Protecting Secondary Containment from Pesticides and Fertilizer

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Concrete has been the material of choice for building secondary containment structures because it is relatively inexpensive and has structural properties that make it ideal for supporting the loads of vehicles and large tanks. The chemical properties of concrete, however, make it susceptible to corrosion by common fertilizers. Though fairly impervious to water penetration, concrete is easily penetrated by vapors and organic solvents. This article describes research requested by the Environmental Protection Agency’s (EPA) Office of Pesticide Program to help identify coatings for concrete that could reduce the migration of pesticides through containment structures. EPA’s concern with concrete is due in part to the poor performance of drip pads at some lumber treating facilities. At these sites, wood preserving chemicals have passed through concrete pads and contaminated underlying soil and ground water. A preliminary study revealed that herbicides penetrated concrete samples after 5 simulated spills despite being rinsed shortly after each spill.

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Some states require concrete secondary containment to be sealed or protected with a coating when exposed to fertilizers and pesticides. Many retailers have used penetrating sealants to satisfy these laws, even though sealers provide little protection from chemical attack, and they do not prevent pesticides from penetrating the surface. Other retailers, who have applied thick film coatings that were properly selected for the intended service, have had disastrous results because the coatings were applied under poor ambient conditions. Consequently, there is much skepticism regarding the performance and benefit of protective coatings. The primary objective of the study described here was to determine the performance of concrete and organic coatings in containment structures designed for bulk agricultural pesticides. Bulk products are a fraction of the pesticides in common use for agricultural purposes; most are herbicides. The coatings tested in this project were evaluated for cost, ease of application, and performance.

Selecting Test Materials

Resource constraints made it impossible to test the hundreds of available commercial coating products and bulk pesticides. To test many coatings and pesticides, a two-tiered test program was used. Screening
tests identified products for more extensive testing. Pesticides that were nonreactive with many coatings were eliminated from subsequent tests, as were coatings that were sensitive to chemical attack from several pesticides.

In the first series of tests, 12 coatings were screened in 10 pesticides and urea-ammonium nitrate (UAN) fertilizer solution (32-0-0 UAN grade). Free films of coatings were used in screening tests for 2 reasons: first, coatings could be evaluated with exposures as short as 21 days, and second, physical properties of coatings were easier to measure in free films than on concrete.

In the second series of tests, the 4 most corrosion-resistant coatings, 3 most corrosive pesticides, and the UAN solution were tested. Concrete specimens were coated to determine the ability of coatings to protect the concrete in test chemicals.

Pesticides were selected from a list of 27 of the most common bulk products used in the U.S. Of these, 13 were combinations that included a product selected or were similar to a product selected. Ten of the 27 products were selected by eliminating aqueous solutions, mixtures, and products with duplicate chemistry (Table 1). Some aqueous solutions were omitted because they are less reactive than solvent-borne materials. Atrazine was selected because it is the most common pesticide in ground water. An insecticide, dimethoate, was added at EPA’s request. Dimethoate was chosen because it easily penetrates and softens certain plastics.

Coatings were selected from a list of candidates supplied by manufacturers that market products for coating concrete at retail agri-chemical facilities of 20 companies contacted. Most offered 1 or 2 products to protect concrete from immersion in pesticides containing organic solvents such as xylene and chlorobenzene. At least 1 of each of the generic coatings commonly used in secondary containment was tested: urethane, epoxy phenolic, polysulfide, urea urethane, vinyl ester, and epoxy novolac. Table 2 lists coatings, resin cost per area, and minimum dry film thickness (DFT).

Test Procedures

Screening Tests
The test procedures, including fabrication of specimens and pre- and post-test evaluations, were designed to conform as closely as possible to 5 ASTM standards: D 823, D 1005, D 3924, D 2370, and D 3363.

Specimen Preparation
Fabrication of the test specimens was based on ASTM D 823. The coatings were prepared in accordance with the manufacturers’ instructions. After thorough mixing and stabilizing, the coating mixture was poured on a thin, flat sheet coated with polytetrafluoroethylene and spread uniformly and evenly using a rod coated with the same material. Since most of the coatings were high solids formulations, only 1 coat was required to obtain the desired thickness.

After drying for a minimum of 94 hours, the test specimens were cut from the sheets using a double-blade precision specimen cutter or a cutting tool made for acrylate resin. Special care was taken to insure that no chipping, shearing, or other defects were introduced. Each specimen was 1 in.
x 6 in. (25 mm x 150 mm). The DFT of the specimens was measured using a digital coating thickness gage if coating thickness was less than 50 mils (1.3 mm), or a digital caliper if coating thickness was 50 mils (1.3 mm) or greater. The average DFT was determined from at least 3 measurements across the length of the specimen.

Following thickness measurements and visual examination, the test specimens were washed in soapy water for 1-2 minutes to remove dirt and oils, and then degreased in alcohol for 2-3 minutes. After the specimens were allowed to stabilize for at least 4 hours, the mass of the test specimens was determined to within 0.1 mg using a calibrated digital balance. The data were recorded in a computer database.

For each coating, 1 specimen was selected for pencil hardness tests in accordance with ASTM D 3363, and 5 were selected for tensile testing.

Tensile tests were conducted using 1 in. x 2 in. (25 mm x 50 mm) specimens. The tests were performed on a computer-controlled universal test machine. Test speeds were based on the rigidity of the coating and ranged from 5 to 55 percent elongation per minute. Test data, including ultimate tensile strength, percent of total elongation, and elastic or Young modulus, were determined by averaging the results of the 5 test specimens.

Test solutions were full strength formulations of pesticides purchased from a local agri-chemical retailer. Table 1 lists common names of these chemicals. Urea-ammonium nitrate fertilizer (32-0-0 UAN) was prepared using 35 weight percent commercial granular urea, 45 weight percent commercial granular ammonium nitrate, and 20 weight percent tap water.

