ALLOWABLE SOLUBLE SALT CONTAMINATION LEVELS FOR INDUSTRIAL PAINTING

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Abstract: Visual evaluation of surface preparation quality is insufficient for predicting coating system performance. Dehumidification to control flash rusting can mask problem surface contamination.

INTRODUCTION

Current industrial coating application practices usually rely on visual surface evaluations before painting. These ratings consider the degree to which visual contaminants, such as existing coatings and corrosion, have been removed. A visual evaluation is also used to gauge the deterioration (flash rusting or rustback) of the prepared surface before painting.

Testing of the blast cleaned surface for invisible surface contamination is becoming popular for critical service applications. These contaminants can promote rusting of the blast-cleaned surface and also degrade the performance of the applied coating system. Dehumidification is often advertised as a means to "hold" the prepared surface for a longer time frame. The test results discussed herein show how dehumidification may mask the effects of surface contaminants.

SURFACE CLEANLINESS STUDIES

We will review two practical studies of invisible surface contamination and its effects. They will show the effects of invisible surface contamination both before and after the coating system is applied. The surface rustback (flash rusting or re-rusting of blasted steel) research showed a general correlation between invisible contamination and the propensity for re-rusting of a blast cleaned surface. We will also review a study that showed the effect of painting over contaminated and flash rusted surfaces.

Surface Rustback - Experimental Procedure

An experiment was performed with the objective of justifying a longer "hold" time that specified for internal tank relining operations (1). This would allow painting contractors more time to abrasive blast the tank before having to clean-up and prime the prepared areas.

Two sets of steel panels were exposed to three levels of relative humidity to simulate the conditions inside storage tanks during relining work. The first set of panels consisted of new test panels covered with mill-scale. These were detergent cleaned and abrasive blasted to a high quality SP-10. These panels were intended to simulate areas of a tank with intact coating prior to blast cleaning (previously uncontaminated then blasted steel). The second set consisted of previously rusted steel panels (via marine atmosphere exposure). These were also detergent cleaned and abrasive blasted to a high quality SP-10. These panels were intended to simulate areas of a tank that were previously corroded and/or pitted prior to blast cleaning.

Replicates of the panel sets were tested for surface contamination levels and placed in constant humidity chambers. The humidity was controlled by exposing the solid phase of a deliquescent chemical in a saturated aqueous solution of the same chemical within the desiccator. The chambers were held at relative humidities of 94%, 42%, and 22% (corresponding to dew point/surface temperature differentials of 3°F, 25°F, and 41°F respectively). The ambient temperature, steel surface
temperature, and relative humidity within the desiccator were monitored during the exposures.

Panels were visually inspected daily for one week and then weekly for a total exposure time of one month. Inspections were conducted in general accordance with ASTM D610 "Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces." Panels were photographed before, during, and after each exposure.

Surface Rustback - Results and Discussion

The level of invisible contamination on the surface of the blasted panels was quantified by using a Bresle sampling kit to test for soluble chlorides. A contamination check was also made by measuring the conductivity of deionized water exposed to the blast cleaned surfaces. Both chloride and conductivity measurements were taken by allowing the extraction liquids to rest 5 to 6 minutes on the panel surface (2).

The results of the Bresle testing indicated that the chloride contamination on both sets of panels was below the detection limit of the test kit of (2.0 µgCl-/cm²). The conductivity testing resulted in an average conductance (using 3 ml of extract) of 26 µS/cm for the mill-scale/blasted panel sets and 61 µS/cm for the pre-rusted/blasted panel sets.

The levels of conductivity measured for the extraction water were approximately twice as high for the pre-rusted panels as they were for the mill-scale panels. The conductivity results (when converted to theoretical chlorides) also indicate that the contamination measured by this technique was not all chloride ions. The Bresle tests resulted in <2.0 µgCl-/cm² while the conductivity measurements converted to higher theoretical amounts of chloride. This indicates that the abrasive blast performed on the pre-rusted panels was unable to fully remove all contamination.

Figures 1, 2, and 3 graphically represent the rust ratings for the panels exposed at the three relative humidities. In each figure the ASTM D610 rust ratings are shown along the Y-axis. A rating of 10 indicates zero visible rust. The X-axis of the figures indicates the time in days of the exposure in the various humidity cabinets. Each plot shows the approximate amount of staining that is allowable under the definition of SP-10 (ASTM D610 of 4.58 or 5% surface rust).

Figure 1 shows the driest of the three conditions. This environmental chamber was held at approximately 22% relative humidity (RH) throughout the testing. At these conditions, the pre-rusted/blasted steel samples were the only samples to develop any rusting and none of the samples rusted beyond the 5% criteria of SP-10.

Figure 2 shows the data from the chamber held at 42% RH throughout the testing. Under these conditions the pre-rusted/blasted panels (more invisible contamination) rusted to approximately 5 – 10% of the surface area. The mill-scale/blasted panels exhibited minimal rusting at this environmental condition.

Figure 3 shows the rusting data from the 94% RH test chamber. Again, at this environmental condition the mill-scale/blasted panels did not significantly rust. The more contaminated (yet visually identical) panels rusted beyond 5% in two to three days. The amount of rust leveled off at 40 - 50% area rusted after approximately one week of exposure.

