High performance protective coatings often fail in the severe environment of the headspace in domestic wastewater collection and treatment systems. Coating failures are attributed to many factors, including extensive permeability to hydrogen sulfide gas (H2S) and other corrosive gases present. Although the chemical and physical properties of coating systems can be determined in the laboratory, this is not the case for the effects of environmental conditions, such as exposure to severe environments within wastewater headspaces.

Unfortunately, specifiers for the wastewater sector are often faced with selecting from an array of protective coatings that have not been subjected to testing specifically for the wastewater environment, in large part because of inadequate laboratory tests for coating performance in all the conditions in the facility. This article discusses the advances in a novel cabinet testing protocol designed to simulate the effects of the severe conditions in a wastewater headspace. This test protocol produces data that can be interpreted in 28 days.
Background

The U.S. municipal wastewater infrastructure is deteriorating rapidly due in part to the effects of biogenic sulfide corrosion. Biogenic sulfide corrosion is a bacterially-mediated process in which hydrogen sulfide (H$_2$S) is formed and subsequently undergoes biological oxidation to form sulfuric acid (H$_2$SO$_4$). Sulfuric acid attacks concrete and steel within wastewater headspaces.

Domestic wastewater varies widely in composition. The main component is water (~95%) added during flushing to carry the waste to the drain. Other components of wastewater include pathogenic and non-pathogenic bacteria, organic particles, inorganic particles, animals, macro-solids, and emulsions. The typical pH of domestic wastewater is 6.0 to 9.0. Although septic in nature, the untreated wastewater itself is not particularly detrimental to the concrete or steel infrastructure. Rather, H$_2$S gas in the headspace above the waterline in enclosed sewer pipes and structures is principally responsible for subjecting the concrete and steel appurtenances in the headspace to highly corrosive exposures.

Hydrogen sulfide gas has always been present in collection systems up to 10 parts per million (ppm). However, in the past few decades, as a result of changes related to water conservation, industrial pretreatment, and design philosophies, the conditions in wastewater collection and treatment have become more aggressive. The changes have produced H$_2$S concentrations exceeding 100 ppm (and occasionally measured upwards of 1,000 ppm). The changing conditions have contributed to rapid deterioration of the wastewater infrastructure.

Traditional coatings, as well as high-build protective coating and lining technologies, are routinely failing under severe wastewater conditions, leading to a need for costly renovation of sewer networks and treatment structures. Low permeability of coatings and linings to the corrosive gases and liquids in the wastewater vapor phase has been shown to substantially increase coating performance. Although perhaps not linear, the atmospheric H$_2$S concentrations appear to be proportional to the rate of sewer corrosion. It can also be assumed that increased H$_2$S concentrations contribute to greater permeation of polymeric coatings and linings. These sewer gases, particularly H$_2$S, compromise the barrier qualities of a protective coating. In the presence of moisture, H$_2$S can be biologically oxidized to form H$_2$SO$_4$, which rapidly attacks the underlying substrate.

Several notable wastewater testing programs, including the “Evaluation of Protective Coatings for Concrete,” performed by the Sanitation Districts of Los Angeles County and the “Chemical Resistance Pickle Jar Test,” developed by the Standard Specifications for Public Works Construction (Greenbook), have been performed throughout the years to
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test the viability of high-performance protective coatings in wastewater exposures.8,9 These testing programs provide a good qualifier for the suitability of a coating for wastewater structures. However, simulated wastewater environments have been studied mainly by using sulfuric acid (and other individual reagents) directly as the corrosive agent and may not reflect corrosive conditions found in wastewater headspaces, which include H2S gas. In other words, the testing basis of these programs is strictly chemical immersion. Likewise, some investigations have shown that even if concrete shows a certain resistance to H2SO4, it does not always indicate resistance against biogenic sulfide corrosion.10 In addition, these existing programs have no readily available means to characterize a coating and the quality of permeation resistance.

