Abstract

The outer shell of a Multi-layered Urea reactor ruptured while the plant was in operation. The plant made an emergency shutdown, samples were collected, and an investigation carried out to establish the root cause of the failure. The paper explains the detailed analysis performed, corrective action implemented, and a health assessment for the equipment's safe working. The paper also includes references to similar failures in old multi-layered urea reactors subsequently and the mitigation plans to avoid such failures.

Keywords: Case Study, Failure Analysis, Field Repair, Multi-layered Urea Reactor.

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Introduction

The urea reactor is a critical component in any fertilizer plant. Urea is formed in the urea reactor by the ammonia and carbon dioxide reaction at elevated pressure and temperature. During normal operation of the urea plant, a sudden sound and a rupture were found in the outer shell layer of the urea reactor. Figure 1 shows the rupture of the outer shell, with a tearing of 1.5 m (4.9 ft) length. This multi-layer Urea reactor was supplied in 1982 and has been in operation for more than 35 years, with regular maintenance work carried out every 2 to 3 years, which included inner liner inspections and repairs.

This paper describes the investigation regarding physical and metallurgical findings for the multi-layered shell, detailed analysis, root cause of the failure, lessons learned, and the health assessment results. Furthermore, the paper covers an innovative approach implemented to perform an in-situ replacement of the defective liner segment during the emergency shutdown. Some modifications and the preventive actions implemented to avoid reoccurrence will also be discussed.

General Technical Data

Urea reactors are usually vertical pressure vessels, with either mono-wall, multi-layer, or multi-wall construction. Because of the corrosive nature of the medium, the interior surface of the vessel is lined with corrosion-resistant materials.

The design and construction features of the subject Urea reactor are shown in Table 1 and a diagram of features in a multi-layer urea reactor is shown in Figure 2.

Initial Inspection at Site

Visual inspection of the Urea reactor showed tearing of the outer shell layer resulting in a 1.5 m (4.9 ft) wide opening near the davit arm location, as shown in Figure 3a. Deflection of the davit arm and bulging near the ruptured zone were observed.

Heavy scale formation was observed on the outer shell as shown in Figure 3b. Thickness of the shell layer was reduced (~13.5 mm (0.53 in) against original 14.9 mm (0.59 in)), due to corrosion under insulation.

The fracture surface is shown in Figure 4, which shows pre-existing crack face and fresh rupture face. This pre-existing crack seems to have initiated near the edge of fillet weld between the manway davit arm pad plate and the outer shell layer.

The reduction in thickness of the outer shell layer near the ruptured zone was found to be ~2-3 mm (0.08–0.12 in).
Table 1: Design & Construction details

<table>
<thead>
<tr>
<th>No.</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Construction</td>
<td>Multi-layered (7 layers)</td>
</tr>
<tr>
<td>2.</td>
<td>Total shell thickness</td>
<td>115.8 Mm</td>
</tr>
<tr>
<td>3.</td>
<td>Inner liner</td>
<td>1 X 7 mm Sa240 tp</td>
</tr>
<tr>
<td></td>
<td>Layers</td>
<td>thickness thickness</td>
</tr>
<tr>
<td></td>
<td>Dummy layer</td>
<td>1 X 4.5 mm Jis ss41</td>
</tr>
<tr>
<td></td>
<td>Outer layers</td>
<td>7 X 15 mm Sa 724 gr a</td>
</tr>
<tr>
<td>5.</td>
<td>Design pressure</td>
<td>169 Kgf/cm2(g)</td>
</tr>
<tr>
<td>6.</td>
<td>Design temperature</td>
<td>200°C</td>
</tr>
<tr>
<td>7.</td>
<td>Operating pressure</td>
<td>159 Kgf/cm2(g)</td>
</tr>
<tr>
<td>8.</td>
<td>Operating temperature</td>
<td>170°C (top)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>190°C (bottom)</td>
</tr>
</tbody>
</table>

No traces of carbamate/residue were observed in the ruptured portion of the shell/immediate inner shell surface in the vicinity of failure.

An air test was performed to find the leakage in the liner at the top portion of reactor (Top head + Shell). The test was not successful due to weep holes blockage. Further steam purging was carried out to clear the weep holes. Deposits were observed at the weep hole opening and collected for analysis (Figure 5).

Visual inspection and dye penetrant test (DPT) of the inside liner surface showed signs of corrosion on the longitudinal and circumferential weld joints, as shown in Figure 6. Corrosion was also observed on the weld built-up area buttering (old cleat removal area). Localized dents and bulges on liner surface were also observed.

After cutting and removal of the outer shell layer, the inside surface of the layer revealed discoloration towards weep hole-W9, as shown in Figure 7.

Air testing was conducted after the blockage in weep holes was removed. This revealed leakage in the liner long seam, in the area between the 1st and 2nd trays, as shown in Figure 8a. The leakage location was then verified by dye penetrant test (Figure 8b and 8c).

After cutting the liner plate at the leakage location, the inner dummy shell and 1st layer shell were corroded through as shown in Figure 9.

