

Development and Testing of Molecular Adsorber Coatings

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ABSTRACT

The effect of on-orbit molecular contamination has the potential to degrade the performance of spaceflight hardware and diminish the lifetime of the spacecraft. For example, sensitive surfaces, such as optical surfaces, electronics, detectors, and thermal control surfaces, are vulnerable to the damaging effects of contamination from outgassed materials. The current solution to protect these surfaces is through the use of zeolite coated ceramic adsorber pucks. However, these pucks and its additional complex mounting hardware requirements result in several disadvantages, such as size, weight, and cost related concerns, that impact the spacecraft design and the integration and test schedule. As a result, a new innovative molecular adsorber coating was developed as a sprayable alternative to mitigate the risk of on-orbit molecular contamination.

In this study, the formulation for molecular adsorber coatings was optimized using various binders, pigment treatment methods, binder to pigment ratios, thicknesses, and spray application techniques. The formulas that passed coating adhesion and vacuum thermal cycling were further tested for its adsorptive capacity. Accelerated molecular capacitance tests were performed in an innovatively designed multi-unit system containing idealized contaminant sources. This novel system significantly increased the productivity of the testing phase for the various formulations that were developed. Work performed during the development and testing phases has demonstrated successful application of molecular adsorber coatings onto metallic substrates, as well as, very promising results for the adhesion performance and the molecular capacitance of the coating. Continued testing will assist in the qualification of molecular adsorber coatings for use on future contamination sensitive spaceflight missions.

Key words: molecular adsorber coating, adsorption, adsorptive capacity, molecular capacitance, on-orbit molecular contamination, outgassing, thermal coating

1. INTRODUCTION

On-orbit molecular contamination is a major concern on many of NASA's spaceflight missions. It occurs when materials outgas and deposit onto sensitive surfaces of the spacecraft and instruments. Sources of outgassing include potting compounds, epoxies, tapes, lubricants, and other materials from within the spacecraft. Sensitive surfaces, such as optics, electronic boxes, detectors, solar arrays, and thermal control coatings, are vulnerable to the damaging effects of these outgassed materials. It has the potential to degrade the performance of spaceflight hardware and diminish the lifetime of the spacecraft. For instance, the deposition of outgassed materials on optical surfaces can result in the attenuation of image and sensing performance. It can also change the thermal control characteristics of coatings, such as solar absorptance and emittance, which can directly impact the spacecraft temperatures. As a result, it is imperative to mitigate the risk of on-orbit molecular contamination.¹

The current solution to protect sensitive surfaces from outgassed materials is through the use of adsorber pucks. As illustrated in Figure 1, adsorber pucks are constructed of honeycomb patterned ceramic materials. They are mounted on spacecraft interiors and instruments to collect and minimize the transfer of on-orbit molecular contamination. The use of adsorber pucks have been evaluated and proven to be very effective with flight data and ground simulation testing for many of NASA Goddard Space Flight Center (GSFC) missions, such as Hubble Space Telescope (HST) and Tropical Rainfall Measurement Mission (TRMM). However, as seen in Figure 2, adsorber pucks require the use of additional hardware for installation that results in size, weight, and cost related concerns.¹ For example, the mounting hardware is often times very bulky and can contribute to additional mass loading to the spacecraft. It is costly to fabricate and manufacture and thereby, can easily impact the project budget. The use of additional

hardware can also delay the Integration and Test (I&T) project schedule due to complex hardware designs for interface requirements on the spacecraft.

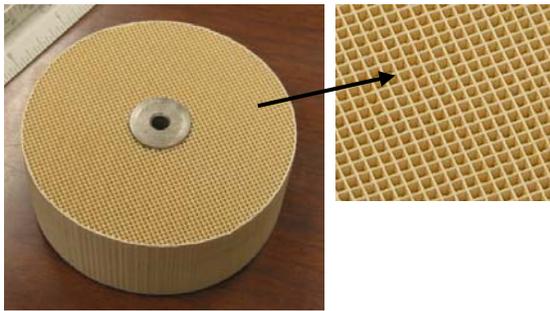


Figure 1. Honeycomb patterned ceramic material on adsorber pucks



Figure 2. Mounting hardware associated with adsorber pucks

The use of molecular adsorber coatings (MAC) are proposed as a new sprayable alternative to adsorber pucks and its complex mounting hardware. Molecular adsorber coatings have been formulated and optimized to adsorb on-orbit molecular contamination. This innovative coating is expected to be directly sprayed onto internal instrument surfaces, structural walls of the spacecraft, and on the inside or outside of electronics boxes. The coating also has the potential to function as a thermal control coating for those surfaces. The application of this new sprayable solution would not require the use of any additional mounting hardware. As a result, it will eliminate and reduce the concerns that are associated with the current adsorber puck technology. Recent development and testing of molecular adsorber coatings have shown promising results for use on future contamination sensitive spaceflight missions.

