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Understanding, Optimizing, and Measuring Water in Xenon-Arc Accelerated Weathering for Automotive Exterior Coatings

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Introduction

Continual demands from consumers for highly durable coatings with excellent appearance motivate automotive manufactures and paint suppliers to introduce new coating systems into the marketplace to meet those demands. However, because of the potential risk inherent in introducing new coating systems, significant testing of paint systems must be completed before commercial introduction. Unfortunately, testing programs require significant time and resources to reduce the risk of long-term failure to acceptable levels. Typically, automotive manufacturers required up to five years of Florida exposure for a new paint system before they will implement new technology. This time constraint could be significantly reduced if a trustworthy accelerated test were available to the industry that would rapidly and accurately assess the long-term weatherability of automotive coating systems.

The current state-of-the-art in accelerated weathering for automotive paint systems is SAE J2527 (based on the previous SAE J1960), which calls for weathering in a chamber containing a xenon arc light source filtered to produce radiation with a wavelength distribution similar to that of sunlight. In addition, the temperature, humidity, and spraying of liquid water on the paint samples is controlled to attempt to stress the coatings to failure in a shortened amount of time compared to Florida. Acceleration is presumed to occur due to the increase in the average intensity of the radiation, the temperature, and the duration of humidity/liquid water contact compared to Florida. In attempting to improve on any accelerated test protocol, these are the variables that can be adjusted to improve correlation to field results.[1]

Another school of thought contends that attempting to reproduce potential failures observed in the field in accelerated weathering devices is futile, as the diversity of the climate across any market regions (such as North America) is sufficiently wide that potential failures observed in one region may not be observed in other regions due to climatic differences.[2,3] In addition, the year-over-year variability in climate in any given region, i.e. south Florida, is significant enough that reproducing all of the germane environmental variables in an accelerated device is not possible nor productive. Climatic data lend some support to this approach and significant progress is being made in looking at the fundamental changes that occur in model coating systems during well controlled exposure conditions. The results from these exposures can then be used as input for statistically founded predictions about the performance of these systems under different exposure conditions. This service life prediction approach, shows promise for changing the nature of accelerated testing, but requires enormous amounts of data and has yet to be demonstrated to work on highly durable coating systems containing modern stabilizer additives exposed to the full array of environmental variables.[4,5]

For the practical coatings technologist, the best hope in the short term is to advance the existing accelerated tests by examining and improving those testing variables that can be shown to affect their accuracy and acceleration factor. Previous work has shown that the spectral power distribution (SPD) the light source within the accelerated testing machine can have a tremendous influence on the accuracy of the accelerated tests. Gerlock et. al. have shown that only those tests that use light sources that accurately reproduce the SPD of terrestrial sunlight in the UV region will produce chemical composition changes in coatings that are equivalent to those produced in coatings exposed outdoors.[6] Recently a new optical filter for xenon arc weathering devices has been commercialized that does accurately reproduce the sun's SPD. Chemical changes observed in coatings exposed using this optical filter accurately reproduced those observed from outdoor exposed coating systems.

Irrespective of the exposure variable, an accurate accelerated test must reproduce the chemical changes observed in coatings exposed outdoors in coatings exposed to the accelerated test. Extensive analytical work has developed a number of methods for measuring these chemical composition changes. These methods are significant improvements over the standard gloss measurements used in the coatings industry, which have proved inadequate for anticipating the long-term weatherability of basecoat/clearcoat paint systems.[7,8] Ideally, an accelerated test would reproduce not only the chemical changes, but the physical failures (cracking, blistering, delamination, gloss loss, color change…) that are observed in coatings exposed outdoors.

In addition to the light source, the other main environmental variables that must be correctly reproduced are the temperature and moisture exposure. The effects of heat are thought to be mainly restricted to the production of the thermal stresses in the paint system and driving the rate of any chemical reactions taking place during weathering.

