ•m-2•nm-1 with no water spray. The target chamber temperature is 47 °C and the panel target temperature is 70 °C.

Brief Overview on Color Measurements

Color measurements such as “L”, dL, and DE are provided throughout this report. When measuring the color of a composite through a color eye device multiple values are given: “L”, “a”, “b”, “C”, and “H”. These 5 values are used in two separate coordinate systems to identify color, LAB and LCH.

An “L” value describes how light or dark an object is, with an increasing value corresponding to increasing lightness. This is the dominant measurement for the work detailed in this report due to the formulations starting with a deep black color. Since “L” is the dominate value, the other values are not reported due to their contributions being negligible.

For the LAB system, an objects color is described by the “L”, “a”, and “b” measurements. As detailed above, “L” is lightness, while “a” and “b” are the difference in red and green and yellow and blue, respectively. A larger “a” value represents a redder object and a larger “b” value represents a yellower object. The change in these values is described by the “d” designation. Hence, a dL value represents the change in the initial lightness of an object.

The dE value represents the square root of the sum of the squares of the change in “L”, “a’, and “b”. This is commonly used as a specification metric since it represents the overall deviation from the original color.

While not reported herein, for the LCH system, “C” represents the change in chroma, with a larger value being brighter and “H” represents the difference in hue.

EXPERIMENTATION

South Florida Exposure Testing

South Florida exposure testing was performed by Q-Lab Testing Services from November 2018 through November 2020 per SAE J1976 Procedure A.

The reported total radiation measured during that time was 12638.28 MJ/m2 and the total UV radiation was 659.21 MJ/m2.

QUV Exposure Testing

Testing was performed at INEOS Composites R&D facility per SAE J2020 using a Q-Sun QUV unit.

Xenon Arc Exposure Testing

Testing was performed at INEOS Composites R&D facility per SAE J2527 using a Q-sun xenon arc unit.

Color Measurements

Color measurements for the accelerated methods were taken at 500-hour intervals with a Datacolor 800 spectrophotometer.

SMC Formulations

The SMC formulations are described herein and designated as “Formulation A”, “Formulation B”, and “Formulation C”. All three are UV-resistant SMCs consisting of thermoset resin, carbon black filler, and light stabilizing additives.

Formulation A is an Arotran™ 805 based, 1.73 specific gravity compound that is currently used in the marketplace. It was considered a control for the purposes of this experimentation.

Formulation B is an Arotran™ 805 based, 1.55 specific gravity compound that is designed for applications that require high flow SMC and lower mass.

Formulation C is a next-generation formulation. It is based upon the new Arotran™ 2805 resin system, and is designed for applications requiring improved weathering resistance, deeper color, 1.55 specific gravity compounds, and higher strengths and stiffness.

SMC Processing Conditions

SMC was prepared using a 24” wide laboratory SMC machine. SMC was maturated at ambient humidity and temperature for seven days before molding.

SMC Molding Conditions

Test panels were molded using a 50-ton press fitted with a 30.5 cm x 30.5 cm textured chrome tool. The panels were molded at 150-160 ˚C at a pressure of 3.59 MPa for 120 seconds.

The Florida and xenon arc exposure samples were molded using a texture pattern that was half camera case and half Corinthian leather. The QUV samples were molded later with a heavy grain size, equivalent to the grain type and depth of current production applications.

Results

South Florida Exposure Testing

The 6-month interval measurements, along with initial “L” measurements are shown in Table 1 below and followed by the same results in graphical format.

Interval	Formulation A			Formulation B		Formulation C	
	dL	dE		dL	dE	dL	dE
Initial	26.48			26.97		25.75	
6 Months of Exposure	1.28	1.83		1.01	1.04	-0.77	0.90
12 Months of Exposure	-1.53	2.27		0.87	0.95	-1.51	1.60
18 Months of Exposure	-3.66	4.43		5.58	5.60	-1.69	1.72