2. BACKGROUND

Molecular adsorber coatings are comprised of two key components. These include zeolite and Ludox® binder. The formulation and optimization of these two key components resulted in the development of a highly permeable and porous coating that can adsorb on-orbit molecular contamination. Zeolite is the white pigment of the coating. It acts as a molecular sieve and adsorbent material for the coating. As illustrated in Figure 3, the crystal structure of zeolite consists of large open pores, which are referred to as cavities. These cavities provide porous adsorption sites that capture and trap contaminant molecules. Zeolite also has a large surface area to mass ratio, which maximizes the available trapping efficiency.²

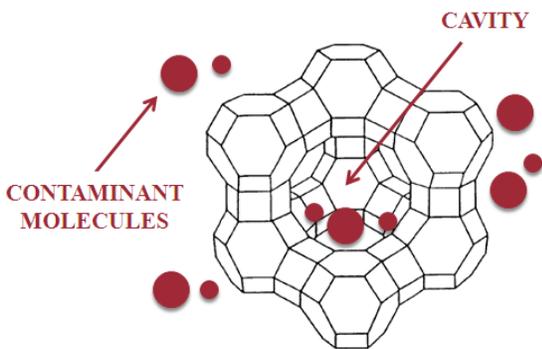


Figure 3. Large pores or cavities on the crystal structure of zeolite capture and trap contaminant molecules

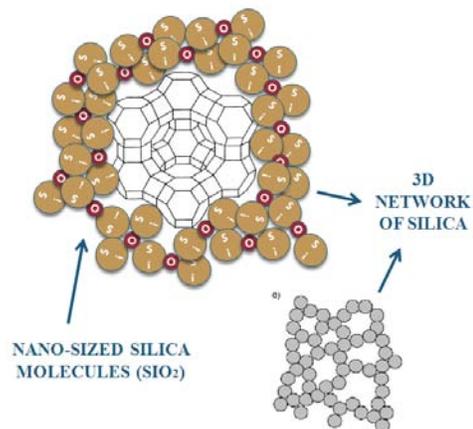


Figure 4. Nano-sized silica molecules of the binder gels around the pigment without blocking the adsorption sites

Ludox® is the transparent binder of the coating. This component is comprised of suspensions of colloidal silica in liquid phase. It acts as the glue that holds the coating together and is also responsible for providing adhesion between the substrate layers of the coating. Ludox® binders consist of nano-sized silica molecules that are not large enough to clog the pores or prevent access to the adsorption sites. As shown in Figure 4, the binder gels into a three dimensional network of silica around the zeolite structure without blocking any of the cavities.

3. APPROACH

The primary goal of this study is to develop and test molecular adsorber coatings. The results will be used to assist in the qualification of the coating for use on contamination sensitive missions as a replacement to the current adsorber puck technology. A two phase test plan was created to accomplish this goal.

The approach for the development of molecular adsorber coatings consisted of:

- Establishing a standard substrate configuration
- Optimizing the formulation process for better adhesion and adsorptive properties, and consistent coating reproducibility
- Refining the spray application technique to produce a coating that can easily be sprayed

Laboratory testing of the developed coating formulas were evaluated under ground handling and on-orbit flight conditions. The approach for the testing of molecular adsorber coatings consisted of:

- Evaluating adhesion performance and thermal stability of the coating with tape tests and vacuum thermal cycle testing
- Determining the molecular capacitance of the coating using an accelerated test set-up and procedure
- Measuring thermal optical property measurements, such as solar absorptance and emittance

4. DEVELOPMENT METHODOLOGY

4.1. Standard substrate configuration

A standard substrate configuration was established for molecular adsorber coatings. As illustrated in Figure 5, the bottom layer consists of a sample substrate that is representative of spaceflight hardware surfaces. These include metal surfaces, such as aluminum, or composite surfaces. The middle substrate layer is comprised of a base coating that is applied before spraying the top coating. A base coating acts as an intermediary by providing better adhesion between the surface and the molecular adsorber coating layer. Examples of base coatings include silicate primers or thermal control coatings that are normally used in spray coating applications for spaceflight missions. The molecular adsorber coating layer is then applied over the base coating as the top substrate layer.