Six specimens of each coating were immersed in approximately 1 L (0.95 qt) of each test solution. The test was conducted for 21 days without agitation. The test specimens were observed every 24 hours. After 21 days, the test specimens were removed, cleaned, weighed once more, and examined for pattern deterioration.

**Post-Test Evaluation**

Coating evaluations were based on comparison of the data obtained before and after the test. Included were changes in color and appearance, expansion, softening, swelling, chemical attack, aging, percentage of weight gained or lost, and deterioration of mechanical properties, such as ultimate tensile strength, percent of elongation, elastic modulus, and hardness.

The volumetric absorption rate (VR) was determined by the weight change per unit of volume. The weight change consisted of the absorption of the test chemical into the body of the test specimen per unit of time or a weight loss due to the coating deterioration per unit of time. This quantity was calculated using the following equation:

\[
VR = \frac{365 \times (M_f - M_i)}{(t \times v)}
\]

where VR is volumetric absorption or deterioration rate in mg/cm³/yr; Mi is initial weight of test specimen in mg; Mf is final weight of the test specimen in mg; v is volume of test specimen in cm³; and t is total exposure time in days.

The percent of weight gain due to ab-
sorption of corrosive species or loss due to coating deterioration was calculated using the following equation.

\[ P = \frac{(M_f - M_i) \times 100}{M_i} \]

A positive value for P indicates a weight gain; a negative P indicates a weight loss.

**Evaluation Criteria**

ASTM standards\(^2\) offer several guidelines for evaluating the deterioration of organic coatings exposed to corrosive environments. Among these are evaluations of blistering (ASTM D 714), rusting (ASTM D 610), and cracking (ASTM D 661). However, these standards and criteria apply only to coatings on steel panels.

NACE International has published guidelines on evaluating elastomer materials in sour gas environments (NACE, 1991).\(^3\) Similarly, ASTM\(^2\) has published standards for evaluating the resistance of plastic to chemical reagents (ASTM D 543) and for evaluating rubber in immersion tests (ASTM D 471).

Papers by Fisher\(^4\) and Fisher and Carpenter\(^5\) include criteria for determining the acceptability of lining materials for immersion service. These papers state that an acceptable lining neither gains more than 10 percent by weight or volume nor suffers any weight loss after a 30-day exposure. Ideally, the lining should sustain no surface attack, embrittlement, softening, blistering, or color change. The basic criteria for assessing a coating’s suitability for pesticide secondary containment were selected from both ASTM and Fisher and Carpenter. However, a slight modification of these criteria was needed because the test duration was only 21 days instead of 30 days. Despite being designed for immersion service, these weight change criteria were chosen for these evaluations because most pesticide secondary containment floors are exposed continuously to spilled materials. Most secondary containment floors are level (poorly drained), and spills are not always cleaned up immediately. Thus, in this series of screening tests, the test coatings were classified by the following criteria.

- Coatings are considered satisfactory for handling chemical media if weight gain is less than 7 percent or weight loss is insignificant in 21-day exposure tests. (A value of less than 2 percent was considered insignificant in the test program.)
- Coatings are considered satisfactory for handling chemical media if there is no significant change in ultimate tensile strength (\(S_{UTS}\)), percentage of elongation (\(e\)), elastic or Young modulus (\(E\)), or pencil hardness. The changes in \(S_{UTS}\), \(e\), and \(E\) must have little effect on the mechanical performance of the coating during service.
- Coatings are considered satisfactory for handling chemical media if there is no surface attack, softening or swelling, cracking

<p>| Table 2 |</p>
<table>
<thead>
<tr>
<th>Coating Type, Minimum DFT, and Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coating Type</td>
</tr>
<tr>
<td>Aromatic urethane elastomer</td>
</tr>
<tr>
<td>Epoxy phenolic resin</td>
</tr>
<tr>
<td>Elastomeric polysulfide</td>
</tr>
<tr>
<td>Urea urethane</td>
</tr>
<tr>
<td>Flake-filled vinyl ester</td>
</tr>
<tr>
<td>Flake-filled epoxy novolac</td>
</tr>
<tr>
<td>100 percent solids polyamide-cured epoxy</td>
</tr>
<tr>
<td>Amino-amine-cured epoxy</td>
</tr>
<tr>
<td>Polyisocyanate epoxy</td>
</tr>
<tr>
<td>100 percent solids epoxy phenolic</td>
</tr>
<tr>
<td>90 percent solids epoxy</td>
</tr>
<tr>
<td>Polysulfide epoxy</td>
</tr>
</tbody>
</table>

\(a\)—Thickness includes aggregate filler for non-skid surface. 
\(b\)—No data available. 
DFT—Dry-film thickness. 
*1 mil=25 microns 
**To convert the value in this column to $/sq m, divide by 0.0929
Coated Concrete Tests

Concrete Specimen Fabrication

Concrete specimens were cylindrical in shape (2 in. [50 mm] in diameter and 4 in. [100 mm] high) and were fabricated in accordance with ASTM C 192-90. Type I Portland cement was selected for testing. The concrete mix was designed to obtain an average compressive strength of 3,000 psi (200 MPa), a slump of 3 in. (75 mm), an air content of 3 percent by volume, and a water-to-cement ratio of 0.5. Cement, sand aggregate, and tap water were mixed for 10 minutes. Slump was then checked and adjusted to approximately 3 in. (75 mm) by using water reducer and super plasticizer. Mixing continued for an additional 10 minutes. Test specimens were prepared by filling plastic molds with the concrete mix, vibrating for 1 minute, curing 28 days before removal, and abrasive blasting. Surface defects and cracks were patched using a ready-mix vinyl concrete patching material.

