

**A Holistic Approach to Iron Transport using FFA and its
Comparison to Polymeric Iron Transport**

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Abstract

A well-planned boiler system will use high-purity feedwater and economizers, and can return a high percentage of condensate to reduce energy, water, and wastewater expenses. In these systems, feedwater hardness concentration is significantly decreased while iron concentration increases. The increase in iron comes from two sources: the returning condensate and the economizer. A well-managed condensate system reduces air in-leakage and process leaks and can maintain pH >8.3. For these systems, the iron in the returning condensate is frequently <10 ppb. However, in facilities with large condensate systems, it can be difficult to maintain these conditions. Condensate polishers can be installed to help reduce the contamination entering the feedwater; however, condensate polishers are frequently left out of the design or not used when present. Economizers in the feedwater systems are also subject to more corrosive environments, as the increased boiler feedwater purity can lead to flow-accelerated corrosion (FAC). The iron entering the boiler from the condensate system and economizer will frequently deposit on evaporator tubes in a watertube boiler or tubes in a firetube boiler because of the high rate of heat transfer. The U.S. Department of Energy reports that heat transfer efficiency loss from 1/32 inches of iron scale will cause 3.1% fuel loss or 7.0% fuel loss if silica is mixed into the iron scale. In addition to the loss in efficiency, tube scaling can increase temperature, causing the tube to overheat and lead to tube failure. The scale can also lead to underdeposit corrosion. Both conditions can result in unplanned outages and sudden production capacity loss. To avoid this, dispersion polymer application is frequently recommended to reduce iron deposition. Recent experience has shown the addition of a film-forming amine to the polymer program significantly lowers feedwater iron and leads to lower deposition.

Introduction

Water reuse, energy savings, and capital investment are major considerations for facilities looking to optimize existing boiler systems or design new boiler systems. Reducing water use, pretreatment, energy loss, and wastewater costs often leads to high rates of condensate return. The iron from an improperly protected condensate system is blended with makeup water and enters the boiler, where it deposits on boiler tubes, developing an insulating scale that reduces boiler efficiency, increases boiler tube surface temperatures, and creates an environment suitable for underdeposit corrosion. High condensate returns can also increase boiler water impurity as well as the potential for flow-accelerated corrosion (FAC). An increased focus on iron residuals in boiler systems can help address these issues.

In boilers operating at <900 psi, sulfonated copolymers are frequently used to transport iron through boilers. In some chemical process industries, special polymers are used in boilers operating up to 1,800 psi. These specialized polymers can achieve transport at a small concentration and are fed such that more than 90% of the polymer breaks down, but the remaining intact polymer is able to transport the iron and limit iron deposition. At higher pressures, more emphasis is placed on limiting corrosion, dumping condensate, and adjusting blowdown to reduce deposition.

In the past few decades, employing film-forming amines (FFA) to reduce overall boiler system corrosion has become more common. FFA is able to reduce corrosion because it bonds to the iron within iron or copper oxide layers and develops a passive film. Modern amine used for filming is volatile, so it is frequently fed to deaerator storage tanks or feedwater systems to form a passive film in the feedwater, boiler, and condensate systems, helping control corrosion in all three areas. In addition to forming a passive film that limits corrosion, FFA also transports iron, reducing reliance on polymers and helping improve overall system reliability.

Film-Forming Amine Applications

Ethanol Plant

Ethanol plants frequently produce food-grade ethanol and have several portions of their system run periodically during the grinding, fermentation, and distillation processes, among others necessary for ethanol production.

One ethanol plant has a low-pressure boiler system that operates at 125 psi. When a portion of the system does not run, the water in the system becomes more corrosive as the pH decreases and dissolved oxygen increases. The treatment program should maintain ASTM limits of ≤ 0.10 ppm iron, ≤ 0.05 ppm copper, and < 0.5 ppm hardness to ensure the boiler operates both efficiently and consistently.