Investigation

To find out the cause of failure, a detailed investigation was carried out on extracted samples. The following tests were conducted:
Chemical Analysis

Chemical analysis by Optical emission spectroscopy was carried out to confirm the chemical composition of the shell layer. The results confirmed a match to the expected composition. These results are shown in Table 2.

Mechanical Testing

Tensile, impact, and hardness testing were carried out in the longitudinal and transverse directions. Tensile properties met the requirements of the material specification, as shown in Table 3.

Visual and Macro Examination

Samples collected from the failure location were thoroughly examined visually and using a stereoscope. The following observations were made:

The outside surface of the shell layer showed the presence of heavy scaling, as shown in Figure 10.

The inside surface shell layer revealed discoloration as shown in Figure 11. The residue was analyzed to be iron and oxygen.

A pre-existing crack (~3 mm (0.12 in) depth) was observed on the fracture face, and shown in Figures 12 and 13. This pre-existing crack appears to have initiated near the edge of the fillet weld between the manway davit arm bracket pad plate and the outer shell layer, as shown in Figure 14.

![Figure 7: Inside surface of outer shell layer](image)

![Figure 8: Leak found in air test and verified in DPT](image)

![Figure 9: Corrosion of dummy shell and 1st shell layer due to liner leakage](image)

### Table 2: Chemical composition of samples

<table>
<thead>
<tr>
<th>Element (%)</th>
<th>Result</th>
<th>Requirement of SA 724 Gr-A</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.16</td>
<td>0.22 max</td>
</tr>
<tr>
<td>Si</td>
<td>0.39</td>
<td>0.60 max</td>
</tr>
<tr>
<td>Mn</td>
<td>1.38</td>
<td>0.92–1.72</td>
</tr>
<tr>
<td>P</td>
<td>0.013</td>
<td>0.025 max</td>
</tr>
<tr>
<td>S</td>
<td>0.006</td>
<td>0.025 max</td>
</tr>
<tr>
<td>Cr</td>
<td>0.025</td>
<td>0.29 max</td>
</tr>
<tr>
<td>Ni</td>
<td>0.003</td>
<td>0.28 max</td>
</tr>
<tr>
<td>Mo</td>
<td>0.002</td>
<td>0.090 max</td>
</tr>
<tr>
<td>Cu</td>
<td>0.007</td>
<td>0.38 max</td>
</tr>
<tr>
<td>V</td>
<td>0.043</td>
<td>0.090 max</td>
</tr>
</tbody>
</table>

### Table 3: Mechanical properties

<table>
<thead>
<tr>
<th>Mechanical properties</th>
<th>Longitudinal direction</th>
<th>Transverse direction</th>
<th>Sa 724 Gr A*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield strength (mpa)</td>
<td>591</td>
<td>506</td>
<td>485 Min</td>
</tr>
<tr>
<td>U.T.S. (Mpa)</td>
<td>758</td>
<td>723</td>
<td>620-760</td>
</tr>
<tr>
<td>% Elongation</td>
<td>22.76</td>
<td>25.72</td>
<td>19 Min</td>
</tr>
<tr>
<td>Impact at 0°C (joules)</td>
<td>208,232,220</td>
<td>114.94.100</td>
<td>....</td>
</tr>
<tr>
<td>Hardness in (hbw)</td>
<td>215.219</td>
<td>219</td>
<td>****</td>
</tr>
</tbody>
</table>
Additionally, a small crack was observed near the fracture surface, which appeared to be generated due to mechanical force during fracture of shell layer (Figure 15).

After visual and macro examination of the extracted samples, specimens containing the failure locations were cut for subsequent analysis. Specimen containing the fillet weld (Pad plate to outer shell layer weld) showed a lack of fusion with shell layer (Figure 16).

**SEM & EDS Analysis**

SEM-EDS analysis was carried out on the specimens cut from extracted samples, scale formed on the outside surface of shell layer under insulation, and deposits collected from the weep hole.

SEM of shell layer side part of the fracture surface clearly revealed a boundary between pre-existing crack and fresh rupture, as shown in Figure 17. The maximum depth of the crack was found to be ~3.7 mm (0.15 in).

A specimen containing the pad side part of the fracture surface that showed lack of fusion in the weld revealed a micro crack in the base metal adjacent to weld edge, as indicated in Figure 18. The edge of the weld was revealed,
Failure Analysis and Field Repair of Urea Reactor

Figure 17: SEM Image for boundary between previously induced crack and fresh rupture

Figure 18: SEM Image for crack on fracture face near weld

Figure 19: EDS on previously induced crack face which appeared to be the initiation point of the pre-existing crack.

EDS revealed a significant amount of oxygen, iron, and other elements like sodium, calcium, and manganese on the pre-existing crack face and at the crack beside the fracture surface (Figures 19 and 20).

A significant presence of oxygen and iron, along with elements like sodium, manganese, calcium, and chlorine was observed on the crack face near the outside surface of shell layer. (Figure 21) A lower amount of oxygen was noticed on the rupture face as compared to the pre-existing crack face. This indicated low degree of oxidation on fresh rupture face.