Coating Application

Surfaces of the concrete specimens were prepared in accordance with ASTM C 811-907 and NACE RP0376-76.8 Surfaces were abrasive blasted using 20/40 grit sand, etched and scrubbed using 20 percent by volume HCl solution, rinsed, and dried. Test specimens were stored at laboratory conditions (80 F [27 C] and 50 percent relative humidity) before coating application. Coatings were formulated and mixed according to manufacturers’ recommendations. All coatings were brush applied (except for parts of the polysiloxane system) to a desired DFT and cured for 4 days before exposure. For the epoxy phenolic coatings, the concrete surface was coated with 2 coats of 100 percent solids epoxy-blend filler sealer to seal pores and cracks. Then, 4 coats of epoxy phenolic topcoat were applied to obtain 25-mil (635-micron) minimum DFT. The epoxy novolac coating system consisted of 3 coats: a primer coat, a base coat, and a topcoat. Total DFT was approximately 25 mils (635 microns). The vinyl ester resin coating system consisted of 3 coats: a primer coating applied using a brush, a base coat containing aggregate filler applied using a putty knife, and a topcoat applied using a putty knife. Total DFT was approximately 35 mils (889 microns).

Chemical Exposure

Each specimen was immersed in 500 milliliters (17 fluid ounces) of the test chemical. Six specimens per chemical were used for each type of coating. Test duration was 90 days. Upon completion, test specimens were removed, cleaned, dried, photographed, and examined for evidence of deterioration. Three specimens were subjected to compressive tests (ASTM C 873-939), and the other 3 were exposed to freeze/thaw cycle tests (ASTM C 666-9210) for 7 days at temperatures ranging from 20 to 55 F (-7 to 13 C) and approximately 7 cycles per day before compressive testing. For control purposes, uncoated specimens were also subjected to chemical exposure, freeze/thaw cycling, and compressive tests.

Abrasion Tests

Abrasion tests were performed only on topcoats in accordance with ASTM D 4060-9011 using a digital abrader. Coating samples for abrasion testing were prepared by spreading the coating on a sheet coated with polytetrafluoroethylene, and cutting four-inch (100-millimeter) square panels. After a four-day cure, panels were exposed
to test chemicals for 3 months and then tested for abrasion. Abrasion resistance was determined using weight loss after 500 cycles under a 1000-gram (2.2-pound) load. Abrasion index (AI) is presented in mg/1000 cycles and calculated using the following formula:

$$AI (%) = \frac{(M_i - M_f) \times 10^3}{C}$$

where $M_i$ is initial weight in mg, $M_f$ is final weight in mg, and $C$ is the number of test cycles.

**Evaluation and Performance Criteria**

Coating performance and chemical effects on concrete were evaluated based on visual examination, in accordance with ASTM D 6608-71, D 661-86, and D 4449-90. Performance and effects were also evaluated on the basis of AI (ASTM D 4060-90) and compressive strength of test specimens. Degree of coating deterioration was determined by comparing coated specimens subjected to chemical exposure to specimens that were not exposed. For a coating to provide acceptable protection, the following criteria must be satisfied.

- Coating must be impermeable to test chemicals with no evidence of chemical penetration into concrete after prolonged exposure. It must not show signs of deterioration, including chalking, swelling, delamination, flaking, and expansion.
- Compressive strength of exposed coated concrete must retain at least 80 percent of the average value of uncoated and unexposed concrete.
- For abrasion service, AI of the test coating must be less than 150 mg/1000 cycles after chemical exposure.

**Results**

**Screening Tests**

Values for mechanical properties and average DFT of coatings before chemical exposure are shown in Table 3. Percentage of weight gain or loss, volumetric rate of absorption (VR), and mechanical properties of coating after exposure to atrazine and dimethoate are listed in Tables 4 and 5. Data for the other pesticides tested are in the original SSPC paper (SSPC 94-19).

**Urethane Resin**

Before chemical exposure, the urethane had a pencil hardness of approximately 5B (Table 3). After the 21-day exposure to pesticides, tests on the urethane coating produced mixed results. In atrazine, an aqueous solution, urethane showed no significant weight gain or loss (less than 1 percent). In bentazon, the coating weight increased slightly (approximately 2-3 percent). However, the weight gain of the coating was greater than 8 percent when exposed to the solvent-borne pesticides. Also, severe weight gains of 25, 27, and 103 percent occurred when the coating was exposed to clomazone, metolachlor, and dimethoate, respectively.

The visual examination showed that the coating failed in most test solutions due to softening, dimensional expansion and contraction, and cracking. The coating was dissolved or converted back to the liquid state when exposed to dimethoate. Due to unreliable mechanical test results, no post-test evaluations were conducted based on the ultimate tensile strength, total elongation, or elastic modulus.

**Epoxy Phenolic Resin**

The mechanical properties of the epoxy phenolic before exposure are given in Table 3. In addition to good mechanical properties, the epoxy phenolic coating exhibited good chemical resistance. Exposure to the pesticides did not damage the coating. The coating had a slight weight loss (less than 2 percent) after the 21-day exposure to atrazine, bentazon, clomazone, metolachlor, alachlor, and trifluralin. However, a slight weight gain was observed.
when the coating was exposed to pendimethalin, imazaquin+monochlorobenzene+pendimethalin (IMP), and dimethoate. The average weight gain in pendimethalin and IMP was about 2 percent, and in dimethoate the weight gain was about 6 to 8 percent.

Inspection of the coating's mechanical properties after exposure to atrazine and bentazon indicated that the pencil hardness of the coating decreased only 1 unit of hardness, from 3H to 2H. In clomazone, the coating hardness decreased about 2 units, from 3H to 1H. The hardness increased 1 unit in alachlor, trifluralin, and dimethoate, but was unchanged in metolachlor, EPTC-thiocarbamate, and IMP.