24 Months of Exposure	-4.64	5.11		5.08	5.16	-1.26	1.28

Table 1: South Florida Exposure Color Testing

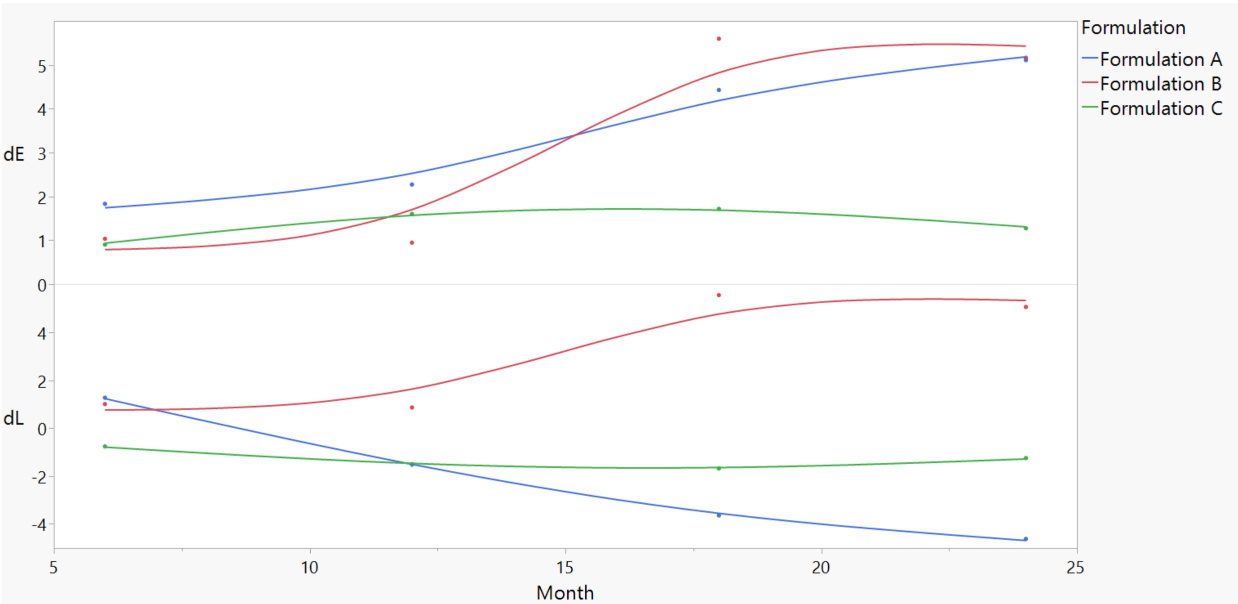


Figure 2: South Florida Color Measurements vs. Time

QUV Exposure Testing

The 500 hour interval measurements, along with initial “L” measurements, are shown in Table 2 below and followed by the same results in graphical format.

Interval	Formulation A		Formulation B		Formulation C	
	dL	dE	dL	dE	dL	dE
Initial	24.96		25.90		25.02	
500 Hours of Exposure	-1.50	1.50	-1.58	1.62	-0.44	0.46
1000 Hours of Exposure	-1.91	1.92	-2.83	2.85	-0.72	0.73
1500 Hours of Exposure	-2.47	2.47	-3.55	3.59	-1.07	1.07
2000 Hours of Exposure	-2.30	2.30	-3.68	3.72	-0.89	0.89
2500 Hours of Exposure	-2.38	2.39	-4.00	4.06	-0.99	0.99

3000 Hours of Exposure	-2.50	2.51	.	.	-1.26	1.26
3500 Hours of Exposure	-2.59	2.60	.	.	-1.20	1.20
4000 Hours of Exposure	-2.90	2.93	.	.	-1.44	1.44
4500 Hours of Exposure	-3.74	3.79	.	.	-2.16	2.16
5000 Hours of Exposure	.	.	.	.	-2.51	2.51