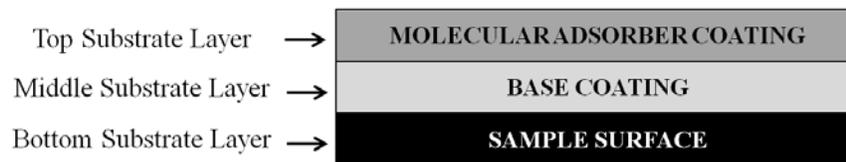


Figure 5. Substrate configuration for molecular adsorber coating application

In previous efforts, AZ-93 has been used as the base coating. AZ-93 is a white silicate based ceramic thermal control coating made by AZ Technology. It is known to adhere well to aluminum substrates. However, not all spaceflight

hardware is comprised of metals. Therefore, to broaden the application of molecular adsorber coatings, recent efforts have been directed towards using MLP-300-AZ as the base coating in the substrate configuration. MLP-300-AZ is a transparent epoxy based primer made by AZ Technology that provides a bondable surface for silicate based coatings. The primer is known to adhere well to multiple substrates, such as composites and metals.

MLP-300-AZ also strongly bonds to aluminum tapes and still remains undamaged and sturdy after bending and flexing movements. This characteristic is beneficial for molecular adsorber coatings because tapes can be easily installed onto hardware, instruments, or near vents. For example, if a contamination sensitive spaceflight project decides that there is a need for a contamination control tool, such as molecular adsorber coatings, it can be quickly applied in its tape form during the I&T phase as a last minute solution. The tape also allows the opportunity for a refresh of the coating before launch, should the coating approach capacity as a result of high outgassing during hardware thermal vacuum testing.

4.2. Formulation and spray application process

The formulation and spray application process was optimized to develop a molecular adsorber coating that can be easily sprayed onto aluminum substrates, tapes, and foil. In this study, a total of 13 formulas were developed and successfully applied onto the MLP-300-AZ/MAC substrate configuration. This consisted of spraying 120 aluminum substrate samples, strips of aluminum tape, and sheets of aluminum foil. A summary of the coated samples is shown in Table 1. The samples had an average MLP-300-AZ thickness of 1 mil. The samples were sprayed with 1 to 3 coats of the molecular adsorber coatings. The top layer thickness varied between 4 to 18 mils depending on the formula used and the coats sprayed. All sample substrates were used for adhesion and molecular capacitance testing purposes.

Sample Size	Quantity	Substrate Material
5.1 cm x 5.1 cm	94	Aluminum
5.1 cm x 10.2 cm	12	Aluminum
15.2 cm x 15.2 cm	8	Aluminum
5.1 cm x 30.5 cm	6	Aluminum
5.1 cm x 30.5 cm	10	Aluminum Tape

Table 1. Summary of sample substrates coated onto the MLP-300-AZ/MAC substrate configuration

Several parameters were considered to optimize the formulation and spray application process onto MLP-300-AZ/MAC substrates. The formulas were developed by varying the binder grades, binder to pigment ratios (BPR), pigment treatment techniques, and viscosity of the coating. Improvements in the spray application techniques were also applied.

Binder grades – Five Ludox® binder grades were used in the formulation of the coatings. These consisted of AM, AS-30, AS-40, HS-40, and TMA. Each binder grade has different properties, such as the dispersion of colloidal silica, silica particle size, chemical stabilizers, and pH level.

Binder to pigment ratios – The binder to pigment ratio (BPR) is the mass of the binder divided by the mass of the pigment relative to the colloidal silica in the specific binder grade used for the formula. The BPR was varied between 0.9 and 4.0 to achieve formulas with good adhesion properties.

Pigment treatment – A technique was developed to provide better adhesion of molecular adsorber coatings to the underlying substrate layers. This consisted of treating the pigment with silica from the binder for bonding purposes.