Scale formed on the outer layer of the shell consisted mainly of iron and oxygen. (Figure 22)

The deposit collected from the weep hole showed significant presence of nitrogen, oxygen, and carbon (Figure 23).

Microscopic Examination

After SEM-EDS analysis; the cross-section surface of the specimen was polished and etched with 2% Nital and observed under a microscope. The following observations were made:

Microstructure of shell layer showed tempered martensitic structure (Figure 24).

The pre-existing crack appears to be initiated from edge of the fillet weld and propagated in heat affected zone (HAZ) in the outer shell layer. (Figure 25)

Secondary cracks beside the fracture surface were also observed (Figure 26)
Weldability Test

Weldability of the shell layer material was verified by welding butt joint and clearing the dye penetrant test and radiography test.

**Summary of Observations**

- The chemical composition and mechanical properties of outer shell layer conforms to SA 724 Gr A, as per drawing requirement. Microstructure of the material was found satisfactory. Weldability test carried out using shell layer material was also satisfactory. Based on this, it inferred that material had not degraded during service.
- During a visual inspection at site, heavy scaling due to corrosion was observed on the outside surface of the outer shell layer. Thickness of the shell was found to be reduced by 1.5 mm (0.06 in) due to scaling. EDS of the scale formed on the outer layer under insulation was found to be mainly oxygen and iron. This indicated moisture and general corrosion under insulation on outer shell layer over the time.
- The lack of fusion in the davit pad to outer shell layer fillet weld was observed in visual examination and SEM of the specimen containing the fracture surface. The presence of such a discontinuity at the edge of the weld increases the possibility of crack initiation and propagation over a period of time.
- Visual, macro-examination and scanning electron microscopy of extracted samples showed presence of a pre-existing crack (~3 mm (0.12 in) depth) on the fracture surface, which is clearly differentiated from the fresh rupture face. It appeared to be initiated at the edge of the weld between the pad-plate and shell layer. This was also verified by micro-examination, which clearly showed crack initiation at the edge of the fillet weld and propagation through the outer shell layer.
- The inside surface of the reactor showed signs of corrosion on the liner long seam, circumferential joints, and buttering welds (old cleat removal area). During the air test, weep holes were blocked due to solidifying carbamate in the leak path. The air test performed after the weep holes cleared leakage in the liner long seam. A corrosion attack was observed on the dummy shell layer and 1st shell layer in the vicinity of the liner leakage.
- EDS testing of the deposit collected from the weep hole showed significant nitrogen, oxygen, and carbon presence. This showed carbamate leaked through liner was solidified and deposited at the weep holes. The inside surface of the extracted outer shell layer revealed discoloration towards weep hole-W9 side, which was likely the path of flowing media generated due to weep hole blockages.
- EDS analysis showed a significant amount of oxygen, iron, and other elements like sodium, calcium, magnesium, aluminium, and silicon on the pre-existing crack face and an additional crack beside the fracture surface. The presence of oxygen and iron indicates the formation of iron oxide on the crack face. Other elements appeared to be contributed by the insulation material and water coming from an external source.

**Observations from recent repairs in Fertilizer plants**

Besides this shell rupture, there have been similar instances of urea reactors leaking through liners and ending up in
long shutdowns from 35-45 days globally [1-6]. Applying innovations, we have designed and developed a special dedicated machine used for in-situ rolling of liners inside vessel, which is patented and used for reducing shutdown durations. Typical causes of these failures and remedies to prevent them are summarized below:

- Vent holes are plugged from outside as these are supposed to be removed post-erection. There is no path for the pressure to be released in case pressure is built-up in the shell inter-layers.
- Vent tubes are clogged / blocked, and the leak detection system does not work, increasing the risk of pressure boundary failure by corrosion / rupture in case the liner leaks.
- Random dye penetrant testing of the liner welds, compared to 100% examination, likely results in defects being missed.
- Plant owners concentrate on the liners from the inside and seldom check the pressure boundary from outside. Random non-destructive testing of pressure shell welds should be institutionalized in every shutdown.
- Attachments for liners are welded with partial penetrations. This increases the risk of corrosive medium attacking the crevices and resulting in liner failures.

**Conclusion**

Based on the observations and analysis, the following conclusions can be drawn for the shell rupture:

- Material of the outer shell layer was not degraded. However, thickness of the outer shell layer was reduced by 1.5 mm (0.06 in), possibly due to corrosion under the insulation of the outer shell layer over time.
- Visual examination revealed the presence of a pre-existing crack, initiated near the edge of the fillet weld between the manway davit arm pad plate and the outer shell layer. This could be due to the utilization of the davit arm beyond its design load for various handling activities during earlier shutdowns. Furthermore, discontinuity in the form of lack of fusion increased the possibility of crack initiation and propagation over a period.
- Leakage in liner and blockages of weep tubes during operation resulted in process media entering the shell inter-layers, which in turn exerted pressure on the outer layer. Thickness reduction due to corrosion under insulation and localized reduction in thickness due to pre-existing crack made the outer layer unable to withstand the operating pressure. This led to the bulging of outer shell layer and subsequent shell rupture.

**References**