Previous work has shown that the effects of water on the weathering of coating systems are numerous.[9] First, water can plasticize a coating, thereby changing its mechanical properties. Second, this plasticization will increase the mobility of small molecules entrained in the coating binder which may have deleterious effects on the paint system. Third, due to differential swelling of the substrate and the various layers in a multilayer paint system, significant hygroscopic stresses can be induced in coatings upon exposure to water. Fourth, the gloss loss of coatings is driven significantly by the removal of degraded material from the surface of the coating due to the washing action of liquid water both outdoors (rain) and during accelerated testing (water spray). Finally, the presence of water is required to drive hydrolysis of the coating, which can become significant, particularly in coating systems that are prone to such degradation.

To accurately capture the effects of water in an accelerated weathering protocol, the type (humidity vs. liquid water) and duration of water exposure must match the type and duration of water exposure experience in a defined location outdoors. In this manuscript we report on a detailed analysis of the amount and type of water that paint samples are typically exposed to outdoors in Florida as well as other locations. In addition we have characterized the amount of water exposure that paint samples are exposed to in standard weathering equipment running the SAE J2527 test method. These results are compared to each other and to the maximum theoretical water uptake in various paint systems. Included in the data is a statistical analysis of the machine-to-machine variability of the amount of water available to paint samples. We conclude with suggestions for how accelerated weathering protocols could be improved to provide more accurate results in a shorter time frame.

Experimental

Materials. The paint systems used in this study were standard automotive paint systems. The substrate was cold rolled steel. All panels were pretreated with zinc phosphate and coated with cathodic electrocoat. The panels were then spray primed with solvent borne polyester primer. The topcoat system was either waterborne basecoat followed by high solids solvent borne clearcoat or solvent borne basecoat and high solids solvent borne clearcoat. All panels were produced at target film builds and target baking conditions appropriate for each layer.

Test Methods.

Outdoor Weathering. All panels were exposed for natural outdoor weathering in south Florida. Panels were exposed according to SAE J1976, 5° from horizontal, facing south.

Accelerated Weathering. Panels were exposed to accelerated weathering in one of two types of apparatus. The first was a rotating drum accelerated testing machine (Ci5000, Atlas Materials Testing Technology LLC). The second was a flat array machine (Q-SUN® Xe-3-HS, Q-Lab Corporation). Both machines were initially configured to run SAE J2527 as the standard accelerated weathering protocol. Additional adjustments to the duration and timing of the water spray cycle are detailed during the description of individual experimental results.

Water Collection. The volume of water delivered to the panels was measured by a variety of methods. As a baseline, the amount of water delivered from the spray nozzles in the machines was measured by attaching a cup to the water nozzle (Figure 1). Water was collected for a specified amount of time and the volume of water then measured. The nozzles were inspected and cleaned prior to the start of the test.

Figure 1 - Experimental set-up for collecting water from spray nozzles during testing in a rotating drum accelerated weathering device.

The water delivered to individual panels was measured by attaching a cup to the bottom of a panel holder in the rotating drum apparatus. Water that impacted the panel then dripped down the panel into the cup (Figure 2). After a given time, the water volume in the cup was measured. In the flat array apparatus, a modified specimen tray (Figure 3) that holds 35 cups was inserted into the chamber and water collected during the spray cycle. The water volume was then measured in each cup. This method allowed for the uniformity of water spray to be measured in addition to the volume during any spray cycle.

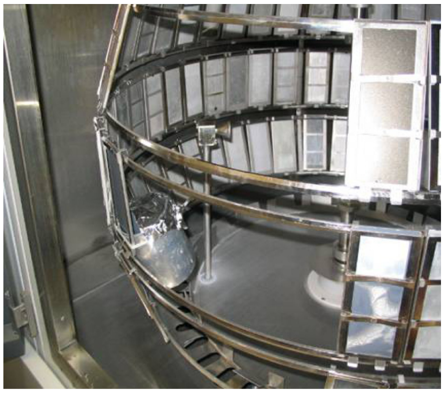

Figure 2 - Collection of water dripping off of standard paint panel during accelerated weathering in a rotating drum device.

Figure 3 - Water collection set-up in a flat array accelerated weathering device.