Viscosity - The viscosity of the coating was varied to achieve one that was desirable for application with a spray gun. This was accomplished through various methods, such as milling the coating with grinding media or adding water to the coating.

Spray application - The spray application process was improved by exploring different surface treatment techniques, such as rub priming, cure durations, and temperatures. In addition, using spray guns with different sized orifices and settings were utilized. For example, the air, fluid flow, and atomizing pressures were adjusted to achieve optimal sprayable conditions.

5. TESTING METHODOLOGY

5.1. Molecular capacitance testing

Molecular capacitance is the measure of a coating's ability to adsorb outgassed materials. Data measuring the adsorptive capability are needed to qualify molecular adsorber coatings as viable replacements for the existing adsorber puck technology on spaceflight missions. An accelerated test procedure for molecular capacitance testing was developed to increase the productivity and capability to run multiple samples with different formulas and thicknesses more efficiently during a single test run. This consisted of using an innovatively designed three unit system that exposes coating samples to a volatile contaminant source. The test configuration is illustrated in Figure 6.

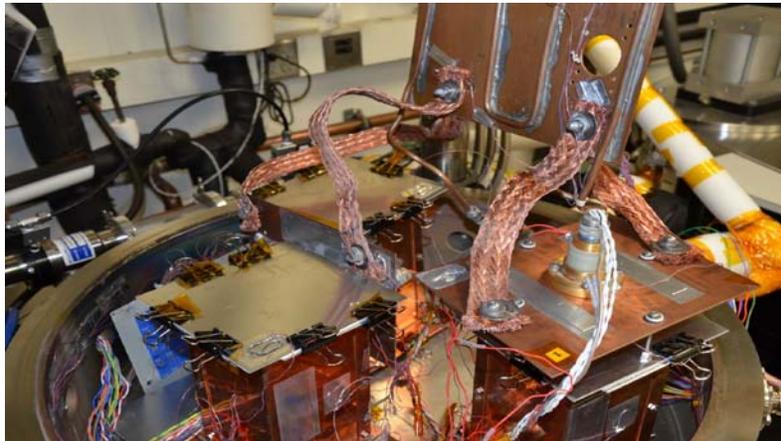


Figure 6. Multi-unit copper test configuration for molecular capacitance testing

The test apparatuses shown in Figure 6 were constructed from copper for better conductivity. Tests were operated at 45 °C under vacuum in a bell jar chamber. These conditions provided an accelerated rate with minimal maintenance. Molecular adsorber coating samples with dimensions of 2.2 cm diameter were enclosed in the upper compartment of each apparatus and exposed to the contaminant source that was positioned on the bottom of the test units. Stearyl alcohol was the selected contaminant for these testing purposes. It is comprised of a volatile condensable material that has a constant vapor pressure to provide constant rates. A quartz crystal microbalance (QCM) was positioned on a cold plate to detect the flux of the contaminants that escaped each copper apparatus. QCM rate data was used to indicate when the samples had reached saturation or approached its surface adsorptive capacity.

In previous studies, the molecular capacitance of the coatings was estimated through molecular transport modeling and analysis of the QCM rates with extrapolations and assumptions. However, for this study, a straight forward approach was used to determine the amount of contaminant adsorbed by the samples. This was achieved by comparing the mass of the samples before and after exposure to the contaminant source. This was challenging due to the hygroscopic nature of the coatings. Preliminary studies have revealed that molecular adsorber coatings retain moisture at a rate of 2.33 mg/min from a surrounding environment of 35% humidity. For example, 70 mg of additional mass was adsorbed within 20 minutes. This made it difficult to distinguish the amount of mass that was gained as a result of adsorption from the contaminant source during testing or moisture from the surrounding environment after testing.

To resolve this issue, the solution was to use a vacuum environment to release moisture from the coating prior to taking any mass measurements. The samples were pre-weighed before exposure to the contaminant source. This was performed in a nitrogen purged dry glove box environment with operating conditions at 25 °C and <10% humidity. After testing, the contaminated samples were re-weighed in the glove box to measure the mass gain. In addition, a reference sample was used to determine the amount of moisture lost or gained during a single vacuum test cycle. The reference consisted of a pristine sample that was not exposed to any contaminant source. The mass loss or gain of moisture was used to incorporate a water adsorption correction factor into the calculation of molecular capacitance. This was used to compensate for any experimental testing errors from the mass measurements taken in the dry environment.

