TROUBLESHOOTING GUIDE 3

Short service runs in a chloralkali brine softening system

A recently installed brine softening system utilizing Purolite S930Plus Chelation Resin experienced significantly reduced service runs and pressure drop across the lead ion exchange bed. Suspecting the resin was either breaking down or fouling, the customer contacted Purolite to investigate.

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Chloralkali brine softening issue – Part A

Query/complaint/issue

A major chloralkali plant located in the United States advised that the Purolite S930Plus Chelation Resin recently installed in their brine polishing system gave considerably shorter service run lengths than expected.

The issue with the KCl softening system began at startup and was on-going at the time of Purolite's investigation. The system showed high pressure drop across the lead ion exchange bed, which lead the customer to believe the resin was either breaking down or fouling.

The lead bed was then taken out of service when the pressure drop across the unit exceeded 26 psig (179 kPa). Run time at start-up was 6 hours compared to the expected 120 hours.

Initial data provided by customer

The resin was part of a new brine softening system installed at the customer's facility, which recently replaced an older mercury cell process with newer membrane technology. The new system required tighter controls on brine purity levels to protect the new membranes, as well as significantly lower maximum limits for hardness, sulfate, and aluminum.

The facility has two brine softening systems, a KCl polishing system and a NaCl polishing system.

The KCl polishing system consists of three vessels operating in series. The lead bed moves to last position when regenerated. The other beds moves forward in service. Each vessel is 10 ft (3 m) in diameter with 386 ft^3 (10.9 m^3) of Purolite S930Plus installed. Service flow was designed at 900 gpm (204 m^3/h), but was operating at 600 gpm (136 m^3/h) since startup.

Troubleshooter's response

The resin was not considered as a likely source for the problem as identical resin was installed in the nearby NaCl system within the same timeframe with no reported problems.

The regeneration process was thoroughly reviewed and tested prior to startup of both systems. To determine the root cause of the problem, the following steps were taken:

1. Obtain representative resin samples when the resin bed was taken offline; send to Purolite for analysis
2. Initiate regeneration and collect backwash samples; determine if resin fines were washing out
3. Observe the entire pretreatment system; learn about system

In the initial investigation, pretreatment of the KCl system, occurring before ion exchange polishing, consisted of chemical feed / clarification to reduce hardness and suspended solids, followed by a leaf filter to catch residual suspended solids (TSS).

The leaf filter consisted of three units each with 60 leaf filters. These are flat plates with fiber spun mesh and nominal opening of 5μm. The plates were pre-coated with cellulose filter media intended to improve filtration and protect the filter mesh.

The following observations were made during pretreatment observation:

1. Pretreatment consisted of a pH adjustment step to 10.0 using potassium carbonate and potassium hydroxide. At 10.0, total hardness (i.e. calcium and magnesium) is reduced to 3 ppm as $CaCO_3$.
2. KCl brine was low in impurities, so $CaCl_2$ was fed to increase solids. Additionally, levels of Al present needed to be controlled with an MgO feed. Because of the MgO, $CaCl_2$, feed was

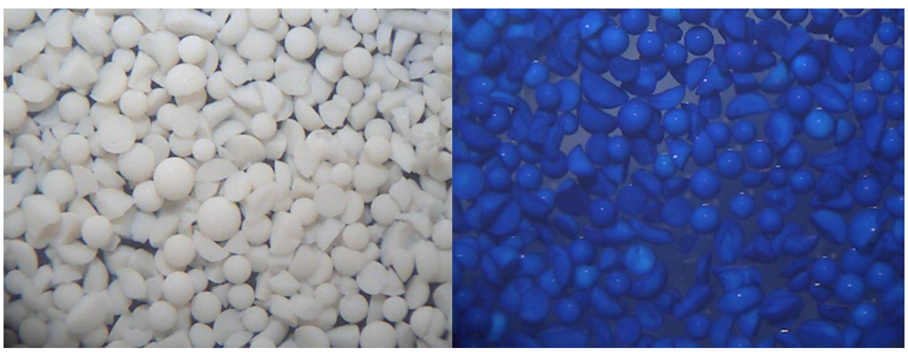
increased along with KOH to maintain alkalinity below 0.2 g/l excess. Additionally, there was a limitation in pump rate for chemicals and tubing size that affected chemical flow. Moreover, if pH levels increased, excess MgO would form very fine flocculant that would be difficult to remove.