An alternative measure of water impacting the panel used a sponge to adsorb the water hitting the panel area. In this experiment, a synthetic sponge (Proline® Professional Deluxe Cellulose Cleaning Sponge model K-10P) was cut to the same size as a paint panel (75mm x 150 mm). The sponge was then moistened and wrung out to remove excess water. The sponge was then weighed and placed in the panel holder (Figure 4). After a given amount of time, the sponge was removed and re-weighed to gage the amount of water that impacted the panel area.

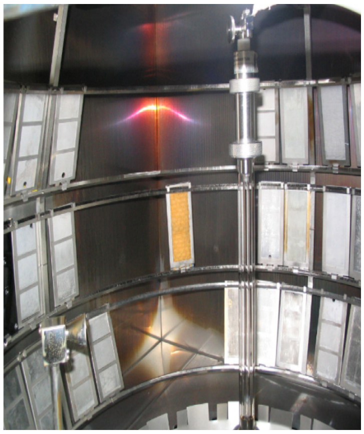

Figure 4 - Synthetic sponge set-up in a rotating drum accelerated weathering device.

Water Exposure Outdoors. Contact with water for panels exposed outdoors was measured with a custom-built device that provided real-time data on the mass of paint panels exposed horizontally in Jacksonville, FL (Figure 5 and 6). Details of this method have been published elsewhere.[10] Briefly, a paint panel was attached to an electronic load cell, which was configured to measure the mass of the panel plus any additional mass due to water either on or adsorbed into the paint system. Sample mass data was taken every 5 minutes and recorded on a computer.

Careful calibration was periodically performed. Measures were taken to assure that birds were not allowed to sit on the panel and confound the data. Using this set up, the mass of a paint panel could be recorded continuously for days at a time. Water and dew events were easily discernible. In addition, temperature, humidity, solar radiation, and wind were recorded at the same site.

Figure 5 - Test site where FL water exposure was measured. Note weather station next to panel racks.

Figure 6 - Paint panel attached to load cell for water exposure study. Load cell is underneath horizontal paint panel.

Panel Evaluations. Gloss was measured on selected panels using a commercial gloss meter (micro TRI-gloss supplied by BYK-Gardner).

Water Absorption. Percent water uptake is performed by weighing a test panel on an analytical balance both before and after a water exposure. Percent uptake is calculated as follows:

(mass wet (g) – mass dry (g))* density water (g/cm^3))/volume of coating layers (cm^3))*100

Results and Discussion

<u>Natural Exposure Conditions.</u> To understand the water exposure required to make a trustworthy accelerated test, one must first quantify the water exposure paint systems experience during natural weathering. Typically, only monthly or annual rainfall data is available for various exposure sites. This data is insufficient, as to correctly mimic the natural wet/dry cycles the daily wetness of naturally exposed panels must be measured. To this end, a weather station was installed in Jacksonville, FL in 2004 (Figures 5 and 6). Multiple publications have described and outlined the measurement capabilities of the weather station (3, 4), including the capability of the station to more specifically measure water and it's effects on coatings.[10,11] The data from this weather station have been used to produce a model of panel wetness in Jacksonville, FL. In evaluating the data from the weather station versus traditional data from South Florida test sites, it appears to be a reasonable assumption that a water model formulated to represent Jacksonville would be similar to a water model for South Florida. That is, the weather rainfall pattern for Jacksonville was similar enough to the rainfall pattern in south Florida to use the rainfall model developed for Jacksonville for the south Florida region as well. To test this hypothesis, the device used to collect the data in Jacksonville, FL will be installed in Homestead, FL. since it is understood that year-to-year variation in the weather patterns make this assumption tenuous. When more accurate and longer-term weather data become available, the model can be refined for various locations. The water model allows one to mathematically describe the water exposure, including the actual amounts/volumes of water contributed from both dew and rainfall. Simple time-of-wetness data does not allow for differentiation between light dew and heavy rain. Such differences are important due to the time required to saturate a coating system with water.

For the formulation of an accurate model of the time/type of wetness a panel is exposed to in Florida, four years of data were required to attenuate annual differences in rainfall and temperatures. Variables included in the model were: ambient temperature, panel temperature, dew point, wind speed, rain fall and solar radiation. The following constants were defined:

1. Maximum amount of water that would reside on the test panel.
2. Low and high dew formation rate as a function of dew point and panel temperature.
3. Wind evaporation rate.
4. Solar evaporation rate.