5.2. Adhesion performance testing

Adhesion performance for most thermal control coatings is evaluated in accordance with Method A of the ASTM D 3359-02 X-Cut Tape Test specification. This test is designed to evaluate any evidence of delamination or separation of the coating from the substrate by creating a crosscut pattern through the coating with a razor blade. 3M Scotch Masking Tape #250 is firmly pressed to each section, removed rapidly at a 180 degree angle, and examined for any sign of adhesive failure.

The brittle nature of thick ceramic coatings does not lend itself well to the razor cut as the blade may induce inconsistent stress fractures within the coating that would influence test results. Therefore, the adhesion performance test was modified to exclude the use of the razor blade to illustrate adhesion without surface fracturing of the top coat. This modified tape peel approach was performed as a preliminary evaluation of adhesion performance on samples before and after thermal cycle testing. The standard ASTM specification was administered on those formulas that showed promising adhesion properties from the modified tape peel test.

5.3. Thermal cycle testing

Thermal cycle testing is performed to evaluate the stability of molecular adsorber coatings for adhesion performance after exposure to cold and hot temperature cycles that are similar to spaceflight conditions. Test samples were mounted to a copper test stand using grafoil and metal clamps to ensure thermal coupling to the test stand. Thermocouples were mounted at several locations in the test set-up to monitor and control temperature. After assembly, the test stand was attached to heaters and connected to a cold plate.

This test was performed in a bell jar vacuum chamber operating between -40 °C and 70 °C for approximately 50 cycles. This temperature range encompasses the intended use for molecular adsorber coatings with a 30 degree margin for both hot and cold temperature cases. These coatings are expected to operate at temperatures that are representative of electronics boxes and other interior surfaces, which typically reach temperatures between -10 °C to 40 °C.

5.4. Thermal optical property testing

Thermal optical properties, such as solar absorptance and emittance, are important for thermal control coatings because they are used to achieve spacecraft temperature requirements. Molecular adsorber coatings were made to mitigate on-orbit molecular contamination rather than achieve specific thermal control properties. Since these coatings are comprised of a white pigment, the thermal properties of molecular adsorber coatings were evaluated and compared against a common white thermal control coating, such as AZ-93.

Solar Absorptance – The solar absorptance is the measure of the proportion of solar radiation the coating absorbs. It was calculated using the AZ Technology LPSR-300 instrument. The LPSR-300 measures the reflectance of the sample's surface for a spectral range of 250 to 2800 nm, at a 15 degree angle of incidence using the ASTM E903-82 standard test method. The instrument's measurement of accuracy is +0.02 for solar absorptance.

Normal Emittance – The emittance is the measure of the relative ability of the coating to radiate absorbed radiation. It was calculated using the Gier-Dünkle DB-100 Infrared Reflectometer. The DB-100 measures the normal emittance of the surface from 5 to 40 microns at room temperature using the ASTM E408-71 standard test method. The instrument's measurement of accuracy and repeatability are ± 0.02 and ± 0.001 , respectively.

6. TEST RESULTS

6.1. Molecular capacitance testing

In this study, a total of six test runs were completed. Within these test runs, 26 samples coated on MLP-300-AZ/MAC systems were exposed to stearyl alcohol. The samples were coated with 7 of the formulas that were developed and had total coating thicknesses between 5 and 19 mils. Figure 7 demonstrates the QCM data collected from Test Run #5. In this test run, two molecular adsorber coating samples were placed into each copper apparatus. These six samples were exposed to stearyl alcohol for 64 hours. As illustrated in Figure 7, the rates show an increasing trend between 4 and 32 hours. At the beginning of the test run, the rates were lower because the flux of contaminants were captured and trapped onto the cavities or pores of the samples. The rates began to increase as the surface adsorption sites on the coating filled up. As a result, a smaller flux of contaminants was adsorbed by the samples and a greater amount was deposited onto each QCM.