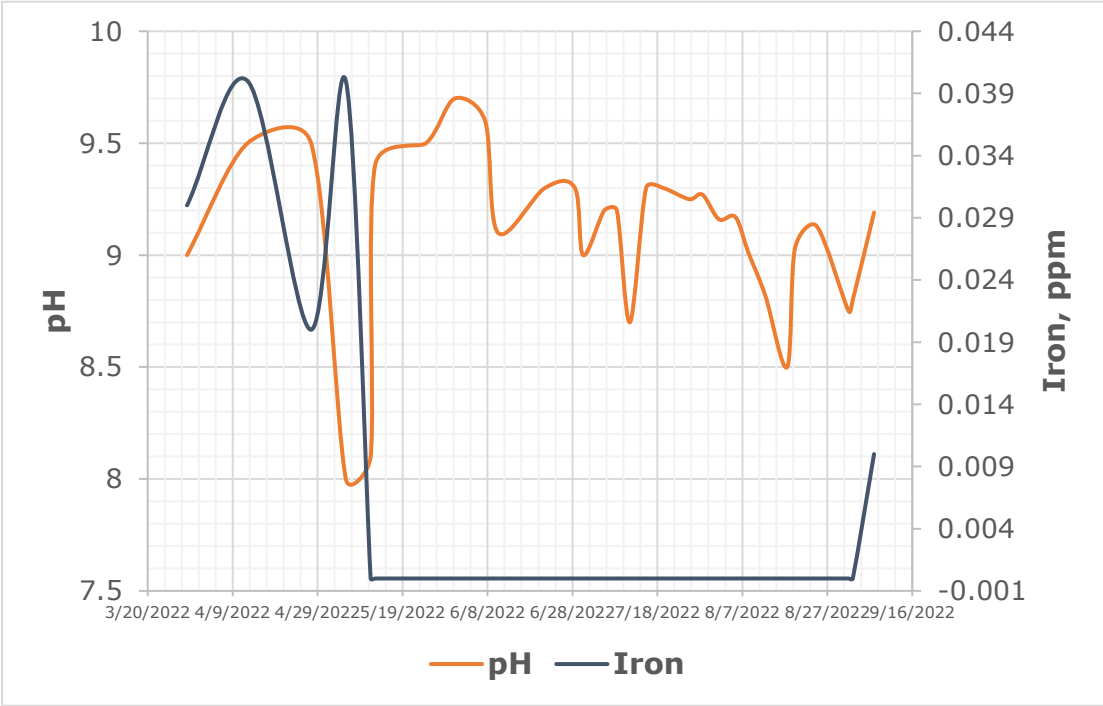


Figure 1. Ethanol plant condensate pH and iron. FFA treatment started March 30, 2022.

One month after introducing FFA to the standard polymer and neutralizing amine treatment program, the iron in the returning condensate decreased from 30 ppb to < 2 ppb Fe, with the pH fluctuating between 8.0 and 9.7. Since the plant has 80% condensate return, the decrease in condensate iron significantly decreased feedwater iron.

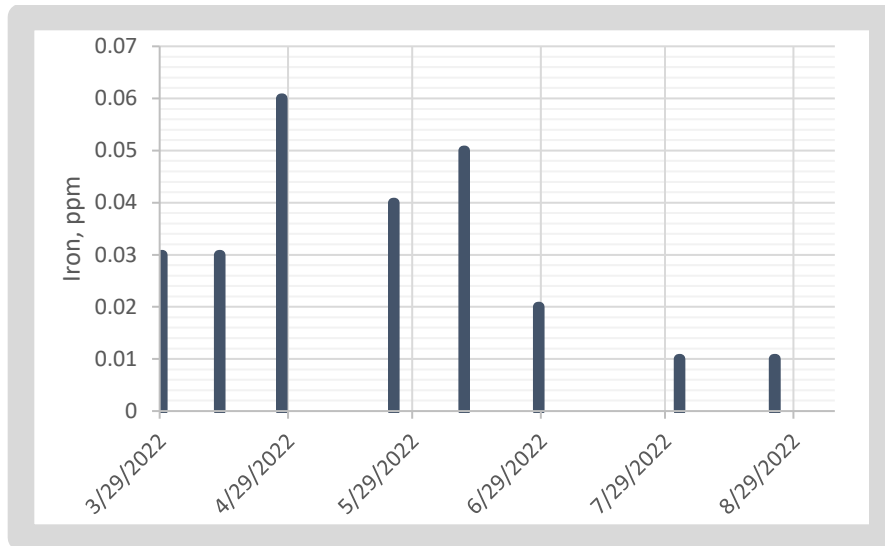


Figure 2. Ethanol plant boiler water laboratory total iron tests

Laboratory tests of the boiler water show a decrease from 30–60 ppb Fe to 10 ppb Fe in the five months of treatment, as illustrated in Figure 1. The relatively quick reduction in condensate iron indicates the active corrosion in the condensate system was low enough for the FFA to remove the loose iron and develop a passive film within one month. However, the more delayed response in the boiler indicates that the surfaces within the economizer had more active corrosion, increasing the time needed to develop a passive film. Since this high-purity feedwater had an average pH of 8.9, the economizer was likely experiencing flow-accelerated corrosion (FAC) conditions.^{3,4}