The complexity of the model lies not in its mathematics, but rather the logical flow of conditions that are required before an evaluation of the water exposure can be made. Because of this a syntactical description of the model is a more straightforward expression of the algorithm.

The following simple logical expressions were programmed to predict the amount of cumulative sitting water on the test panel:

1) While raining the residing water was set at maximum.
2) If the panel temperature was below the high or low dew point trigger the appropriate mass would be added to the cumulative residing water mass up to the maximum.
3) If the wind speed was above the wind evaporation trigger then the wind evaporation rate mass would be subtracted from the cumulative residing water mass.
4) If solar radiation was above the solar radiation trigger then the solar evaporation rate mass would be subtracted from the cumulative residing water mass.
5) If panel temperature was above 40C the cumulative residing water was set to zero.

Using the above algorithm the predicted cumulative residing water mass was very close to the measured water mass on the test panel. Trigger points in the above expressions were determined using a statistical review of the weather data. In mathematical terms, the equation is generically as follows:

Total water = ΣOver time (Panel water)
Panel water = Rain + Dew - Wind Evaporation - Solar Evaporation
Rain: If any rain is detected by the rain gauge, panel water is set to 70 grams.
Dew: If panel temperature is below the dew point, then depending on the temperature spread the dew constant is set to Dew Low formation at 0.005 grams or Dew High formation at 0.2 grams being added to the panel water. (The highs and lows are determined by the temperature spread (high and low delta of air temp.)

Wind Evaporation: If the wind is above a trigger level and panel water is present then a constant is subtracted from the panel water. Wind evaporation was set to 1.0 grams.
Solar Evaporation: If the solar radiation is above a trigger level and panel water is present then 0.15 grams are subtracted from the panel water.

The water exposure model has proven useful in verifying the amount of water exposure that is required in an accelerated weathering protocol. One must, however, be mindful of the specificity of the model. First, it has been formulated in detail only for exposure in certain locations in Florida, USA. Second, the response of different coating systems to water exposure can be quite different. For example, waterborne coatings can take up significantly more water than solvent borne coatings. Third, the surface energy or state of degradation of a coating can affect the details of water uptake.

Accelerated Weathering. Once an accurate description of the water exposure during natural exposure has been formulated, a protocol for water exposure in accelerated tests can be designed. In developing a specific water exposure protocol, the conflicting demands of acceleration versus water absorption dynamics must be reconciled. Florida exposure data clearly shows that over one hour is required to saturate panels with water outdoors. However, long water cycles during accelerated testing require the light to be off. Thus, dose-based weathering times will be lengthened. A realistic compromise must be reached to have both an accurate and useful accelerated test protocol.

The goal of any new accelerated weathering test is to fit within the confines of the new SAE J2527 test protocol. This standard is machine agnostic in that either rotating drum or flat array test instruments can be used, as long as they meet the proper control and reproducibility standards. The data in Table 1 show that similar water uptakes can be reached in either type of machine when run under nominally the same conditions (6 hours, 50ºC). Previous work has shown that flat array tests deliver significantly more water to the samples, then rotating drum configuration while running SAE J2527. To reproduce the results of the spray system of a rotating drum the flat array was programmed to have the water spray system on for 5 second & off for 55 seconds for any given minute of a programmed spray cycle. In essence delivering 1/12th of the maximum amount of water available[12.] In addition, these water absorption experiments agree with the values predicted by the water model, given the operating conditions of the machines. This is based on the results from lab experiments performed from the model as well as actual Florida panel uptake data. [1]

Panel Weight (g)	Rot. Drum	Flat Array
Start	59.3259	59.1582
Finish	59.341	59.1726
Delta	0.0151	0.0144
Coating vol (cm3)	1.1185	1.1338
% Uptake	1.35%	1.27%

Table 1 - Water absorption in two accelerated weathering devices.