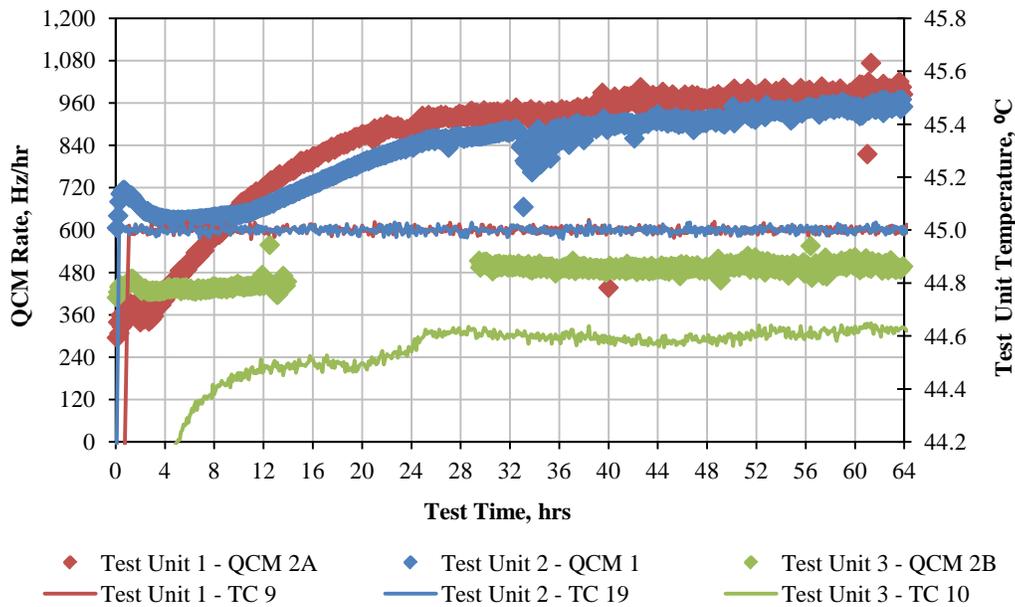


Figure 7. QCM rate and temperature data for test run #5 for a total of six samples

As the test progressed, the samples became saturated and could no longer adsorb. This trend is depicted in Figure 7 between 40 and 64 hours. At this point, Test Units 1 and 3 have stabilized to a constant rate due to the limited availability of surface adsorption sites. However, the rates for Test Unit 2 still depict a slightly increasing trend. This suggests that the samples may not have reached maximum capacitance. These samples can be further exposed to the contaminant source in a future test run. In addition, Test Unit 3 data between 14 and 29 hours were removed due to malfunction and instability issues that occurred on QCM 2B during testing.

Results from the six test runs revealed that molecular adsorber coatings will operate at an equal or greater efficiency and capacity when compared to the existing adsorber puck technology. Previous studies have shown that the molecular capacitance of ceramic honeycomb adsorber pucks is 3.3 mg/cm^2 . This value pertains specifically to HST style adsorbers that were dunked in coated carbon loaded zeolite. As illustrated in Figure 8, the molecular capacitance for molecular adsorber coatings developed in this study is between 1.9 to 5.2 mg/cm^2 depending on several factors. For example, the formula used, coating thickness or the length of time the samples were exposed to the contaminant source may have a role. Currently, no correlations could be made to these suggested factors. Further investigation and testing may be

required to determine the exact cause. Regardless, this data demonstrates that molecular adsorber coatings can be used as a possible alternative to adsorber pucks.