Contrary to the hardness test results, the results of the tensile tests showed that the coating aged significantly when exposed to most of the test chemicals, with the exception of dimethoate. The SUTS increased 21 to 37 percent in atrazine; 42 to 58 percent in bentazon; 57 to 66 percent in clomazone; 59 to 65 percent in metolachlor; and more than 100 percent in EPTC-thiocarbamate, alachlor, IMP, and trifluralin. The total elongation decreased 50 to 57 percent in atrazine, 21 to 43 percent in bentazon, 22 to 57 percent in clomazone, up to 29 percent in metolachlor, 21 percent in EPTC-thiocarbamate, 7 percent in alachlor, and 21 percent in trifluralin. Total elongation increased in pendimethalin, IMP, and dimethoate by 29, 14, and 43 percent, respectively.

### Table 3

<table>
<thead>
<tr>
<th>Coating Type</th>
<th>Avg DFT mils*</th>
<th>$S_{UTS}$ (psi)**</th>
<th>$\varepsilon$ (%)</th>
<th>$E$ psi**</th>
<th>Pencil Hardness</th>
</tr>
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<tbody>
<tr>
<td>Urethane</td>
<td>33</td>
<td>254</td>
<td>176</td>
<td>554</td>
<td>5H</td>
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<tr>
<td>Epoxy phenolic</td>
<td>35</td>
<td>2118</td>
<td>14</td>
<td>$6.50 \times 10^5$</td>
<td>3H</td>
</tr>
<tr>
<td>Polysiloxide</td>
<td>77</td>
<td>4861</td>
<td>230</td>
<td>$8.20 \times 10^3$</td>
<td>6H</td>
</tr>
<tr>
<td>Urethane urethane</td>
<td>90</td>
<td>2022</td>
<td>64</td>
<td>$1.20 \times 10^5$</td>
<td>2H</td>
</tr>
<tr>
<td>Vinyl ester</td>
<td>40</td>
<td>4805</td>
<td>1</td>
<td>$6.74 \times 10^5$</td>
<td>9H</td>
</tr>
<tr>
<td>Epoxy novolac</td>
<td>100</td>
<td>4119</td>
<td>2</td>
<td>$6.86 \times 10^5$</td>
<td>9H</td>
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<td>100% solids polyamide-cured epoxy</td>
<td>115</td>
<td>3542</td>
<td>1</td>
<td>$1.13 \times 10^5$</td>
<td>9H</td>
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<tr>
<td>Aminoamine cured epoxy</td>
<td>40</td>
<td>482</td>
<td>1</td>
<td>$1.15 \times 10^5$</td>
<td>HB</td>
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<tr>
<td>Polysiloxane</td>
<td>65</td>
<td>a</td>
<td>a</td>
<td>a</td>
<td>a</td>
</tr>
<tr>
<td>100% solids epoxy phenolic</td>
<td>68</td>
<td>4037</td>
<td>2</td>
<td>$1.48 \times 10^5$</td>
<td>6H</td>
</tr>
<tr>
<td>90% solids epoxy</td>
<td>30</td>
<td>629</td>
<td>12</td>
<td>$3.3 \times 10^3$</td>
<td>B</td>
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<tr>
<td>Polysulfide epoxy</td>
<td>26</td>
<td>1219</td>
<td>43</td>
<td>$1.9 \times 10^3$</td>
<td>HB</td>
</tr>
</tbody>
</table>

*Note: The ultimate tensile strength, total elongation, and elastic modulus were calculated from the average values of six coaxial tension test results for 3 test specimens. The pencil hardness test was performed on 1 test specimen each.

Avg—Average value based on measurements on 6 specimens
VR—Average volumetric chemical absorption
$S_{UTS}$—Ultimate tensile strength
$\varepsilon$—Total elongation
E—Elastic or Young modulus
*Not reported due to unreliable test data
$^*$1 ml=0.03 mm
$^{**}$1000 psi=6.895 MPa
dimethoate, the coating was softened. The $S_{UTS}$ decreased 36 to 41 percent.

Visual examinations showed no sign of deterioration on any test specimens. A slight discoloration was observed on the specimens exposed to pendimethalin and IMP. However, this was attributed to the color of chemical reaction between the test chemical and the coating.

**Polysulfide Resin**

Results of mechanical tests performed before chemical exposure indicated that the polysulfide was strong, very elastic, and soft (Table 3). After exposure to most test solutions, test results indicated unsatisfactory performance. The weight change was less than the 7 percent criterion in atrazine and bentazon. However, the coating weight increased significantly in EPTC-thiocarbamate (21 to 22 percent) and trifluralin (about 11 percent) and increased greatly in clomazone (51 to 64 percent), metolachlor (82 to 88 percent), alachlor (55 to 59 percent), pendimethalin (42 to 49 percent), and IMP (51 to 58 percent). In dimethoate, all test specimens failed completely due to swelling and softening and were not recovered. Visual examination showed no discoloration of the test specimens. Expansion and swelling due to the accumulation of solvent were the primary causes of coating failure. Due to the severe weight change of the coating specimens, no post-test mechanical evaluations were performed.

**Urea Urethane Resin**

The urea urethane is soft, but strong and elastic. The coating had an $S_{UTS}$ similar to that of epoxy phenolic but was approximately 4 to 5 times more elastic (Table 3). Similar to the urethane and polysulfide coatings, urea urethane did not deteriorate when exposed to water-borne herbicides.
atrazine and bentazon, but lacked resistance to solvent-borne products. Coating weight increased only 1 percent or less in atrazine and bentazon, approximately 5-8 percent in metolachlor, and 3-5 percent in trifluralin. However, the weight gain of the coating exceeded the 7 percent criterion in the other pesticides.

Visual inspection showed no sign of chemical attack on specimens exposed to atrazine and bentazon. Also, no dimensional changes were observed. These specimens were in excellent condition after the 21-day exposure. However, in solvent-borne materials, urea urethane failed due to softening and expansion caused by absorption of the chemicals. The coating also failed by cracking when exposed to clomazone and dimethoate. Due to the severe weight change of the specimens, no mechanical evaluations were performed after chemical exposure.

