New Specification for Internal Liquid Epoxy Coating Systems For Carbon Steel Pipes and Line Pipes

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Project Background – Internal Pipe Coatings

Problem:
• Shortage of local FBE applicators for Oman
• Projects not being done to schedule
• No documentation for qualification of alternative coatings

Project:
• Prepare specifications for liquid coating
• Market research liquid coatings
• Identify, source and test candidate coatings
Liquid Coatings vs. FBE

**FBE**
- base and converter together
- high application temperatures
- phosphate steel treatment sometimes recommended
- DSC useful as QC tool
- powder application equipment required
- typically very fast cure of coating
- usually very very flexible

**Liquid Coatings**
- base and converter separate
- application temperatures lower than for FBE
- phosphate treatment not required
- DSC not such a QC tool
- variable and different application equipment used
- typically require longer time to cure coating
- often less flexible than FBE
## Operating Conditions

<table>
<thead>
<tr>
<th>Parameter</th>
<th>System 1 (water only)</th>
<th>System 2 (Production fluids containing oil, gas and water)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Design Temp.</td>
<td>85 °C</td>
<td>85 °C</td>
</tr>
<tr>
<td>Design Pressure</td>
<td>Not Applicable</td>
<td>50 bar</td>
</tr>
<tr>
<td>GOR</td>
<td>0</td>
<td>&lt;300</td>
</tr>
<tr>
<td>% Water Cut</td>
<td>Not Applicable</td>
<td>50%</td>
</tr>
<tr>
<td>CO₂ Pp</td>
<td>Not Applicable (0)</td>
<td>3 bar</td>
</tr>
<tr>
<td>H₂S Pp</td>
<td>Not Applicable (0)</td>
<td>0.5 bar</td>
</tr>
<tr>
<td>Solid/Velocity</td>
<td>Less than erosional velocity</td>
<td>Less than erosional velocity</td>
</tr>
<tr>
<td>Chlorides (Cl⁻)</td>
<td>&lt;1000000 ppm</td>
<td>&lt;1000000 ppm</td>
</tr>
</tbody>
</table>
Market Research for Coatings

- 40 coating companies were contacted
Market Research for Coatings – contd.

- Only 8 suppliers indicated that they had appropriate coatings
- 10 coatings were proposed
- Study of product data sheets and test data resulted in elimination of 4 products
- Only 6 products for initial testing!

From…
Specification - objectives

Specification details to cover:

• define responsibilities of end-user, supplier and applicator

• qualification procedures for coating and applicator

• application process details

• QC of materials and equipment

• inspection requirements and procedures

• coating tests for qualification and QC/QA
Qualification of Coatings

- consider factors in service conditions that are most likely to lead to coating failure
- tests to demonstrate performance $\geq$ FBE for service conditions
- develop screening tests to eliminate inappropriate coatings
- market research available liquid coatings
- identify and source candidate coatings
- develop test program for screening and qualification
- conduct testing to qualify coatings
Qualification Testing

- To ensure good performance and ≥ FBE in the service conditions, a broad spectrum of tests were selected.
- Tests include destructive and NDT with the latter particularly suitable for QC on site.
- Tests examine properties related to the application on steel and also coating properties.
Physical Properties Tests

- Film Thickness – how evenly was it applied and how well does it cover the surface?
- Holiday detection – are there holes or cracks in the coating?
- Hardness – how hard is the coating?
- Porosity – what level of foam is present in the coating film near the substrate and in cross section?
- Interface contamination – how clean was the steel surface when the coating was applied?
Physical Performance

- **Impact** – how well does the coating resist damage when impacted?
- **Flexibility** – how well will it cope with bending of the pipeline?
- **Abrasion** – how well does the coating resist film loss when abraded?
- **Penetration** – how much does the film thickness reduce under a heavy localized load?
Physical Characteristics

Adhesion and coating integrity:

• Knife adhesion – leverage at base of coating particularly examines adhesion to substrate.
• Pull-off adhesion – examines adhesive and cohesive properties of the coating; shows any weak regions in the coating film.