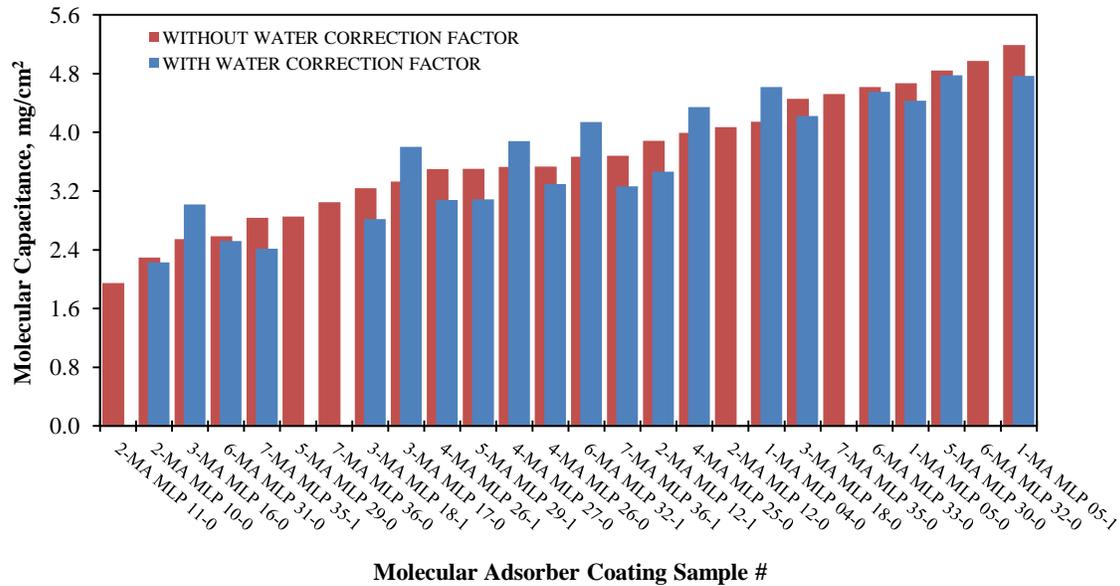


Figure 8. Molecular capacitance test results for 26 samples

Figure 8 displays a comparison of the molecular capacitance with and without the correction factor from the reference sample. The water correction factor varied between ± 0.5 mg/cm² depending on the sample’s exposure under vacuum conditions. This data suggests that the use of the dry glove box and vacuum environment to measure mass was effective in limiting water adsorption. For example, the molecular capacitance of Sample #1-MAMLPO5-1 dropped from 5.2 to 4.8 mg/cm² with the addition of the correction factor. The decrease indicates that there was an error of 0.4 mg/cm² of moisture that was not removed under vacuum. The correction factor has a greater impact on the raw molecular capacitance value when the errors involved in the mass measurements are greater. Experimental testing errors are associated with the amount of water released under a vacuum environment or adsorbed during transport or placement in a low humidity environment. Therefore, if the errors from water adsorption are minimal, the mass measurements recorded for molecular capacitance are reliable.

6.2. Adhesion performance and thermal cycle testing

In this study, approximately 48 samples coated with 13 different formulas were evaluated for adhesion and thermal stability performance. Table 2 illustrates a summary of the formulas that passed adhesion performance and thermal cycle testing using the x-cut tape test and modified tape peel test methods. An ASTM D3359 value of 3A or higher is considered passing for the x-cut and no significant loss of material greater than 1/32nd of an inch for the modified peel test. Samples coated with formulas 4, 5, and 9 showed very minimal separation of the coating from the substrate layer. The remaining 10 formulas resulted in very poor to moderate adhesion performance. As a result, these three formulas are expected to undergo further testing for repeatability and will be considered as viable candidates on future qualification plans for molecular adsorber coatings.

Formula Number	Total Thickness Range (Mils)	Modified Tape Peel Test for Pre and Post Thermal Cycle	X-Cut Tape Test for Post Thermal Cycle
4	6.7 - 18.5	Pass (4A)	Pass (4A)
5	5.9 - 10.4	Pass (4A)	Pass (4A)
9	7.1 - 7.6	Pass (4A)	Pass (4A)

Table 2. Adhesion performance thermal cycle test from - 40 °C to + 70 °C for 50 thermal cycles

6.3. Thermal optical property testing

An additional 32 samples were sprayed using the three formulas that passed adhesion and thermal stability performance. These samples were used for thermal optical property testing and will be thermal cycled in the future for repeatability. Table 3 summarizes the average solar absorptance and normal emittance values for each formula. This data indicates that molecular adsorber coatings with total thicknesses between 1.6 and 8.2 mils have an overall average solar absorptance and normal emittance of 0.37 and 0.93, respectively.

Formula Number	Batch Number	Total Thickness Range (Mils)	Average Solar Absorptance	Average Normal Emittance
4	2A-1	4.0 – 4.8	0.37	0.94
4	1C-1	2.0 – 3.2	0.42	0.93
5	2A-1	1.6 – 3.2	0.47	0.91
5	13	3.2 – 8.2	0.33	0.92
9	2B-1	2.1 – 6.7	0.31	0.93
9	1D-1	4.7 – 5.5	0.32	0.92

Table 3. Thermal optical property measurements for molecular adsorber coatings

In comparison, AZ-93 with standard coating thickness between 4 to 5 mils have solar absorptance and normal emittance values of 0.16 and 0.92, respectively. The average normal emittance for molecular adsorber coatings is similar to AZ-93. However, the average solar absorptance is approximately 2.3 times greater. The average solar absorptance of 0.37 does not represent the coating's thermal optical property accurately because solar absorptance varies based on the coating thickness. As illustrated in Figure 9, the solar absorptance of molecular adsorber coatings is inversely proportional to the coating thickness. When the coating is sprayed on lighter, it provides more transparency to the aluminum substrate and thus increases the solar absorptance. However, when the coating is sprayed on thicker, it provides a more opaque surface and thus decreases the solar absorptance.