### Vinyl Ester Resin

The vinyl ester showed excellent resistance to all test chemicals. The weight change was minimal in all test conditions. In dimethoate, the most severe chemical, the coating weight increased only 0.4 percent. The greatest weight gain was caused by IMP, but it was less than 2 percent. All weight losses were less than 1 percent. Mechanical examination before chemical exposure revealed that the vinyl ester resin tested is a strong but brittle coating (Table 3). Hardness tests performed after chemical exposure showed only minor changes in the hardness values. The coating hardness decreased slightly in metolachlor and dimethoate to 5H and 3H, respectively. Results of tension tests showed slight changes in $S_{UTS}$ and elastic modulus, but not in the total elongation. The total elongation of the coating (e) was still about 1 percent after exposure to most of the chemicals. In

### Table 5
Results of Screening Tests on Coatings in Dimethoate

<table>
<thead>
<tr>
<th>Coating Type</th>
<th>Avg VR mg/cm²/yr</th>
<th>Weight Change, %</th>
<th>Pencil Hardness</th>
<th>$S_{UTS}$ psi*</th>
<th>e %</th>
<th>E psi*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Urethane</td>
<td>2757.2</td>
<td>103</td>
<td>68</td>
<td>a</td>
<td>a</td>
<td>a</td>
</tr>
<tr>
<td>Epoxy phenolic</td>
<td>1579</td>
<td>10.1</td>
<td>4H</td>
<td>1285</td>
<td>18</td>
<td>0.07</td>
</tr>
<tr>
<td>Polysulfide</td>
<td>4113</td>
<td>10.7</td>
<td>6B</td>
<td>a</td>
<td>a</td>
<td>a</td>
</tr>
<tr>
<td>Urea urethane</td>
<td>10444</td>
<td>98.0</td>
<td>6B</td>
<td>a</td>
<td>a</td>
<td>a</td>
</tr>
<tr>
<td>Vinyl ester</td>
<td>90</td>
<td>0.4</td>
<td>3H</td>
<td>3273</td>
<td>1</td>
<td>4.76</td>
</tr>
<tr>
<td>Epoxy novolac</td>
<td>-4</td>
<td>0.0</td>
<td>8H</td>
<td>4268</td>
<td>1</td>
<td>6.55</td>
</tr>
<tr>
<td>100% solids polyamicid-epoxy</td>
<td>2877</td>
<td>11.6</td>
<td>6H</td>
<td>2187</td>
<td>2</td>
<td>7.89</td>
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<tr>
<td>Amino-amine-cured epoxy</td>
<td>9586</td>
<td>41.4</td>
<td>6B</td>
<td>220</td>
<td>9</td>
<td>0.270</td>
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<tr>
<td>Polysulfide</td>
<td>-803</td>
<td>1.6</td>
<td>a</td>
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<td>a</td>
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<tr>
<td>100% solids epoxy phenolic</td>
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<td>90% solids epoxy</td>
<td>1793</td>
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<td>4H</td>
<td>528</td>
<td>18</td>
<td>0.066</td>
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<td>19328</td>
<td>84</td>
<td>a</td>
<td>223</td>
<td>16</td>
<td>a</td>
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</table>

Avg—Average value based on measurements on 6 specimens
VR—Average volumetric chemical absorption
$S_{UTS}$—Ultimate tensile strength
e—Total elongation
E—Elastic or Young modulus
a—Not reported due to unreliable test data.
*1000 psi=6.895 MPa
atrazine and alachlor, the $S_{UTS}$ ranged from 5,000 to 5,400 psi (35 to 37 MPa), corresponding to increases of 4 to 13 percent. In other test chemicals, the $S_{UTS}$ decreased 1-30 percent. However, in trifluralin, the $S_{UTS}$ measured was over 3,500 psi (24.1 MPa). The elastic modulus of the coating decreased in all test solutions. The greatest reduction (43-50 percent) was observed in pendimethalin. In other test chemicals, the reduction of the elastic modulus ranged from 8-42 percent.

The post-test visual examination of the specimens revealed no signs of chemical attack. A slight discoloration, similar to that found on the epoxy phenolic resin, was observed on the specimens exposed to pendimethalin and trifluralin. However, it was caused by the color of chemical residues retained on the specimen surface, not by any chemical reactions between the test chemical and the coating.

**Epoxy Novolac Resin**

Similar to vinyl ester, epoxy novolac was strong, but inelastic (Table 3). Upon exposure to pesticides, epoxy novolac suffered no obvious effects of chemical attack. The weight gain or loss in each case was less than 2 percent and therefore not significant.

Mechanical evaluation indicated no change in coating hardness in any of the test chemicals, except dimethoate, in which the hardness decreased to 3H. No significant change was observed in total elongation. The $S_{UTS}$ changed slightly in most test chemicals, but still ranged from 3500-4500 psi (24 to 31 MPa). The elastic modulus decreased in most test chemicals, but increased slightly in trifluralin. Decreases ranged from 5 to 43 percent; increases ranged from 8 to 29 percent.

Visual examination showed no evidence of chemical attack or deterioration after the 21-day exposure. A slight discoloration was found only on the specimens exposed to pendimethalin and IMP.

**100 Percent Solids Polyamide Epoxy**

The 100 percent solids polyamide-cured epoxy had a milky color and was strong and brittle (Table 3). After the 21-day exposure, atrazine had little effect on the coating films. Weight gains were 1 percent or less. No discoloration, cracking, or swelling was observed on the test specimens.