Table 2: QUV Color Measurements

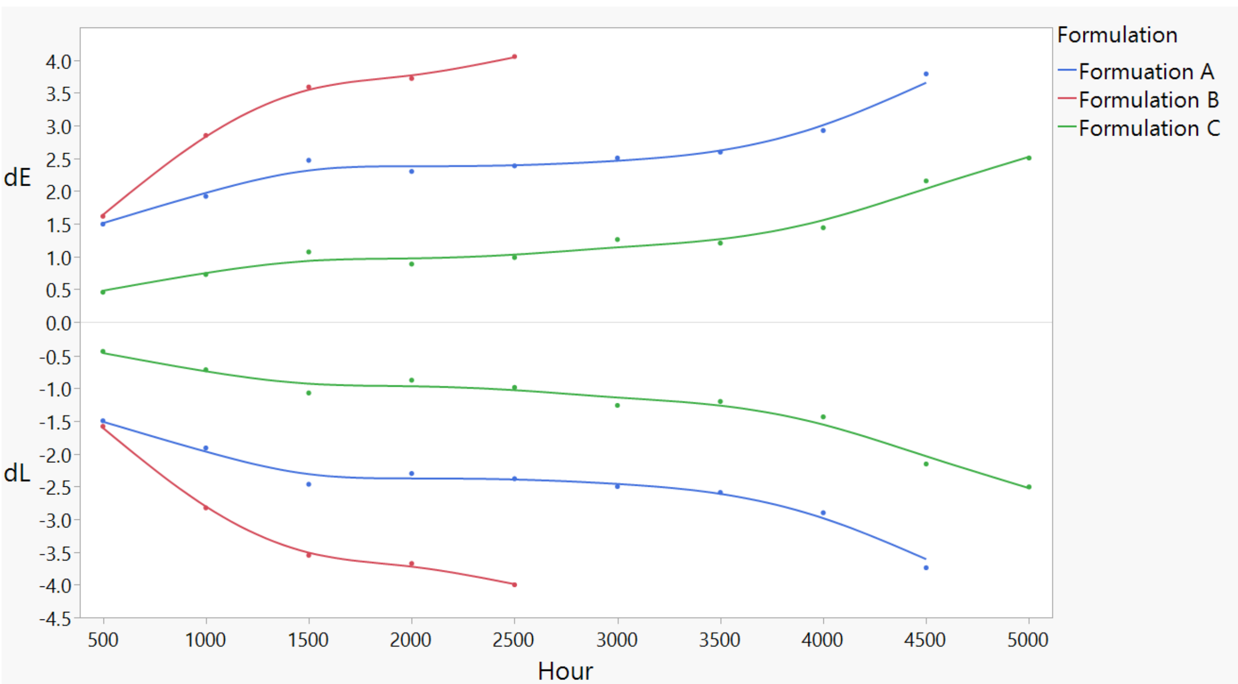


Figure 3: QUV Color Measurements vs. Time

Xenon Arc Exposure Testing

The 500 hour interval measurements, along with initial “L” measurements, are shown in Table 3 below and followed by the same results in graphical format.

Interval	Formulation A		Formulation B		Formulation C	
	dL	dE	dL	dE	dL	dE
Initial	26.92		28.34		28.67	
500 Hours of Exposure	-0.85	0.92	-0.36	0.48	-0.56	0.6

1000 Hours of Exposure	-1.16	1.3	-0.96	0.96	-0.52	0.54
1500 Hours of Exposure	-1.71	1.86	-1.83	1.87	-0.58	0.61
2000 Hours of Exposure	0.47	0.49	-2.36	2.42	-0.17	0.21
2500 Hours of Exposure	-2.46	2.48	-5.64	5.83	-0.28	0.29
3000 Hours of Exposure	-3.43	3.6	-7.7	7.88	-1.16	1.16