As reported previously,1 the typical accelerated protocol for automotive exterior coatings (SAE J2527) lacks by about a factor of five the water uptake expected in a wet day in a South Florida summer. The water absorption of a solventborne basecoat/clearcoat system is shown in Figure 7. Neither the spray during the dark or the light cycle attain the same level of water absorption that is observed after an extended (16 hr, 75 °F) water soak or sixteen hours of QCT humidity testing.

Using Florida summer water data as a starting point, modifications can be attempted to match the water condition in an accelerated test as well as in other outdoor locations. Water exposure is known to differ depending on the location and micro-climate in the specific test locale. For example, Arizona is known to be a relatively dry exposure location as compared to South Florida. Is it possible to implement an artificial water condition in Arizona that would simulate a Florida condition? Previous work has shown that the weathering conditions in Florida can result in significantly different chemical and physical changes occurring paint systems compared to the changes that take place in the same paint systems exposed in Arizona. It has been postulated that these differences are due to the amount of liquid water that the paint system is exposed to in the two locations.[9] Samples exposed in Florida tend to lose gloss more quickly when the degraded surface material is washed away by rain and dew. Samples exposed in Arizona tend to retain their gloss longer, as the degraded material has little opportunity to be washed away. Measurements of the locus of degradation in Florida and Arizona exposed samples tend to show more degradation at the surface of the paint systems exposed in Arizona due to the lack of removal by water. This work also demonstrated that soaking panels overnight in water can make accelerated outdoor exposure in Arizona more Florida-like (gloss loss and chemical composition change).

Thus, additional extended water soaks can make Arizona exposure much like Florida exposure.

Trying to achieve Florida-like water exposure in an accelerated weathering device is logistically more challenging due to the aforementioned balance between water exposure and acceleration. In starting to modify the accelerated cycles to achieve the correct water exposure, numerous issues had to be addressed, such as how to identify the target water uptake, how to control and deliver water to the equipment, and how to calibrate water as a regular part of the accelerated weathering process. The water absorption of a basecoat/clearcoat paint system is shown in Figure 8. The absorbed volume of water continues to increase with time in the dark+spray cycle. Temperature and spray type were the same as are specified in SAE J2527. Only the time of spray was increased. For the tested coating system, approximately six hours were required to match the uptake of the system as tested in a field scenario. All coatings examined to-date required more than 1 hour to approach the saturated conditions typically seen in Florida exposure. Using the 6 hour dark+spray cycle, a different coating system was tested in rotating drum and flat array machines and compared to known data from measurements of that system during a South Florida summer (Figure 9). Both the flat array and rotating drum machines were able to achieve greater water absorption than the maximum value obtained during Florida exposure. This result is unusual and may be due to the sensitivity of this particular coating system to either temperature or surface tension effects. The greater absorption of water in the rotating drum device is also counter to previous data that showed greater water absorption in flat array machines and is most likely be due to the reduce water volume programmed into the flat array to match the rotating drum since the water volume delivered in the rotating drum can not be adjusted.

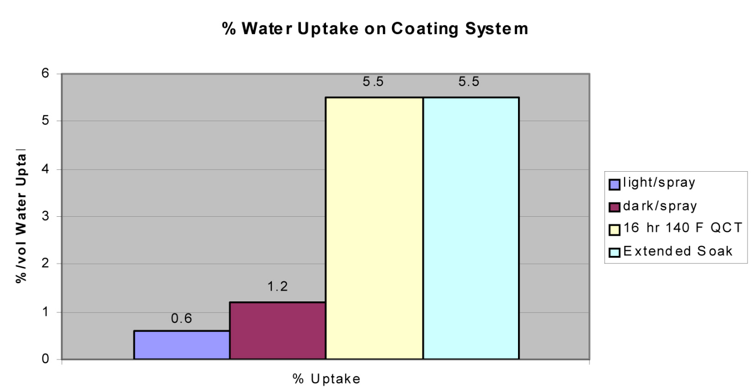

Figure 7 - Water absorption in SBBC/SBCC paint system after different water exposure treatments.