The 22% RH condition produced minimal to no rusting of any panels. Photographs for this condition are not shown.

As was shown in figure 2, the 42% RH condition resulted in rusting of the pre-rusted/blasted panels. Figure 4 shows a photograph of the samples exposed in the 42% RH environmental chamber after one month. The mill-scale/blasted panel has not rusted as much as the pre-rusted/blasted panel. Figure 2 showed how the rusting progressed over time on these panels.

The 94% RH chamber produced significant rusting of the pre-rusted/blasted panels. Figure
5 shows that, during exposure, significantly more rust developed on the pre-rusted/blasted panels than on the mill-scale/blasted panels. The relative amount of rust on the mill-scale/blasted panels was similar to that seen at the 42% RH condition.

Surface Rustback - Test Summary

- More rusting was evident on panels exposed at higher relative humidities, however the effects of invisible contaminants on the amount of rust was greater than the effects of relative humidity.

- Virgin steel (mill-scale covered) that was blasted to a high quality SSPC SP-10 had relatively low levels of invisible contamination as detected using conductivity measurements. These panels did not significantly rust under any of the tested environmental conditions.

- Steel that was previously rusted to an SSPC initial surface condition D and then blasted to a high quality SP-10 had higher amounts of invisible contamination as detected using conductivity measurements than blasted virgin steel. These pre-rusted/blasted panels had roughly 5-10% rust at a 25ºF dew point spread, and rusted well beyond SP-10 at a 3ºF dew point spread.

- Conductivity measurements of a blasted steel surface are a recommended quality assurance step. The results of this testing indicated that an extraction water conductivity of 30 µS/cm or less resulted in minimal rusting (conductivity measured using 3 ml deionized water and Bresle patch).

Performance Testing - Experimental Approach

The following study was performed to investigate the performance of epoxy coatings applied over chloride contaminated and flash rusted surfaces (3). The test matrix for the flash-rust evaluation was as follows. Duplicate panels were prepared for each test condition.

1. Nine Surface Preparations (all initially began in the SP-5 condition). Listed in increasing degree of visual flash rusting.
   a. SP-5 abrasive blast (Al2O3 2-3 mil profile)
   b. Dipped in DI H2O, Dried with Heat Gun
   c. Dipped in salt solution, Dried with Heat Gun
   d. Dipped in DI H2O, 8 hrs. ambient dry
   e. Dipped in salt solution, 8 hrs. ambient dry
   f. Dipped in DI H2O, 80 hrs. ambient dry
   g. Dipped in salt solution, 80 hrs. ambient dry
   h. Dipped in DI H2O, 72 hrs. ambient dry, re-dipped, 8 hrs dry
   i. Dipped in salt solution, 72 hrs. ambient dry, re-dipped, 8 hrs dry

2. The panels were prepared with two versions of MILSPEC coating systems. These were MIL-P-24441 Type IV two-coat system with a 12 mil target system DFT and MIL-P-24441 Type IV two-coat system with an 18 mil target system DFT.

3. For the flash rusting analysis, we exposed panels in the ISO 6270 test for coating resistance to condensing humidity, under static seawater immersion, and at our Sea Isle City Marine Atmosphere Exposure site.

   Determination of Resistance to Humidity (Continuous Condensation)

   We performed this test by International Standards Organization (ISO) 6270 "Paints and Varnishes - Determination of Resistance to Humidity (Continuous Condensation)." The test apparatus was a heated water bath in a closed container maintained at 40 ± 2ºC where the cover incorporated the panels tested. The cover maintained the panels at a 15 ± 5 degree angle from horizontal to permit drainage of condensed water. Condensation did not drain from one panel to another.
We inspected the panels for blistering by removing each from the apparatus, blotting dry with absorbent paper, and checking by the standard blistering (ASTM D714) inspection procedures.

**Seawater Exposure**

Duplicate coated panels were immersed in static natural seawater. The panels were exposed in a non-metallic chamber at ambient temperature. Sufficient make-up seawater was added to the chamber to eliminate stagnation effects. Panels were inspected at regular intervals for rusting and blistering.

**Sea Isle City Marine Exposure Site**

At the marine exposure site, duplicate test panels were exposed on test racks facing south at a 45 degree angle to the horizontal. The test site is located 100 feet from mean high tide of the Atlantic Ocean in Sea Isle City, New Jersey. The panels are sprayed two times daily with natural seawater. They were inspected at regular intervals for rusting and blistering.

**Performance Testing - Results and Discussion**

Figure 6 shows (9) nine panels representing each of the flash rust conditions before painting. The visual appearance of the flash rust for each condition was consistent. Also identified in the figure is a visual flash rust classification to be used in the current analysis. These classifications are as follows:

a. SP-5 Control - 4 panels  
b. SP-10 Appearance - 16 panels  
c. Slight Rust - 12 panels  
d. Medium Rust - 4 panels

After 14 months of exposure, the test panels in the seawater immersion and marine atmosphere exposure are showing only minor corrosion and blistering around the panel edges. The test panels in the ISO test are showing the results that will be analyzed herein.