Table 3: Xenon Arc Color Measurements

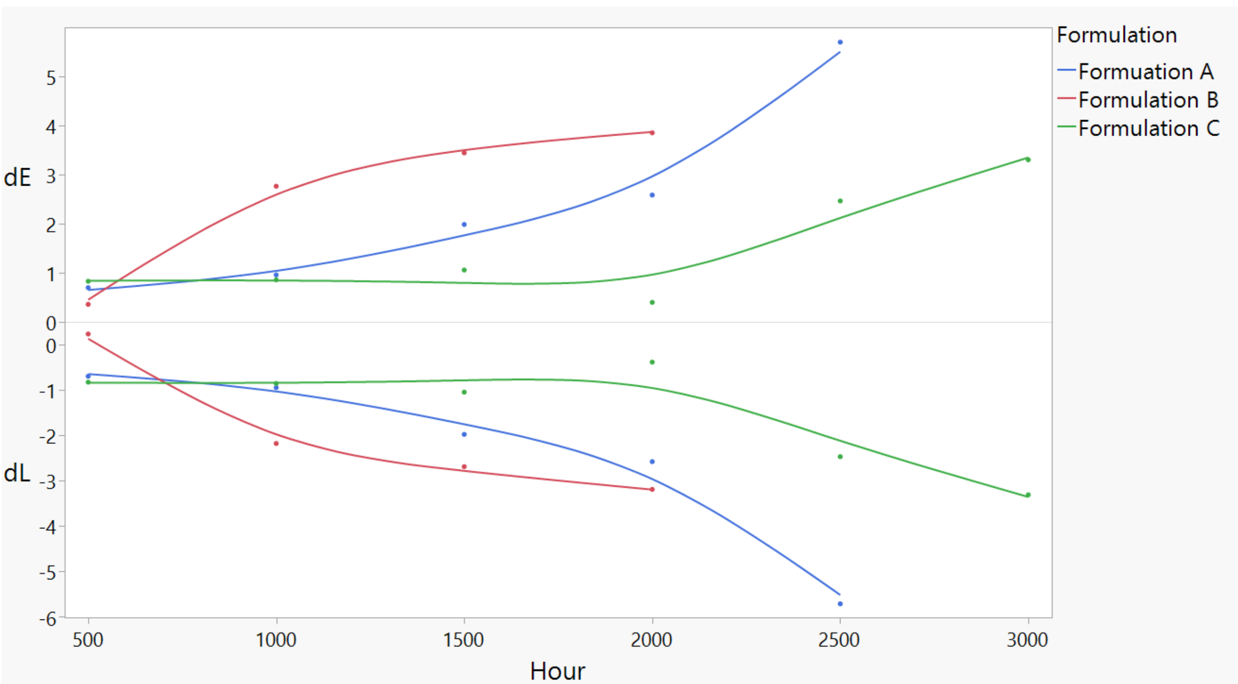


Figure 4: Xenon Arc Color Measurements vs. Time

Discussion

General Trends Observed Throughout All Three Methods

Table 1, Table 2, and Table 3 provide the color results from each interval measurement for all three methods. The researcher's metric for determining the performance of a given formulation is the number of periods that the formulation takes to reach a dE value of greater than three. While not provided in this report, the color values other than "L" have little to no effect on the dE, which is the reason for their lack of inclusion.

Based on the dE metric, a ranking of the weathering resistance of the three formulations can be determined. All three methods agree that Formulation C has the most robust weathering resistance. Formulation A is also superior or equal to Formulation B in each test, implying that it has the superior resistance capabilities of the two.

The agreement of the three methods allows for the conclusion that accelerated testing will give a general indication of a formula's expected weathering performance under real world conditions. In other words, the results presented herein suggest that by performing accelerated testing prior to real-world exposure testing, the performance ranking of a group of formulations will be able to be obtained.

Comparison of Florida Exposure to Accelerated Methods

When comparing the two accelerated methods to Florida exposure, there are some similarities but a few key differences. For example, all three methods agreed that Formulation C displayed the strongest weathering resistance, followed by B and then A.

Figures 5 and 6 help to display the key difference between Florida exposure and the accelerated techniques. Figure 5 shows the completed coupons after Florida exposure and Figure 3 QUV (pictures of the formulations post-xenon exposure are not available, but their failure mode was like those from QUV testing). When comparing Figures 5 and 6, it is readily apparent that the failure mechanism for Formulation B differs between the two exposure methods. Florida exposure resulted in a whitening effect on the surface, likely from glass fiber becoming exposed, where-as QUV showed a darkening effect, likely from exposure of carbon black. These trends are also observed in the "L" value measurements found in Tables 1 and 2. Formulation B showed a trend of increasing "L" value (whitening) over time for Florida exposure, where-as QUV showed a decreasing value. Interestingly, this effect was only observed in Formulation B.

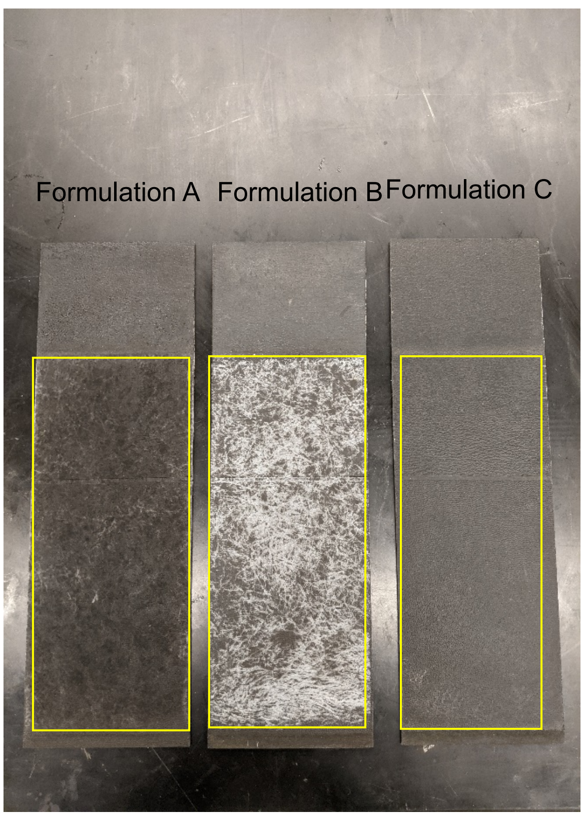


Figure 5: Completed 2-year Florida coupons. The region enclosed by the yellow rectangle represents the exposed sections of the coupons.