*Note that these tests are also used after immersion tests to examine for changes in the coating film as a result of the testing.*
Physical Characteristics

Barrier properties and effects of heat:

• Water vapour transmission – how good is the coating as a barrier to the passage of water?
• DSC – is the Tg above the pipeline rated temperature and does it agree with data sheet?
• Linear thermal expansion – what happens to film dimensions when the temperature changes and how does this compare to steel?
Chemical Resistance Tests

• Hot water resistance (85°C) – how well does the coating retain adhesion when immersed?
• Chemical resistance + autoclave – this test exposes the coating to different chemicals for 90 days and then exposes the coating to pressure and high temperature before examining for blisters, cracks, delamination, or other defects.
Chemical Resistance Tests

- CD (85°C) – how does cathodic protection affect coating adhesion?
- Water absorption (23 and 85°C) – how much water will the coating absorb with long-term immersion?
- Gas pressure variation (10 cycles) – how well does the coating withstand changes in pressure?
- Salt fog – how well does the coating withstand high salt environments and how well does it hinder undercreep?
Screening Tests

• In order to minimize the testing conducted, the candidate coatings were initially only tested using selected tests.
• Based on the results in these screening tests coatings were selected for testing in the remaining qualification tests.
Screening Tests - Adhesion

• All coatings showed very good adhesion to the steel and passed Specification requirements
• Only Coating 2 passed the test, Coating 5 was next best and Coating 4 was the least flexible
Screening - Cathodic Disbondment

- Coatings 1, 2 and 3 had severe chemical attack but Coatings 4 and 5 passed with Coating 5 best.
Screening Tests - Autoclave

- Coatings 1, 2, 4, and 5 had excellent resistance with no blisters, no adhesion loss, & only slight color change.
- Coating 3 blistered in both phases.
Screening Tests - Water Vapor Transmission

Test at 85°C

Coating 1
Coating 2
Coating 3
Coating 4
Coating 5

WVT gm / m² / 24h

Fail
Pass

Coating 2 fails, Coating 3 and Coating 5 pass.
Screening Tests - Water Absorption

Water Vapor Transmission (g/m²)

Coating #

PASS

FAIL

Days

Coatings:
1
2
3
4
5
## Comparison Table

<table>
<thead>
<tr>
<th>Test</th>
<th>Coating 1</th>
<th>Coating 2</th>
<th>Coating 3</th>
<th>Coating 4</th>
<th>Coating 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesion</td>
<td>😊</td>
<td>😊</td>
<td>😊</td>
<td>😊</td>
<td>😊</td>
</tr>
<tr>
<td>Flexibility</td>
<td>😞</td>
<td>😊</td>
<td>😞</td>
<td>😞</td>
<td>😞</td>
</tr>
<tr>
<td>Porosity</td>
<td>😊</td>
<td>😊</td>
<td>😊</td>
<td>😊</td>
<td>😊</td>
</tr>
<tr>
<td>CD Test</td>
<td>😞</td>
<td>😞</td>
<td>😞</td>
<td>😊</td>
<td>😊</td>
</tr>
<tr>
<td>Autoclave</td>
<td>😊👍</td>
<td>😊👍</td>
<td>😞</td>
<td>😊👍</td>
<td>😊👍</td>
</tr>
<tr>
<td>WVT</td>
<td>😊👍</td>
<td>😞</td>
<td>😞</td>
<td>😊👍</td>
<td>😞</td>
</tr>
<tr>
<td>Water Absorption</td>
<td>😊</td>
<td>😊👍</td>
<td>😊👍</td>
<td>😊👍</td>
<td>😊</td>
</tr>
</tbody>
</table>
Screening Test Conclusions

- Although all coatings were epoxy novalac based, there were large differences in performance.
- **Coatings 4 & 5** were selected for further qualification testing.
Coatings 4 & 5 Qualification Testing

**Additional** tests conducted:

- WVT and water absorption extension to 90 days
- Hot water resistance: 85°C for 90 days
- Penetration: 85°C
- Impact resistance: 0 and 20°C
- Abrasion resistance: 23°C
- Chemical resistance: 23°C for 90 days
- Autoclave after chemical resistance
- Gas pressure variation: 23°C at 100 bar, 10 cycles
- Salt fog: 35°C for 1000 hours
- DSC
- Linear Thermal Expansion: -10°C to + 90°C
Following are highlights of performance and differences.

Note the following:

• Hot water resistance: both passed well
• Penetration: both passed well
• Gas pressure variation: both passed well
• Salt fog: both passed well
• Other tests are summarized on following slides…
Water vapour transmission (WVT)

WVT at 23°C met requirement of ≤ 3.0 g/m²/24h

At 85°C neither coating met requirement but test vessel could not handle pressure at elevated temp.