The ISO test panels were evaluated at five intervals during the first 14 months of exposure. Rusting was only observed on two of the thirty-six panels at apparent pinholes.

The blistering data alone serves as the basis for the following discussion. ASTM D 714 blister ratings include a rating for blister size (0 through 10, with 10 being no blisters) and density (Few, Medium, Medium Dense, and Dense). To allow easier analysis of the data, a conversion table was developed that translates the alphanumeric rating into a simple 1-10 scale. These numeric ratings can be easily averaged and manipulated to evaluate the various variables in the test. In all figures the performance of the SP-5 control is included as a reference. The data for the SP-5 panels is the average of duplicate panels for the two coating systems (i.e., four panels total). The data showed no differences between conventional and high-build 24441 Type IV systems over any of the surfaces. Thus for the remainder of the subject analysis, the data from all panels of each system type will be averaged, i.e., the analysis will not segregate the results by conventional vs. high-build system.

Figure 7 shows the effect of visual rust level on the blistering rating. To develop this graph each condition was classified as described above and in figure 6. Based on this visual classification, one might expect that the Medium Rust panels ought to have performed the worse. Yet these systems were closest in performance to the SP-5 control systems. This suggests that something other than the visual appearance is affecting the coatings’ blistering resistance.

Figure 8 shows the blistering ratings for the panels that were rusted by dipping in the chloride contaminated water versus those that were rusted by dipping in the deionized water. Clearly the panels dipped in the chloride-laden solution blistered more than those exposed to the deionized water solution.

Figure 9 shows the blister ratings observed on the panels dipped in deionized water before the flash-rusting period. Note from Figure 6 that the only panels of those exposed to the deionized water to "rust" were those that were allowed to dry for 72 hours with a "re-dip" 8
hours before the conclusion of the pre-exposure. These panels performed the worst in terms of blistering resistance.

Figure 10 shows similar data for the panels pre-exposed to the chloride solution. Different results were obtained. The worst performers were those force-dried immediately after dipping or those allowed to dry in 8 hours. The panels allowed 3 days dry-time, with or without a re-dip, performed similarly to those from the deionized water pre-exposure. The force-dried and those dried over 8 hours performed significantly worse than any other system.

One possible explanation for this behavior may involve reactions on the panel as it dries and "flash-rusts." Those panels pre-exposed to the chloride solution and allowed to dry quickly or be force-dried may have more soluble substrate-chlorides compared to those with longer dry times. Over a longer dry time, chlorides may be complexed into less soluble corrosion products. Following moisture penetration through the coating and accumulation at the substrate, the osmotic forces are proportional to the soluble species concentration in this electrolyte. Thus, the more highly complexed and potentially less soluble species do not generate the same osmotic pressure (i.e., force) for blistering. Should this be the case, evaluation of the propensity for blistering must consider the actual under-coating wetting process. In this process the dissolution of various substrate complexed species will be related to the local volume of the solvent.

**CONCLUSIONS**

- During the subject study, re-rusting or flash rusting of a blast cleaned substrate was investigated. Corrosion was more related to surface contamination than to high humidity levels.

- Caution should be exercised when assuming the visual absence of flash rust on a blast-cleaned steel surface equates to a suitable painting surface. Invisible contamination may still exist and be comprised of soluble species other than chlorides. The presence of these contaminants will cause flash rusting/coating deterioration over time.

- The propensity for flash rusting to affect coating performance may not always be judged by the surface visual appearance (i.e., in this research, more rust was not always more detrimental to coating performance). Some accounting probably has to be made of the ionic species that may be present on the surface after flash rusting.

- The use of dehumidification to retard the development of flash rusting may mask a surface condition deleterious to coating performance.

**REFERENCES**


2) Ake Bresle, "Conductimetric Determination of Salts on Steel Surfaces," Materials Performance, Volume 34, Number 6, June 1995.

3) James A. Ellor, "The Effects of Surface Contamination on Paint Performance," prepared for Naval Sea System Command, SEA 03M1, April 1996.
Figure 1. Rust vs. Time at 22% RH.

Figure 2. Rust vs. Time at 42% RH.

Panel Rust Development
40deg Dewpoint spread (22% RH)

Panel Rust Development
25deg Dewpoint spread (42% RH)
Figure 3. Rust vs. Time at 94% RH.
Figure 4. Pre-rusted/blasted and mill-scale/blasted, one month, 42% RH.

Figure 5. Pre-rusted/blasted and mill-scale/blasted, one month, 94% RH.
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<th>DIW Dipped Hot Air Dry</th>
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<th>DIW Dipped 3 Days to Paint</th>
<th>DIW Dipped 72 Hours to Dry then re-dip and 8 Hours to Dry</th>
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<td>&quot;SP-10&quot; Appearance</td>
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Figure 6. Appearance of Panels for Flash-Rust Study Before Coating.
Figure 7. Visual Rusting vs. Performance.

Figure 8. Surface Contamination vs. Performance.
Figure 9. Water Dipped Panel Performance.

Figure 10. Chloride Exposed Panel Performance.