Objective of Study
In 2000, through a collaborative effort, researchers pioneered a laboratory testing protocol to rapidly evaluate the performance of coating systems for their resistance to permeation by H2S and H2SO4. This testing program, named Severe Wastewater Analysis Test (S.W.A.T.), was based on a testing chamber (Fig. 1) that permits the simulation and acceleration of the conditions characteristic of severe environments in wastewater headspaces. The evaluation method allows a comparative evaluation of the performance of commercially available products intended for severe wastewater exposures. It differs considerably from other laboratory testing methods by evaluating a material’s permeation resistance to elevated concentrations of H2S gas.

For the ultimate in coating evaluation for wastewater, field exposure is still the gold standard. However, some of today’s advanced high-build protective linings with low permeation characteristics often require over 10 years of field testing to generate usable data. Additionally, field conditions may be mild (relative to H2S), inconsistent, or changeable during the test period. As a result, performance claims have often been based upon anecdotal evidence of field histories. The role of the chamber is to provide a standardized, accelerated method for the evaluation of a coating’s performance in wastewater headspace conditions.

In 2003, Briand and Nixon presented data on common high-performance protective coatings subjected to the Severe Wastewater Analysis Testing program.11 They concluded that permeation resistance is the key factor in the successful performance of coatings placed in wastewater headspaces. The authors also proposed a laboratory testing protocol as a measure of a coating’s permeation resistance to these corrosive environments. This article updates the advances in the testing procedure for coatings in severe wastewater exposure.

Accelerated Testing Parameters
It is generally accepted that cabinet tests provide comparative results and not absolute results.12 Hence, the role of the wastewater chamber is to provide an accelerated evaluation of a coating’s relative performance under simulated wastewater headspace conditions. The corrosion protection of steel and concrete by a protective coating or lining may be altered by exposure to elevated gases and by the composition of the corrosive media. Exposing coated steel and con-
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crete panels to the wastewater chamber can help determine the suitability of these coatings or linings.

**Severe Wastewater Analysis Test (S.W.A.T.)**

The severe wastewater chamber is nothing more than a static testing vessel used to expose coated test specimens to a corrosive environment at elevated temperatures, so that the effect of such an environment can be evaluated. The test simulates severe wastewater headspace conditions by cyclic wetting of coated samples with a corrosive solution containing sulfuric acid and exposing the wetted samples to air containing high concentrations of hydrogen sulfide gas. The S.W.A.T. procedure is unique in that it simulates the headspace environment of enclosed wastewater structures, where permeation by hydrogen sulfide gas (and other gases present) alters the properties of high-performance lining systems. It is ultimately this aggressive mixture of liquid droplets in the presence of \( \text{H}_2\text{S} \) that rapidly permeates a protective film.

Chemical selection for the S.W.A.T. is based on the easily detectible corrosive species in headspaces: \( \text{H}_2\text{S} \) and \( \text{H}_2\text{SO}_4 \). (Other gases are also present in these environments and can be incorporated into the wastewater chamber.)

Prior work by Briand and Nixon established gas concentrations and testing duration by evaluating the permeation performance of various polymers at varying levels of \( \text{H}_2\text{S} \) gas.\(^1\) It was concluded that the permeation performance of various protective coatings tested in the S.W.A.T. for a period of 28 days paralleled their performance in the field.

Additionally, preliminary studies using bare (uncoated) concrete specimens exposed to the S.W.A.T. chamber indicate a concrete mass loss of approximately 0.877 in. (2.2 cm) in a year. This mass loss parallels many documented cases of concrete paste loss of nearly 1 in. per year from walls and soffits (ceilings) under severe field conditions.\(^1\)\(^3\)

The average temperature of wastewater in the U.S. is between 10 and 21 C (50 and 70 F). The S.W.A.T. operates at a temperature of 65 C (150 F) to induce an accelerated reaction rate that is approximately 3 times the actual rate for wastewater headspace conditions.