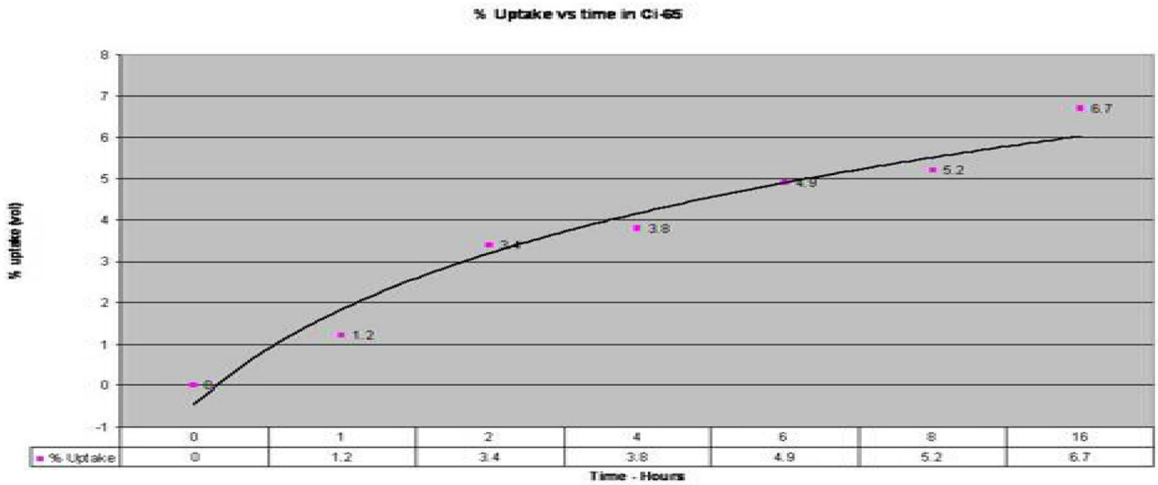

Figure 8 - Water absorption in SBBC/SBCC paint system as a function of water exposure time in a rotating drum accelerated weathering device.

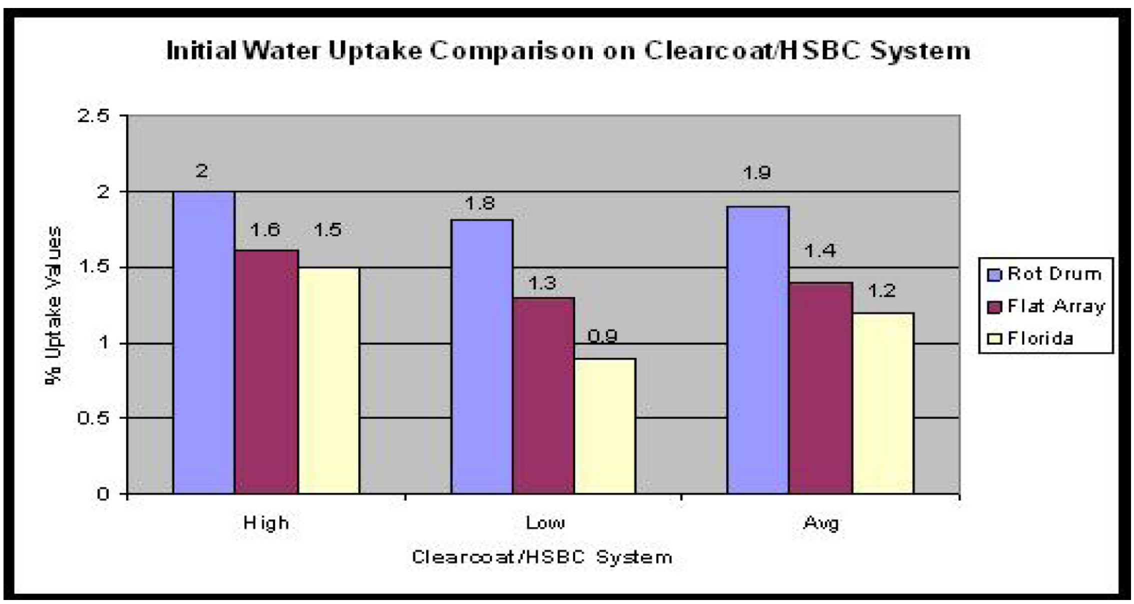

Figure 9 - Water uptake after 6 hours of water exposure in rotating drum and flat array accelerated weathering devices. Results are compared to Florida exposure. The high, low, and average values were taken from multiple laboratory and field measurements.