WVT = 0.95 gm/m²/24h at 23°C

Coating 5

WVT = 0.22 gm/m²/24h at 23°C

Coating 4
Water absorption

Coating 5 - 23°C
Coating 5 - 85°C
Coating 4 - 23°C
Coating 4 - 85°C

Fail
Pass
Impact resistance

Impact Strength (J)

- 23C
- 0C

Fail
Pass
Abrasion resistance

Weight loss mg/1000 cycles

Coating 4

Coating 5

Fail

Pass
Chemical Resistance

Samples were exposed to test media for 90 days and then to autoclave testing and rating.

<table>
<thead>
<tr>
<th>Test Media</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCl aq. pH 2.5-3.0</td>
</tr>
<tr>
<td>HF aq. pH 2.5-3.0</td>
</tr>
<tr>
<td>H$_2$SO$_4$ aq. pH 2.0</td>
</tr>
<tr>
<td>NaCl and H$_2$SO$_4$ aq. 100,000 ppm chlorides pH 3.0</td>
</tr>
<tr>
<td>10% NaCl aq.</td>
</tr>
<tr>
<td>Distilled water</td>
</tr>
<tr>
<td>Toluene</td>
</tr>
</tbody>
</table>

**Autoclave conditions:**
- 93ºC, 2000 psi/14MPa
- 50% of sample in 5% NaCl aq.
- Gas: 0.5% H$_2$S, 5% CO$_2$, 94.5% CH$_4$
- 48 Hours
- Rapid decompression at 47ºC (after 4 hours cooling)

Coating 4: no visible degradation to chemicals except surface uniformity in toluene; after autoclave no adhesion loss but severe colour change and slight surface roughness; also in toluene F#4 blisters at gas-water interface - **PASS**

Coating 5: no visible degradation to chemicals or autoclave except severe colour change in latter and slight adhesion loss in gas phase - **PASS**
Tests on the samples as received resulted in Tg values NOT ≥ 10°C above max. operating temp.

BUT additional tests on samples after hot water immersion showed increases in Tg values.

<table>
<thead>
<tr>
<th>Coating 5</th>
<th>Sample ID</th>
<th>Tg (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample before hot water test</td>
<td>#1</td>
<td>78</td>
</tr>
<tr>
<td></td>
<td>#2</td>
<td>78</td>
</tr>
<tr>
<td>Sample after hot water test</td>
<td>S1</td>
<td>119</td>
</tr>
<tr>
<td></td>
<td>S3</td>
<td>117</td>
</tr>
<tr>
<td>Sample stored 90 days at 23°C</td>
<td></td>
<td>100</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Coating 4</th>
<th>Sample ID</th>
<th>Tg (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample (before hot water test)</td>
<td>#1</td>
<td>76</td>
</tr>
<tr>
<td></td>
<td>#2</td>
<td>77</td>
</tr>
<tr>
<td>Sample after hot water test</td>
<td>HWR2</td>
<td>92</td>
</tr>
<tr>
<td></td>
<td>HWR4</td>
<td>93</td>
</tr>
<tr>
<td>Sample stored 90 days at 23°C</td>
<td></td>
<td>79</td>
</tr>
</tbody>
</table>
Coefficient of linear thermal expansion (CLTE)

Average Coefficient of Linear Thermal Expansion

Av. CLTE -10 to 20°C
Av. CLTE 20 to 90°C

Steel | Coating 4 | Coating 5

Graph showing the average coefficient of linear thermal expansion (µm/m.°C) for steel and different coatings.
Coatings 4 & 5 Qualification Tests

- Both good BUT did not meet all criteria, **differences**!
- Both had good hardness, adhesion, and resistance to impact, penetration, chemicals, hot water, salt fog, autoclave and gas pressure variation.
- Lower than required Tg but this increased after 85°C immersion, especially for Coating 4.
- Lower than required abrasion resistance and flexibility.
- Coating 5 more flexible than Coating 4.
- Coating 4 better WVT than Coating 5.
- Coating 5 passed water absorption test but Coating 4 showed increasing absorption at 85°C up to full 90 days of test.
- Coating 4 had lower CLTE (better = closer to that of steel)
- Coating 4 showed some chemical attack in CD test.
Conclusions

Both Coatings 4 and 5 showed good performance in the qualification tests but did not meet all the specification criteria.

Ultimately based on performance in all the tests it was decided to include Coating 5 in field trials that are now starting.