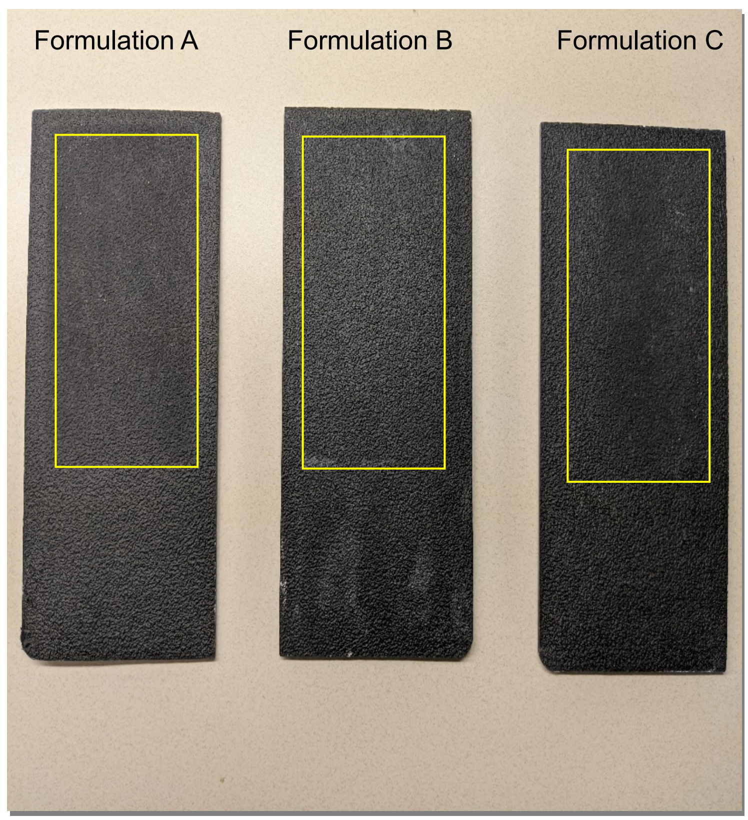
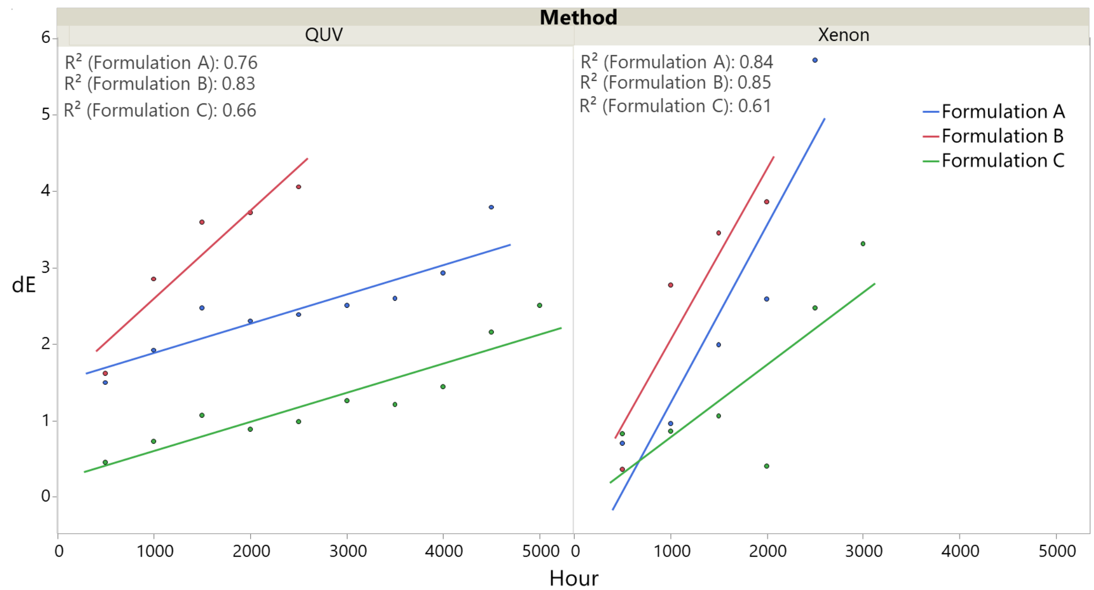


Figure 6: Completed testing (dE>3) QUV coupons. The region enclosed by the yellow rectangle represents the exposed sections of the coupons.