FAC is a condition where the higher solubility of magnetite at lower temperatures and below pH 9.4 make it difficult for the economizer tube to maintain an adequate passive coating.^{1,3,4} This is especially problematic around bends and just after orifice plates.³ The increase in boiler pH at this plant makes it unlikely that the FAC was occurring in the boiler. The boiler pH was operating at approximately pH 10.5. The solubility of magnetite at that pH is very low, so the iron in the boiler likely came from the economizer, not the boiler itself.

The FFA needed to penetrate through the colloidal iron, remove loose iron from the surface, and develop a film dense enough to reduce FAC.⁴ Figure 2 illustrates how the initial 30 ppb iron at product introduction increased to between 40 and 60 ppb iron for three months before settling down to 10 ppb iron for two months prior to outage. The 40–60 ppb iron period was the stage at which the surface was being prepared and the film developed.



Figures 3 & 4. Before treatment (left) and five months after treatment (right). The inspection of the boiler internals confirmed treatment efficacy after five months of application.

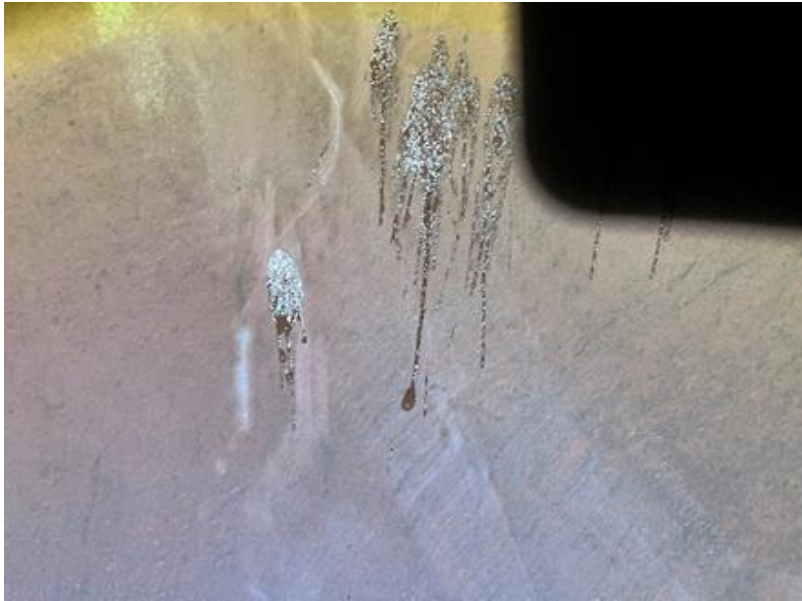


Figure 5. Hydrophobicity in the steam drum indicates FFA passivation.

After five months of treatment, a boiler inspection showed significant improvement in iron scale; the color of the iron oxide shows a good blend of hematite and magnetite and water beading on the surface. Water beading indicates the steam drum has a passive FFA layer on the inside to inhibit corrosion and help protect the drum from corrosion during layup.

Power Plant

FFA was successfully applied at a power plant with >95% condensate return and high-quality makeup water that was using ammonia-only pH control programs. Ammonia-only pH control reduces the byproducts of organic amine degradation and the possibility of salt carryover to the steam, so it is frequently used in power plants. However, this treatment approach leaves the system susceptible to both single- and two-phase FAC, as the volatility of ammonia at lower pressures means it will not be present in early condensate or in the low-pressure (LP) circuit in high enough concentrations to elevate the pH enough to reduce the corrosivity of the condensing water.

This power plant was experiencing 500–1,000 ppb iron in the low-pressure drum (LPD) prior to FFA treatment. While the iron manifested in the LPD, it actually came from the feedwater/condensate system as a result of FAC. Combined cycle units can be prone to FAC because of their design, specifically the high angle elbows throughout the system. Considering the amount of iron present and the small area contributing the iron, wall thinning is a concern. The intermediate-pressure (IP)/high-pressure (HP) economizers are also a concern, because their operating temperature is low enough to potentially cause FAC. EPRI recommends <5.0 ppb iron in all combined cycle drums to reduce the potential for iron deposition on the evaporator tubes.