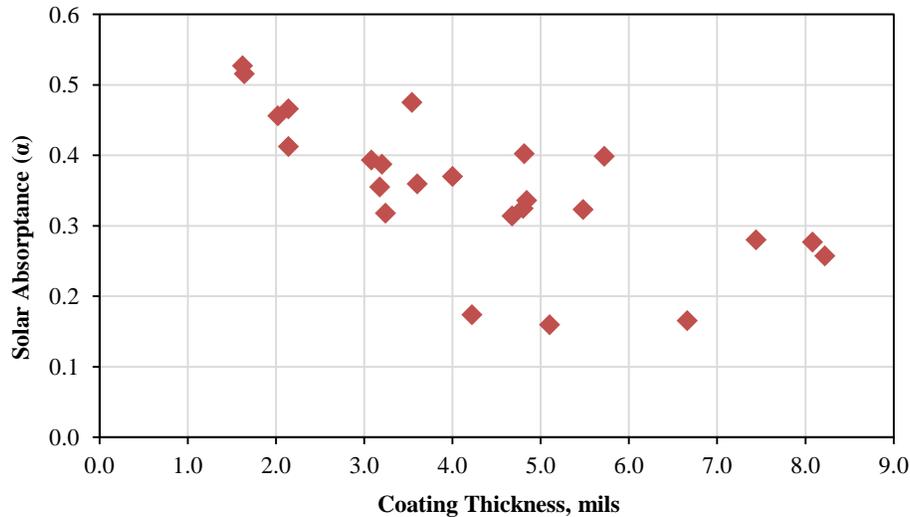


Figure 9. Effect of coating thickness on solar absorbance of molecular adsorber coatings

7. CONCLUSIONS

In conclusion, this project successfully demonstrated molecular adsorber coatings as a sprayable replacement to adsorber pucks for the mitigation of on-orbit molecular contamination. Development and testing of the coating has proven its adhesion performance, thermal stability, and adsorption capabilities. These results are very promising and have shown that molecular adsorber coatings have the potential to protect sensitive surfaces from the damaging effects of outgassed materials.

The following is a list of accomplishments for this study:

- Established a standard substrate configuration using MLP-300-AZ as the base coating
- Optimized the formulation process by varying binder grades, binder to pigment ratios, pigment treatment, and viscosity
- Refined the spray application technique by using different surface treatments and spray gun control settings
- Developed an accelerated test method and test configuration for molecular capacitance testing that increased productivity to run multiple samples more efficiently
- Determined that the molecular capacitance of the coatings are comparable to the current adsorber puck technology
- Verified the formulas that passed adhesion performance with tape tests and thermal stability in vacuum thermal cycle testing at temperatures between -40 °C and 70 °C
- Characterized thermal optical property measurements, such as solar absorbance and emittance, as a function of coating thickness
- Selected formulas for further development and testing for qualification purposes

8. FUTURE WORK

Future work involves the qualification of molecular adsorber coatings for use on NASA's contamination sensitive spaceflight missions. This will involve finalizing the formula and spray application technique for repeatability and reliability. The qualification plan will consist of further testing molecular capacitance, adhesion performance, thermal cycle, and thermal optical properties for the final coating product. In particular, future molecular capacitance testing will replace stearyl alcohol with a contaminant source comprised of chamber residue, such as hydrocarbons, plasticizers, and other outgassing chemicals from lubricants and adhesives. New testing methods will also be conducted in the future. These include acoustic cleaning by shedding loose particles off the coating and outgassing testing by releasing the adsorbed contaminants from the coating. Lastly, black and conductive molecular adsorber coatings will be explored for use on optics and electrostatic discharge sensitive missions.

9. ACKNOWLEDGEMENTS

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