Comparison of Accelerated Methods

Figure 7 gives the color change of each formulation over time fit to a simple linear model for the accelerated methods. The r-squared parameters suggest a reasonable, but not perfect, model fit for Formulations A and B, but not C. This suggests that for formulations with strong exposure resistance, a linear relationship for modeling dE over time is appropriate to model its degradation, but a poorly performing one would be better served by a more complex model.
The findings for the relative rankings in weathering resistance were comparable between each method, with Formulation C providing the most robust resistance, followed by A and then B.

Figure 7: Comparison of DE Over Time of Accelerated Weathering Methods

One notable difference between the two methods is the initial change in color. After 500 hours of QUV exposure, Formulations A and B yielded a dE of approximately of 1.5 and 1.6, respectively. Compared with their 500-hour xenon arc measurements of 0.9, and 0.5, the initial QUV exposure provided a rapid change. Following that initial change, however, the slope of the color change was significantly less for the QUV samples than the xenon arc. This phenomenon could be explained by a few reasons: (1) simple operator error, or, (2) there is surface layer, such as mold release, that is quickly stripped from the samples by QUV exposure, but not by xenon arc. Then once that surface layer is eroded, the QUV may act more slowly on the remaining sample, while xenon arc affects the composite more uniformly. A possible mechanism for this could be attributed to the wavelengths of light utilized in each method. Since QUV utilizes a tighter spectrum, that spectrum could be adept at deteriorating the outer layer of the composite, but not the inner, while the broader spectrum provided by xenon arc exposure could have wavelengths equally capable of eroding all layers.

Summary and Next Steps

Usefulness of Each Method

The results presented in this paper give evidence for the utility of each exposure method. Since the general trends in weathering resistance were the same for each method, all three have the potential to be used for development purposes.

For R&D development, when comparing the accelerated methods, xenon arc exposure appears the most practical. Results were obtained in the shortest amount of time and there was a clear delineation between the relative strength of the three formulations. The same is true for

QUV, however, meaningful differences in all three formulations were not obtained until 4500 hours of exposure, compared to 2500 for xenon. Though, it is possible that this may be considered a strength. While more work is needed to fully support this hypothesis, it can be easily imagined that QUV would be useful for determining small differences in the exposure resistance of similar compounds since it causes a slower degradation which could allow for smaller differences to be observed. Consequently, if development work involves small changes to a formulation in hopes of minor increases in exposure performance, QUV may be the preferable method.

While the accelerated methods provide a good baseline for determining the relative exposure resistance when comparing two formulations, the results discussed in the comparison of Florida to accelerated methods section highlight why outdoor exposure is still required. The whitening effect observed in Formulation B during Florida exposure is an important discovery of how the SMC will perform in the field. It is reasonable to believe that a part designer would prefer that the color of a part that is exposed to the elements to remain dark after exposure, as opposed to whitening, even if the DE metric is the same between the two options. If part qualification relied solely on either accelerated method, this discovery would not have been made until years after initial production.

Industry Recommendations and Final Thoughts

Prior to receiving the results detailed in this paper, it was the hope of the researchers to determine a statistically sound correlation model between the three methods, but such a model could not be developed. A possible reason for this may be due to lack of data. Only three formulations were tested in this study and very few replicates were used, resulting in low statistical power. To address this, a suggestion for future work in this area is to test as many samples and replicates as possible to provide more statistical power.

However, even without the development of a statistical model, the data generated from this study does allow general recommendations to be made on the deployment of weathering tests and the strength of the conclusions drawn from them. The xenon arc results suggest that if a dE of less than 3 is obtained after 3000 hours of exposure, it is likely that a dE of less than 3 will be obtained after two years of Florida exposure. The same guidance can be given for QUV after 5000 hours of exposure

Still, care must be taken to not only rely on color change measurements. As mentioned earlier, the failure mode of a formulation, while still technically passing a color change metric, may disqualify that formulation upon visual inspection. Therefore, it is the recommendation of the authors of this paper, that when formulating mold-in color composites, to test by as many methods as possible and to test as many replicates as possible to truly understand how a formulation will behave in a real-world application.

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