This power plant operates two combined cycle units, three drums each operating at 64 psi (LP), 479 psi (IP), and 2,390 psi (HP) with a net steam generation of 70, 77, and 675 thousand pounds per hour (kpph), respectively. The feedwater pH is maintained at 9.8–10, and dissolve oxygen is maintained at 20–30 ppb. Each combined cycle unit has its own turbine (1 x 1 configuration) and is configured to have the condensate/feedwater to feed the LP, IP, and HP separately. This is frequently referred to as a standalone LP configuration (SALP).

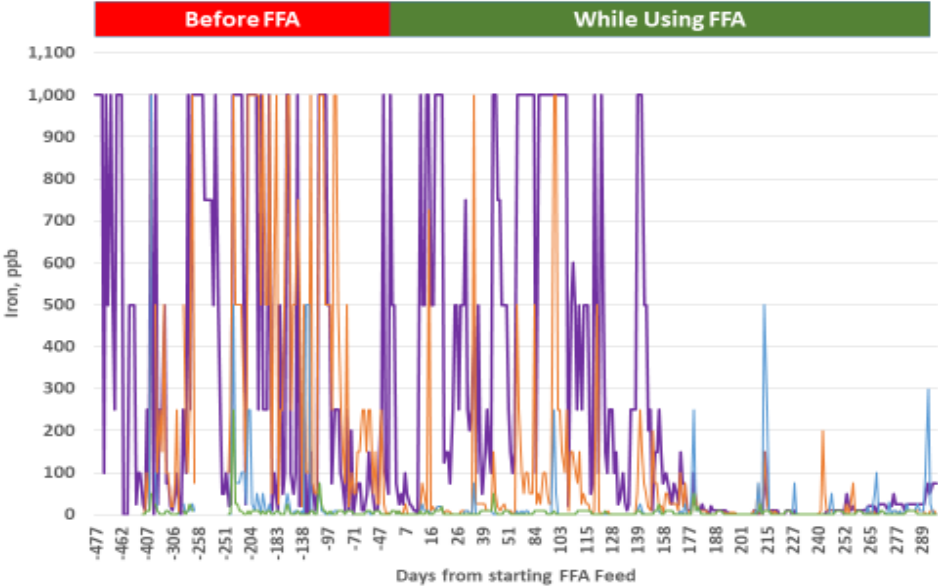


Figure 6. Millipore reading before and after FFA treatment

Figure 6 shows the iron residuals in the condensate, LPD, IPD, and HPD measured with a Millipore test before and after FFA treatment. LPD and IPD residuals were initially the highest, but the treatment program effectively reduced the iron residual. FFA treatment in this power plant's system resulted in a reduction of 413, 47, and 41 pounds of iron per year, respectively.

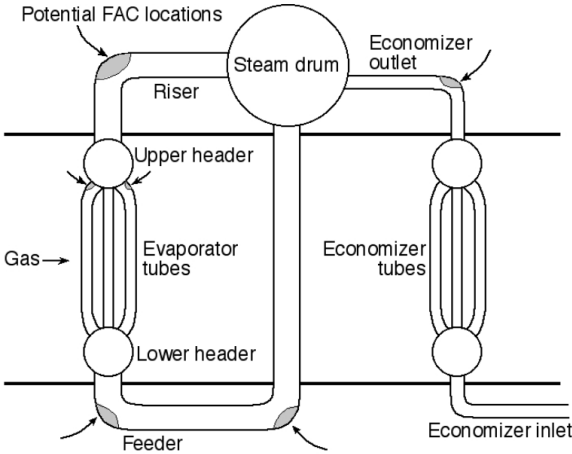


Figure 7. Areas that undergo FAC. EPRI FAC Guidelines Figure 1-2 page 1-6.³

As illustrated in Figure 7, wall thinning in several areas of the system can contribute to increased iron residual.³