Exposure to solvent-borne herbicides produced mixed results. Metolachlor was not corrosive to this coating. The weight gain was only 0.3 percent. The hardness, $S_{UTS}$, and elongation of the coating did not change significantly; however, the elastic modulus (E) decreased slightly. In clomazone, the coating weight increased 4-6 percent, but the increase did not exceed the test criterion of 7 percent. Mechanical tests revealed no significant decrease in the mechanical properties of the coating. In contrast to metolachlor and clomazone, IMP and dimethoate caused excessive weight increases: 11 and 14 percent, respectively. Softening also occurred during the exposure of the coating to these test chemicals. The $S_{UTS}$ and hardness decreased while the elongation increased.

**Amino-Amine-Cured Epoxy**

The mechanical properties of the amino-amine-cured epoxy resin determined before and after exposure are given in Tables 3, 4, and 5. Like the 100 percent solids polyamide-cured epoxy, the amino-amine-cured epoxy deteriorated in the solvent-borne herbicides (clomazone, IMP, and dimethoate) but not in atrazine. The weight gain of the coating films was less than 2 percent in atrazine. Metolachlor also had little effect on the coating; weight gain was less than 1 percent. Clomazone and dimethoate caused excessive weight gains to the amino-amine-cured epoxy coating. Weight gains ranged from 26-44 percent during exposure to these chemicals.

The mechanical properties of the coating did not change significantly after
Testing Coatings for Pesticides

exposure to atrazine and metolachlor. However, the coating did soften when exposed to clomazone and dimethoate. The hardness decreased from HB to 6B, while the tensile strength decreased about 60 percent in clomazone and 90 percent in dimethoate. The total elongation increased after exposure to both chemicals.

**Polysiloxane Epoxy**
The polysiloxane epoxy exhibited excellent corrosion resistance to both water- and solvent-borne herbicides. The coating gained very little weight, less than 2 percent, when exposed to atrazine and IMP. The coating suffered minor weight loss, less than 2 percent, when exposed to clomazone, metolachlor, and dimethoate. Since the coating was very brittle, only hardness tests were performed. No other mechanical tests were conducted on this coating.

**100 Percent Solids Epoxy Phenolic**
In addition to good mechanical properties, the 100 percent solids epoxy phenolic coating exhibited good corrosion resistance. The highest weight gain, approximately 7 percent, was observed in IMP. The weight gain was about 6 percent in both clomazone and dimethoate, but only 1 percent or less is atrazine and metolachlor.

Inspection of the coating's mechanical properties after chemical exposure indicated that the pencil hardness of the coating decreased only 1 to 2 units of hardness in most of the test chemicals. The SUTS increased in most test chemicals, with the exception of IMP. The elongation increased slightly, 2 to 5 percent, in most solutions.

**90 Percent Solids Epoxy**
Before chemical exposure, the SUTS of the 90 percent solids epoxy coating was only 628 psi (4,33 MPa), and the films were very ductile. The coating had a hardness of B and a total elongation of about 12 percent. The coating hardened during chemical exposure. That is, the SUTS and hardness increased, and the total elongation decreased. The SUTS increased by approximately 400 percent, and the hardness increased by 5 units, while the total elongation decreased from 12 percent to 1 or 2 percent after exposure to atrazine, metolachlor, and IMP.

---

**Table 6**

<table>
<thead>
<tr>
<th>Test Chemical</th>
<th>Compressive Strength, psi* Without Freeze-Thaw Testing</th>
<th>Compressive Strength, psi* After Freeze-Thaw Testing</th>
</tr>
</thead>
<tbody>
<tr>
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<tr>
<td>Dimethoate</td>
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<td>3790</td>
</tr>
<tr>
<td>32-0-0 UAN</td>
<td>1763</td>
<td>2148</td>
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</tbody>
</table>

*a—Comparison with average compressive strength of concrete without chemical exposure.

*b—Severe stain
c—Total loss due to chemical attack.

*1000 psi = 6.895 MPa.
During exposure to clomazone and dimethoate, the coating continued to age, but the hardening was offset by softening due to chemical absorption. As a result, the SUTS increased only slightly (from 628 to 777 and 928 psi [4.33, 5.36, and 6.40 MPa], respectively, in clomazone and dimethoate), and the hardness was unchanged. Total elongation changed from 12 percent to 16 percent in clomazone and from 12 percent to 18 percent in dimethoate. The coating failed to meet the test criteria due to excessive weight loss in atrazine and metolachlor. In dimethoate and clomazone, the coating gained weight, approximately 6 and 12 percent, respectively. In IMP, the coating lost less than 4 percent of its initial weight.

**Polysulfide Epoxy**

The polysulfide-epoxy coating had both strength and ductility before chemical exposure (Table 3). However, during the chemical exposure, polysulfide epoxy resin deteriorated substantially. In clomazone and dimethoate, the coating failed completely. Four of the 5 test specimens exposed to each of the chemicals failed due to cracking and softening and could not be retrieved. One test specimen in each chemical remained intact but had a large weight gain, 51 percent in clomazone and 84 percent in dimethoate. The coating weight also increased about 24 percent in IMP.

Atrazine and metolachlor did not harm the coating. Weight loss in atrazine was 0.3 percent. In metolachlor, the coating weight increased less than 2 percent.

**Corrosivity of UAN to Coatings**

Results indicated that UAN had little effect on the coatings. With the exception of the 90 percent solids epoxy resin, the coatings performed very well in UAN. The 90 percent solids epoxy resin had a weight loss of 2.7 percent, failing the weight loss criterion. The other coatings exhibited only minor weight gains (less than 1 percent). The fact that UAN solution has little effect on coatings is important for applications where pesticides and UAN solution are both used. Coating selection can then be based on the pesticides involved.