After demonstrating that it is possible to deliver sufficient water to paint systems in accelerated weathering devices, the matters of reproducibility and control were addressed. A number of original ideas were generated for measuring and quantifying water in the accelerated weathering equipment. The water emitted by the spray nozzles using the set-up shown in Figure 1 is shown in Table 2. Water was collected and the volume was measured after a 5 minute dark+spray cycle. The results were fairly consistent with tests performed in two different rotating drum cabinets within the same laboratory. It has been determined that different laboratories will produce different water volumes from these tests depending on the water source and engineering of the accelerated weathering machines.[13] While this technique could be used to guarantee water delivery from each spray nozzle inside a particular machine, it still does not insure a particular amount of water contacting the coating surface. For example, the spray nozzles could be directing water unevenly within a chamber or to the walls of the machine.

Water measurement data nozzle collection

Rotating Drum Machine #1			Rotating Drum Machine #2		
1)	317		1)	274	
2)	315		2)	298	
3)	320		3)	304	
4)	313		4)	314	
5)	315		5)	298	
6)	308		6)	308	
7)	310		7)	287	
8)	314		8)	297	
9)	318		9)	296	
10)	314		10)	293	
	Average	315 ml		Average	297 ml

Table 2 - Water collected from spray nozzle in two rotating drum machines during a 5 minutes spray during the dark cycle.

Measurement of the water impacting the panels was attempted with the cup attached to the bottom of the panel. The water volume collected after 5 minutes of a dark+spray cycle was measured and recorded at various positions in the machine. (Figure 1). The results from this testing were inconsistent and believed not to be representative of the water actually contacting the panel surface.

The capture system used for the flat array system showed increased consistency and was useful determining and adjusting the uniformity of the spray heads. Water was collected during the dark+spray cycle for five minutes (Table 3). While this is closer to what is needed for water control, there are gaps between the cups where water is not measured, and this method could not be adopted for vertical exposure equipment such as rotating drum apparatus.

	Row 1 Ave.	Row 2 Ave.	Row 3 Ave.	Row 4 Ave.	Row 5 Ave.	Machine Ave.
Flat Array # 1	13.4	23.7	35.9	34.1	20.2	25.5 ml
Flat Array # 2	24.7	29.3	21.4	21.9	18.6	23.2 ml
Flat Array # 3	25.3	23.4	21.7	19.4	14.4	20.9 ml

Table 3 - Water collected in 3 flat array accelerated weathering devices at 2 different labs during a five minute spray. Each row has 7 collection points for a total of 35. The spray system was set to 60 seconds on, 0 seconds off for any given minute of spray.

The use of the sponge method provided the most reproducible and consistent method for measuring the water impacting the panel surface. Again, measurements were taken at various locations within the weathering device during a five minute dark+spray cycle (Table 4). The data from testing on the various locations on rotating drum machines does show some differences depending on location on the drum. This is evidence for nonuniform water distribution within the machine. This was also noted on similar tests performed in a flat array machine. However, given that rotation of specimens should typically be done in both horizontal and vertical equipment, the variations could be attenuated to a less significant level. This means that rotating drum specimens need to be rotated between the top, middle and bottom rows on the drum to achieve uniform water and light exposure13. Table 4 demonstrates the water uniformity of a rotating drum machine using the sponge method. Table 5 demonstrates the water uniformity of a flat array using the same test procedure as used in Table 4. One idea for a new test method would be a water calibration using a specific sponge from a specific manufacturer. Testing using the sponge would require a minimum and average water weight gain with testing in various locations in both horizontal and vertical accelerated weathering machines.