3. Stabilization of the chemical dosing improved run time to 20 hours yet remained far short of expectations.

Resin analysis showed:

- 60% of the beads were broken
- Loading was only 6% of the capacity
- There was no surface impairment on the bead that would cause fouling of the resin as revealed through a dye test performed to determine fouling (Figure 1)

Figure 1 - Photo of Purolite S930Plus from the KCl softening bed



The dyed sample showed no fouling on the beads.

The percentage of broken beads was a concern, but the low loading was not a concern since the beds were taken out of service due to high pressure drop.

Further investigation revealed that resin was not a representative core sample as we had expected—but merely taken off the top of the bed. The excessive bead breakage shown was to be expected from the high pressure drop across the bed. To complicate matters further, we found that the backwash flow rate was insufficient to remove most of the fines, so operators needed to enter the vessel and remove a top portion of resin.

Chloralkali brine softening issue – Part B

Diagnosis/answers/suggestions

The current backwash rate was set to increase or decrease as water temperature changes. The flow varied slightly, but was approximately 390 gpm (88m^3/h) at 5gpm/ft^2 (12.5 m/h). Duration was set to 15 minutes, which was deemed sufficient to de-compact the bed.

Backwash samples were collected every 5 minutes for 15 minutes to see if broken beads were being removed. Results showed no broken beads in the samples, but a significant amount of suspended solids were detected.

Collected samples were filtered and the filter pads dried and weighed. The current backwash was determined to be insufficient, and that 35 - 40 minutes of backwash was required to remove the majority of solids from the bed.

Table 1 – Suspended solids removal from lead bed during backwash

Time (Minutes)	Total Gallons @ 390 gpm (88 m^3/h)	BVs	TSS (mg / l)
5	1,950	0.7	0.0
10	3,900	1.3	0.02
15	5,850	2.0	54.8

The Millipore filter for the samples was observed under a compound microscope. A noticeable amount of white fibrous material, a small amount of resin fines, and an under layer of dark brown sediment was present.

This observation supported the theory that the cellulose fiber and suspended solids were passing through the leaf filters and collecting in the resin bed. A second set of samples were taken from the backwash at 5 minute intervals for 40 minutes. A visual inspection indicated that the backwash needed to increase to 35 - 40 minutes to properly remove cellulose and silt. Residual silt remaining after the backwash would likely be removed with the subsequent HCl regeneration step that would dissolve calcium and magnesium solids.

The investigation was done using a polymer from Hercules to improve sludge blanket formation in the KCl clarifier as well as performance of the leaf filters. We recommended the backwash increase until the cellulose was sufficiently removed from the resin and run times improved. We also recommended an additional sample of backwash water be taken after all vessels were thoroughly backwashed to confirm cellulose was not passing through the leaf filters. If cellulose carry-through continued to be an issue, we recommended that that the leaf filters be inspected and repaired as necessary.

After the KCl system run time returned to target level, we recommended that the KCl and NaCl polishers be evaluated for hardness loading and operating efficiency.

Final outcome

The initial 15-minute backwash time was insufficient to remove accumulated solids in each operating cycle. Extending the backwash time solved the problem and the KCl brine softening system now operates efficiently with run times meeting and exceeding expectations. Our investigation showed that Purolite S930Plus Chelation Resin removed impurities so efficiently, it was difficult to establish and maintain an adequate height for the clarifier sludge blanket. Without this trapping mechanism, a greater amount of solids were carried over to the leaf filter, causing significant pressure drop across the filter. In turn, the pressure drop caused the filter membrane to fail, allowing suspended solids and flocculant to pass into the resin bed. The customer agreed to repair the leaf filters and to review options to improving pretreatment and clarifier operation.

Lessons learned

When investigating a problem, think beyond the basic data that a customer may provide. Upsets in operation of the pretreatment system to the ion exchange plant can be the prime cause of performance deterioration within the ion exchange plant. As found with this case, the use of relatively clean brine, ironically, had its own inherent issues. Lastly, the first point of investigation may mask the true issue.

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