The corrosion of sewers and other facilities and odorous gases in sewers are principally related to the generation of hydrogen sulfide. It has also been found that permeation by hydrogen sulfide gas (with other sewer reactants) alters film properties of protective coatings and contributes to their blistering and cracking.\(^1\) The gas content of 500 ppm \( \text{H}_2\text{S} \) was selected for S.W.A.T. based upon earlier studies showing paralleled coating permeation results when testing with greater concentrations (10,000 ppm \( \text{H}_2\text{S} \)).\(^1\) Therefore, the 500 ppm is used to expose samples to realistic levels of hydrogen sulfide that may be encoun-

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**Fig. 4 (above):** Summary of EIS impedance data for 3 different coatings technologies: Control, and Post-S.W.A.T. Product A (left): Coal-Tar Epoxy—no retained impedance properties; Product B (middle): Novolac Epoxy—no retained impedance properties; Product C (right): High-Build Amine Epoxy—98% retained impedance properties.

**Fig. 5 (right):** Example of optical microscopy measurements of coating applied to concrete cylinder specimen.

**Permeability**

<table>
<thead>
<tr>
<th>Control</th>
<th>Post-S.W.A.T.</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.9</td>
<td>11.5</td>
</tr>
<tr>
<td>10.4</td>
<td>10.2</td>
</tr>
<tr>
<td>98%</td>
<td></td>
</tr>
</tbody>
</table>

**Tensile Properties**

<table>
<thead>
<tr>
<th>Product</th>
<th>Tensile Strength, psi (ASTM D638)</th>
<th>Elongation, % (ASTM D638)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Product D</td>
<td>3672 (55%)</td>
<td>1643 (55%)</td>
</tr>
<tr>
<td>Product E</td>
<td>362 (31%)</td>
<td>248 (31%)</td>
</tr>
</tbody>
</table>

**Fig. 6:** Example results for testing the tensile properties. Product D, an elastomeric polyurea, lost 55% tensile strength properties (left) and 31% elongation properties (right) when exposed to S.W.A.T. cabinet.
tered under field conditions. Air containing 500 ppm H$_2$S is bubbled through the aqueous solution to super-saturate the solution.

Other gases commonly found in untreated wastewater include carbon dioxide, methane, and ammonia. Like H$_2$S, these gases are derived from the decomposition of the organic matter present in wastewater. Since the concentrations of the latter three gases are not widely understood relative to H$_2$S within wastewater collection systems, they have been withheld from testing until further research is conducted. (Data collection is currently underway by the authors for future incorporation into the S.W.A.T. protocol.)

As H$_2$S levels have increased within severe wastewater environments, so has the production of H$_2$SO$_4$ by Thiobacillus sulfur oxidizing bacteria (SOB) that colonize in the headspaces. The theoretical concentration of H$_2$SO$_4$ generated by SOB is proposed to be 5 to 7% H$_2$SO$_4$. The S.W.A.T. incorporates 10% H$_2$SO$_4$ into the aqueous phase of testing; this concentration of acid is slightly above the maximum observed produced by the SOB. In coastal areas, salt (sodium chloride or NaCl) in water vapor can be very damaging to steel surfaces. The concentration of 0.4% (or 4,000 ppm) sodium chloride was incorporated into S.W.A.T.

Testing Procedures

The suitability of a particular lining system in severe wastewater environments is based upon the retained properties of the coating with regard to permeability, to enhance the solute conductivity and to duplicate saltwater intrusion found in many of these coastal collection systems. The aqueous solution is also saturated with H$_2$S by bubbling the air-H$_2$S gas mixture through the solution.

Permeability

Polymeric coatings act as a barrier separating the substrate from the corrosive service environment. A key attribute in the performance of the protective coating is, therefore, a low permeability to salts, water, gases, acids, and other corrosive species.

Electrochemical impedance spectroscopy (EIS) analysis is a technique well suited for evaluating a coating’s permeability (barrier) properties based on the electrical resistance provided by the coating. This is referred to as impedance. The impedance of the coating is related to the nature of the polymer, its density, film thickness, and fillers. EIS has been widely used in the laboratory and field within the protective coatings industry for determining a coating’s performance and obtaining quantitative information on coating deterioration. When used with cabinet tests, EIS analysis acts as a quantitative detector of coating quality.