**Tests on Uncoated Concrete**

Table 6 gives the compressive strengths of uncoated concrete with and without chemical exposure. Compressive test results indicated that the unexposed and uncoated test concrete had an average compressive strength of 3,650 psi (25.2 MPa) after a 28-day cure. These specimens withstood the freeze-thaw cycle tests. No evidence of cracking or spading was observed on any of these specimens. The average compressive strength after freeze-thaw cycle tests was 3,108 psi (21.4 MPa).

Clomazone penetrated the entire specimen. Concrete compressive strength decreased significantly, as much as 37 percent. Average compressive strength of 3 exposed specimens was 2,300 psi (15.9 MPa). Freeze-thaw cycle tests caused no additional damage with compressive strength remaining approximately the same. Discoloration due to precipitation and chemical penetration occurred when uncoated concrete was exposed to IMP.

Similar to clomazone, IMP penetrated the entire thickness of the uncoated concrete specimens. The concrete was also softened in IMP. Compressive strength decreased approximately 4 percent. Freeze-thaw tests resulted in an additional decrease in compressive strength (7 percent). Average compressive strengths of concrete tested in IMP were 1,770 and 2,000 psi (12.2 and 13.8 MPa) with and without freeze-thaw testing, respectively.

Tests indicated dimethoate did not affect uncoated concrete. Compressive strength decreased slightly (1 percent or less). Average compressive strength was 3,610 psi (24.9 MPa). Freeze-thaw tests re-
sulted in additional decreases in concrete compressive strength. However, the average decrease was only 7 percent and the average compressive strength was 3,390 psi (23.4 MPa). Dimethoate was detected in specimen surface defects such as voids or cavities. However, no evidence of chemical penetration, tunneling, or absorption was observed on concrete specimens exposed to dimethoate, although a minor odor was detected after compression tests.

UAN solution was extremely corrosive to uncoated concrete. Compressive strength decreased approximately 37 percent to 1,970 psi (13.6 MPa). UAN completely penetrated the test specimens. Other specimens exposed to freeze-thaw tests failed and could not be retrieved for compressive tests.

Tests on Coated Concrete
Table 7 gives abrasion test results on coatings before and after chemical exposure. Tables 8 and 9 give compressive strengths of coated concrete with and without chemical exposure (dimethoate). Data on IMP, clomazone, and 32-0-0 UAN can be found in the original paper in SSPC 94-19.

Epoxy Phenolic Resin
Before chemical exposure, the epoxy phenolic had a shiny finish and was medium gray. The coating had good abrasion resistance with an average AI of 72 mg/1,000 cycles (Table 7). In clomazone, IMP, and UAN, the coating retained its gloss after 3 months of exposure. No evidence of deterioration, such as delamination, spalling, chalking, or softening, was observed when the coating was exposed to clomazone and UAN solution. Discoloration was observed only on specimens exposed to IMP due to staining by the pesticide. In addition to discoloration, the topcoat delaminated when exposed to IMP, but the primer coat did not. The epoxy phenolic coating provided an effective barrier against clomazone, IMP, and UAN penetration. No evidence of chemical absorption or tunneling was detected. The epoxy phenolic coating was attacked by dimethoate. Softening and etching were the primary causes of deterioration. Although the chemical affected the coating, there was little effect on concrete strength. The decrease in compressive strength, to approximately 3,500 psi (24.1 MPa), was less than 6 percent (Table 8). Odor detection of dimethoate in concrete specimens after compressive tests indicated chemical penetration and tunneling into the concrete. Thus, with exposure to dimethoate, potential contamination is more serious than loss of structural integrity.

Mixed results were obtained from abrasion tests. In IMP and UAN, the abrasion resistance of the epoxy phenolic coating was still excellent with average values of 70 and 78 mg/1000 cycles when exposed to IMP and UAN, respectively (Table 7). However, abrasion testing could not be conducted on coating panels exposed to clomazone and dimethoate, because specimens were soft and swollen.

Epoxy Novolac Resin
Before chemical exposure, the epoxy novolac had a shiny finish and was medium gray in color. This coating exhibited excellent abrasion resistance with an average AI of 45 mg/1000 cycles, the lowest among 4 coatings tested (Table 7).

Discoloration due to pesticide deposits was the main problem caused by exposure to IMP. Otherwise, the coating exhibited excellent performance in all test solutions. The coating retained its gloss after 3 months of exposure. No sign of deterioration, such as delamination, spalling, chalking, discoloration, or softening due to chemical penetration or tunneling was observed. No significant decrease in compressive strength with and without freeze-thaw testing was observed. The average com-
pressive strength of coated concrete ranged from 3,900-3,740 psi (26.9-25.8 MPa). The decrease ranged from 0-6 percent (Tables 8 and 9). Abrasion tests also showed that the chemicals had no effect on the coating. Average AI ranged from 47 to 62 mg/1000 cycles after exposure to the test chemicals (Table 7).

**Vinyl Ester Resin**

Before chemical exposure, the vinyl ester exhibited a dull light gray finish. The AI was 89 mg/1000 cycles, the highest of the 4 coatings tested (Table 7). No sign of chemical attack or deterioration was observed on coated concrete when tested in UAN. However, the coating lost its gloss when exposed to the pesticides. There was evidence of etching and thinning on the topcoat, but no swelling or softening. An abundance of filler crystals was found on the exposed surface during visual examination. Chemical penetration by clomazone and dimethoate was indicated by a strong odor emitted after compressive tests. Compressive test results indicated that most damage was due to chemical attack of the coating. Concrete compressive strength decreased 3 percent or less (Table 8).

No significant loss in abrasion resistance was found when the coating was exposed to clomazone, IMP, or UAN. These specimens have AI values of 102, 91, and 85 mg/1000 cycles, respectively. AI of the coating decreased significantly to 171 mg/1000 cycles when tested in dimethoate. Therefore, this coating may be susceptible to abrasion when exposed to dimethoate and heavy traffic.