	Rotating Drum Machine #1				Rotating Drum Machine #2		
	Top rack	Middle rack	Bottom rack		Top rack	Middle rack	Bottom rack
1	8	16	11		7	13	7
2	7	18	16		6	8	8
3	7	16	11		7	12	7
4	7	16	17		5	10	8
5	7	17	17		7	12	7
sum	36	83	72		32	55	37
Total		191				124	

Table 4 - Water collection using synthetic sponge. All data collected in rotating drum accelerated weathering device during a five minute spray cycle.

Spray set to 60 second on 0 seconds off for a given minute

	Far Left	Center	Far Right
Top	54	54	50
Middle	54	56	51
Bottom	53	55	52

Spray set to 10 second on 30 seconds off for a given minute

	Far Left	Center	Far Right
Top	33	31	32
Middle	32	44	39
Bottom	32	48	39

Table 5 - Water collected using exact same size synthetic sponge in exact same type panel holder as used in Table 4. All data collected in a flat array tester during a five minute spray cycle.

The purpose of this work was to match the level of coating degradation and produce the same failure modes in an accelerated test that are seen outdoors. In order to achieve this, known historical data from coating systems in outdoor exposures needs to be documented, plus these systems need to be tested directly against the same systems in a new accelerated test. These systems must contain all of the failure modes known for coatings, including gloss loss, erosion, and delamination at different coating interfaces, blistering, humidity whitening and multiple types of cracking failures (Figure 10). Cracked and uncracked coating systems are shown in Figure 10. Sources of cracking have previously been identified as residual stress, photooxidation induced brittleness, and cracking due to hydrothermal stresses. Water can also lead to blistering (Figure 11). During this failure mode, blisters form due to retained water in the coating systems. As the blisters become larger in the basecoat with increased water exposure, the clearcoat is stressed to the point where cracking will be seen.

In the future we will report on the results of a large round-robin testing program, where a large number of paint systems of known failure mechanisms are being tested in four different accelerated weathering protocols. Three of the protocols have been modified from standard J2527 to improve the water exposure conditions. The results detailed in this

Figure 10 - Cracked and uncracked coatings specimens after exposure in south Florida.

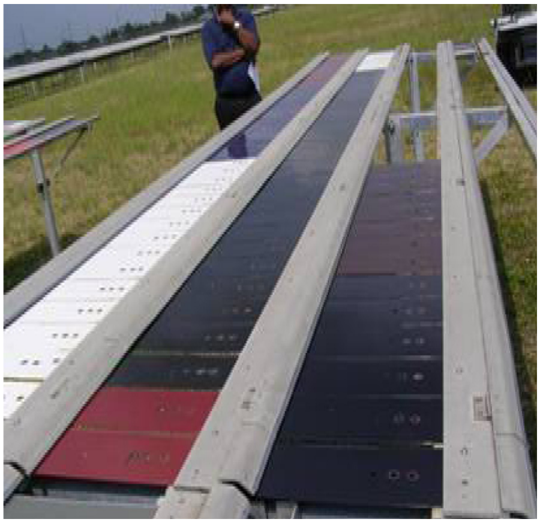

Figure 11 - Blisters in paint panels exposed in south Florida. Blistering is due to water absorption in the paint system.

manuscript were used to design the accelerated weathering conditions and the details of the machine control parameters. The paint systems chosen represent many different coating chemistries and are known to respond to accelerated weathering in different fashions. Concurrently, these paint systems are being exposed outdoors in Florida to confirm the already known long-term weathering behavior of these systems. The results of this study should clearly point the way to a new accelerated weathering protocol for automotive coating systems. The approach behind this will be to provide the minimum water exposure required to produce the proper coating failures while attempting to reach maximum acceleration in a new test cycle.

Conclusions

Significant advancements in the understanding of water and its effects on the weathering of automotive coatings have been made by careful examination of the water exposure in both Florida and standard accelerated weathering devices. New methods have been identified to measure water both in the field and in accelerated weathering equipment. These methods have shown that different accelerated weathering machines and test protocols produced very different levels of water exposure when compared to Florida water exposure. Matching the water exposure in Florida while still maintaining significant test acceleration is difficult due to the length of a dark+spray cycle that will be required to fully saturate most automotive coating systems.

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