When interpreting permeation resistance using EIS, the higher and more stable the retained impedance following exposure, the better the long term permeability resistance and, therefore, the better the long term coating performance. The logarithmic impedance scale presented in Fig. 3 (p. 46) is derived from a large body of literature on laboratory and field work.

EIS control readings are taken before the coating is exposed to the S.W.A.T. and then compared to post S.W.A.T. impedance to determine if the polymer was permeated or attacked during the test. Any polymer degradation is easily detected by a decrease in the measured impedance.

Experimentally, impedance of a coating is determined as a function of the frequency of an applied AC voltage. The data consist of a Bode plot of Log Z versus Log f, where Z is impedance in ohms cm$^2$ and f is frequency in Hertz.
(0.05 Hz to 100 kHz). From the Bode plot, \( \log Z_{0.1\text{ Hz}} \) is determined by interpolation. The \( \log Z \) value at 0.1 Hz is tabulated and used as the basis of comparison between coatings and for monitoring the change of a coating as a function of exposure time to the test environment. Selection of \( \log Z_{0.1\text{ Hz}} \) is somewhat arbitrary but represents a compromise between speed of analysis and the selection of a frequency at which differences in coating performance can be reliably determined.\(^{22}\)

An example of EIS analysis of three products commonly used in wastewater is compared in Fig. 4 on p. 49. (The red line at \( \log Z 6.0 \) is an indication of where "barrier protection begins".) Product A—a coal tar epoxy—exhibited excellent initial impedance values. However, the product blistered and cracked during S.W.A.T. exposure and showed no retained impedance. Similarly, Product B—a high-build novolac epoxy—demonstrated excellent initial EIS impedance values. However, this product also showed no retained impedance following the S.W.A.T. exposure. Product C—a fiber reinforced high-build amine epoxy—showed excellent initial EIS impedance values. Following the 28 day S.W.A.T. exposure, the product retained 98% impedance. This is an indication of the product’s low permeation to the corrosive wastewater species.

In addition to EIS analysis, permeation of a coating following wastewater cabinet exposure can also be assessed by microscopically observing the cross-section of the coating film. Permeation by the severe wastewater reagents typically manifests as discoloration when viewed with 100X microscope with digital imaging.

Figure 5 (p. 49) shows a high-build amine epoxy applied at an average of 74 mils (1,850 microns) dry film thickness (DFT) to a cylindrical concrete specimen. The concrete specimen is cut to expose a cross-section of the coating. The cross-section of the film is then measured with
following post-S.W.A.T exposures.


Tensile control properties are established for each candidate lining system using one of the aforementioned ASTM methods. For comparison, the specimens are then subjected to the S.W.A.T., and tensile strength is measured again. Figure 6 (p. 49) is an example of such tensile testing, where Product D—an elastomeric polyurea—exhibited excellent initial tensile and elongation properties. However, when subjected to the wastewater cabinet, the tensile properties of Product D were significantly reduced by 55% and elongation was reduced by 31%. A deduction can be made that this polymer technology is significantly embrittled by the severe wastewater exposure.

A polymer sample has flexural strength if it is strong when one tries to bend it. Evaluating the flexural properties is important to determine the effects of severe wastewater exposures on the polymer. Like tensile properties, a significant decrease in retained flexural properties may indicate that the polymer is losing plasticity or becoming embrittled and may ultimately crack under long-term field conditions.

Physical Testing

In addition to the measurement of the permeation resistance of a particular lining system, the assessment of physical effects on the lining system is useful in detecting any significant changes a polymer may undergo as a result of severe wastewater exposures. For example, physical testing can reveal whether the lining system loses its tensile or flexural properties and becomes embrittled from exposure to these environmental conditions. When incorporated into the S.W.A.T., the quantitative determination of these properties can be tracked at a small fraction of the exposure time usually required for these changes to be discerned under actual field exposures. Two laboratory tests used to measure tensile strength and flexural strength can be used in a general way to assess a coating’s suitability in wastewater. These mechanical properties are assessed by subjecting coatings to an applied force and determining their behavior under it. Any changes from the control (laboratory) condition are compared to the results using one of the aforementioned ASTM methods.