**Polysiloxane Resin**

Based on the manufacturer’s recommendation, this coating was used with sand filler to increase solids content and abrasion resistance for concrete floors. The coating was dark gray in color. Like other coatings exposed to IMP, the color turned yellow due to pesticide deposit. Otherwise, the coating exhibited excellent performance in all test chemicals and provided excellent protection. No evidence of chemical penetration or tunneling was observed on the concrete specimens. Decreases in compressive strength were 5 percent or less including those measured after freeze-thaw testing (Table 8).

**Conclusions**

Because urethanes and epoxies can be formulated in a number of ways, these conclusions are not necessarily applicable to other types of epoxies and urethanes. Tests similar to those described here or tests of coatings applied to concrete specimens may be necessary to determine a coating’s suitability for secondary containment ser-
vice. The following conclusions have been drawn from the test results (Table 10).

- Urethane, polysulfide, urea urethane, 100 percent solids polyamide-cured epoxy, amino-amine-cured epoxy, and polysulfide epoxy performed well in atrazine and bentazon but unsatisfactory in solvent-borne liquids. They failed due to extensive expansion, softening, cracking, and dissolution.
- The 100 percent solids epoxy phenolic and 90 percent solids phenolic performed well except for 2 specimens with weight gains in dimethoate slightly above the 7 percent criterion. The 90 percent solids epoxy did not receive a good rating because it had weight losses between 2 and 3 percent in atrazine, metolachlor, and UAN.
- Epoxy phenolic, epoxy novolac, polysiloxane epoxy, and vinyl ester performed well in all test chemicals. The polysiloxane epoxy, however, was very brittle, and its mechanical properties could not be tested. Although the mechanical properties of the other 3 coatings changed, the test specimens were still in good condition overall after 21 days of exposure.
- Water-borne pesticides and UAN had little effect on coatings. Only the 90 percent solids epoxy coating failed due to excessive weight loss. Atrazine and bentazon were the least destructive herbicides. The solvent-borne products were destructive to several coatings. The most destructive pesticides in decreasing order were dimethoate, IMP, and clomazone.
- Test results indicated that thermosetting

### Table 8
Compressive Strength of Coated Concrete with No Exposure

<table>
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<tr>
<th>Test Chemical</th>
<th>Low</th>
<th>High</th>
<th>Average</th>
<th>Low</th>
<th>High</th>
<th>Average</th>
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*1000 psi = 6.895 MPa

### Table 9
Compressive Strength of Coated Concrete Exposed to Dimethoate

<table>
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<tr>
<th>Test Chemical</th>
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<th>High</th>
<th>Average</th>
<th>Reduction %</th>
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<th>High</th>
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*1000 psi = 6.895 MPa.
resins (epoxy and vinyl-ester coatings) provide better corrosion resistance than thermoplastic resins when exposed to solventborne pesticides. These thermosetting resin types provide good resistance to waterborne herbicides.

• Uncoated concrete was rapidly penetrated when exposed to clomazone, IMP, dimethoate, and UAN. Absorption of clomazone, IMP, and UAN caused the concrete to soften. However, absorption of dimethoate did not cause softening. Freeze-thaw cycling caused serious damage to concrete exposed to UAN solution but had no effect on concrete exposed to clomazone, IMP, or dimethoate.

• Without proper barrier protection, concrete is penetrated and attacked by UAN solution. All 4 coatings tested (epoxy phenolic, epoxy novolac, vinyl ester, and polysiloxane) provided excellent protection for concrete exposed to UAN solution.

• When exposed to clomazone, epoxy novolac and polysiloxane provide excellent protection for concrete (resist chemical penetration or softening). Epoxy phenolic and vinyl ester coatings provide limited protection from abrasion.

• When exposed to IMP, all test coatings discolored because of suspension deposits. Epoxy phenolic delaminates because IMP penetrates the topcoat and accumulates at the primer/topcoat interface. Epoxy novolac and other epoxy coatings performed adequately in pesticides tested and in UAN. The 100 percent solids epoxy vinyl ester

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Table 10
Summary of Test Results*  

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<tr>
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<th>Alachlor</th>
<th>Bentazon</th>
<th>Clomazone</th>
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<th>Pendimethalin</th>
<th>IMP</th>
<th>Trifluralin</th>
<th>Dimethoate</th>
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</table>

* The pesticides were tested in the formulations in which they are normally sold.

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* e—excellent: expected to withstand prolonged exposure with no appreciable change in physical properties.
  s—satisfactory: expected to withstand brief exposure. A spill or drip will soften the coating after several days.
  u—unsatisfactory: brief exposure will soften or expand coating enough to significantly degrade its physical properties.
  NT—not tested. In the second series of tests, some chemicals were eliminated to reduce the amount of labor and cost involved.
and polysiloxane coatings demonstrated excellent resistance to the pesticides.
- Organic coatings are only required for protection of concrete from dimethoate when contamination is a consideration. To prevent dimethoate from penetrating the concrete substrate, both epoxy novolac and polysiloxane coatings provide excellent barriers. Epoxy phenolic, however, is not acceptable because of its low abrasion resistance. Vinyl ester is not acceptable because of the possibilities of reducing film thickness due to abrasion.
- The emulsifiable concentrates, which contain organic solvents, were the most destructive to the coatings because of their solvents. Generally the inert ingredients and not the pesticides soften or destroy the coatings. Atrazine and bentazon, the 2 water-borne pesticides, had little effect on the coatings tested.

With laboratory tests completed, field trials of 7 coatings from 4 companies are under way at a retail agrichemical facility in western Tennessee.

References

2. Annual Book of ASTM Standards, Section 6, Volume 06.01; Section 8, Volume 08.01; Section 9, Volume 09.01, Philadelphia, PA: ASTM: 1992.
15. D. Weldon, “Understanding Test Data from Coatings Manufacturers’ Product Data Sheets,” JPCL, May 1993, p. 52