Figure 7 (p. 50) compares the initial and post-S.W.A.T. flexural properties of three high-build amine epoxy trowel-applied mortars marketed for severe wastewater applications. A deduction can be made, based upon this comparison, that it is not the greatest initial flexural strength (psi) that is important, but rather the retained flexural properties.

Product E lost only 9% of its flexural properties compared to Products F and G, which were reduced by 20%. This sharp decline of flexural strength in the latter two products indicates greater attack on the mechanical properties of the polymers and perhaps a greater propensity to crack under long-term wastewater field conditions.

Other ASTM laboratory testing methods are available to measure the mechanical or physical properties of coatings and may be incorporated into the S.W.A.T. to measure the effects of the wastewater exposure.

One such testing method is ASTM D4541 Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers. This test method delineates a procedure for evaluating the direct tensile strength (commonly referred to as adhesion) of a coating on steel. The adhesion test consists of dollies made of aluminum, which are glued per-
perpendicular to the coated surface of the samples. After the curing of the adhesive (glue), the testing apparatus is attached to the loading fixture (dolly) and aligned to apply the tension (normal stress) to the test surface. The force applied to the loading fixture is then gradually increased and monitored until either a plug of coating material is detached or a specified value is reached.

Another method used to assess the adhesion of the candidate lining systems is the parallel scribe method, which is often used with NACE TM0185 Evaluation of Internal Plastic Coating for Corrosion Control of Tubular Goods by Autoclave Testing. This test method is conducted by making two cuts, ¼ to ½ in. (3 to 6 mm) apart, through the coating to the substrate (Fig. 8 on p. 49). Adhesion of the coating between the scribe marks is evaluated by prying the coating with a utility knife and comparing the result with the rating scale below.

Although perhaps considered subjective, the parallel scribe adhesion method is useful in determining the adhesion effects of undercut corrosion and black rust that may not necessarily be observed using ASTM D4541 direct tensile adhesion methods.

Similar to the other mechanical testing, the testing of adhesion properties is performed prior to (control) and following exposure to the S.W.A.T. cabinet. A significant loss of adhesion is an indication that the lining material is being permeated and is affecting adhesion to the substrate at the bond line.

Visual Inspection
The last measure to determine the suitability of a candidate lining system for severe wastewater is visual inspection. Visual inspection identifies any physical alterations of a polymer following cabinet
exposure to corrosive conditions. For example, the lining system is assessed for blistering, cracking, or rusting (pinpoint or otherwise) of the coated panel.

The rusting of the surface is assessed in accordance with ASTM D610, Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces. Blistering is assessed in accordance with ASTM D714, Standard Test Method for Evaluating Degree of Blistering of Paints. As seen in Fig. 9 (p. 52), many protective coatings cannot withstand the permeation of the corrosive species found in severe wastewater conditions and ultimately blister and crack.

Any checking or cracking of the film is visually identified on the steel and concrete specimens. The extent of checking or cracking can be identified as described in ASTM D660, Standard Test Method for Evaluating Degree of Checking of Exterior Paints, and D661, Standard Test Method for Evaluating Degree of Cracking of Exterior Paints. Figure 10 (p. 52) shows two novolac epoxy liners that cracked following S.W.A.T. exposure.

Summary

Asset management philosophy has municipalities and water agencies looking to protect their critical wastewater infrastructure from the destructive effects of biogenic sulfide corrosion with high build protective linings. But where other services such as atmospheric or marine have accelerated and other lab tests available, the wastewater coatings industry has not had suitable laboratory testing procedures to evaluate coatings for wastewater environments. Instead, the industry has had to rely on anecdotal evidence as performance markers for use under these corrosive conditions, and anecdotal evidence is not considered adequate to predict product performance.

An accelerated Severe Wastewater Analysis Test has been developed to simulate severe wastewater headspace conditions. The S.W.A.T. protocol provides interpretable data for product evaluation in fewer than 30 days. Manufacturers, recognized testing agencies, and technical organizations need to incorporate this accelerated cabinet protocol into their evaluation programs when comparing materials for the protection of their critical wastewater conveyance and treatment assets.

References

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