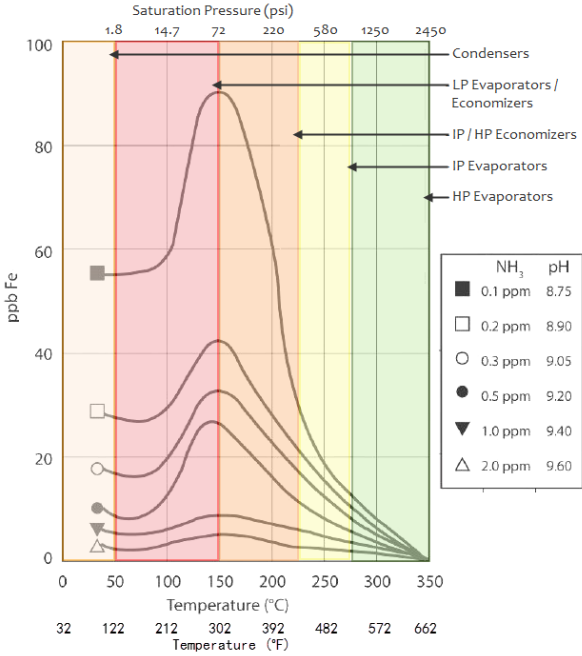


Figure 8. Magnetite solubility as a function of temperature at various ammonia concentrations. EPRI FAC Guidelines Figure 2-5 page 2-11.³

Figure 8 illustrates the solubility of magnetite based on temperature and pH, as well as the ammonia concentration needed to obtain a specific pH. The vapor-to-liquid distribution ratio for ammonia is approximately 11:1 at the LP operating pressure. Thus, a residual of 5 ppm in the feedwater will result in 0.45 ppm ammonia in the LPD, or a pH <9.2. This lower pH results in >30 ppb Fe solubility (302°F) from magnetite in the LP evaporator. Ammonia feed would need to be increased to 12 ppm to reach a pH of 9.4 in the LPD and approximately 10 ppb Fe solubility from magnetite. Since magnetite is a key component of the passivation process, reducing its solubility is important to developing a passive magnetite layer.

This plant has been operating with elevated iron residuals for over 4 years. In addition, the power market in the area created an environment where the plant frequently had to operate on a cycling basis, having to shut down regularly. During these downtime periods, the unit didn't circulate, nor was it drained. This type of intermittent operation can lead to oxygen and CO₂ ingress and pH depression, exacerbating corrosive conditions. The loose iron oxide layers formed under these conditions would ultimately displace when the units started, resulting in even higher iron concentrations.

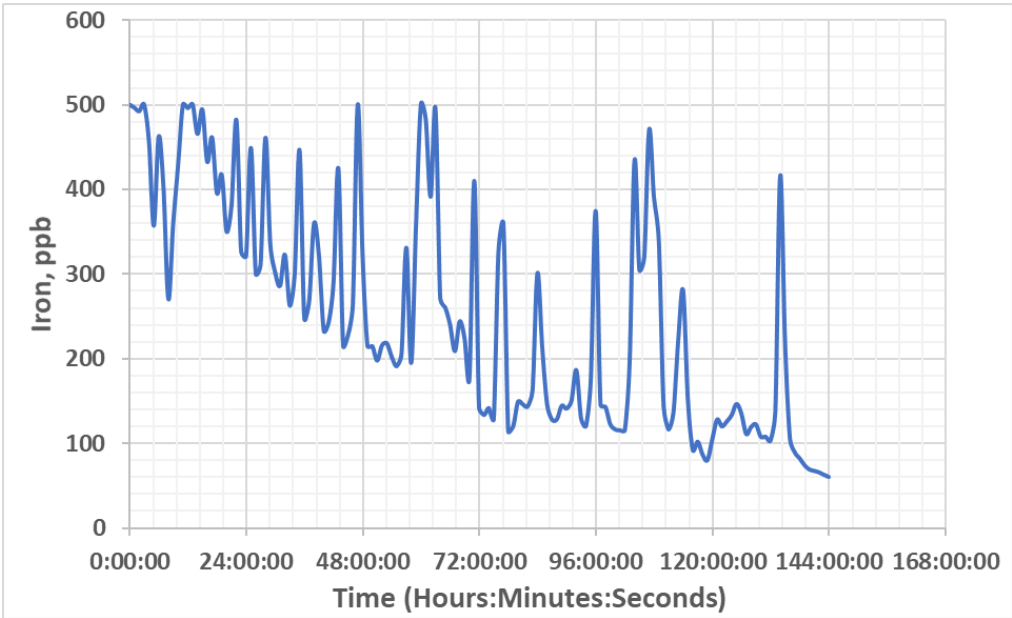


Figure 9. LP drum on-line iron residual logged during unit start 2 weeks after FFA application began

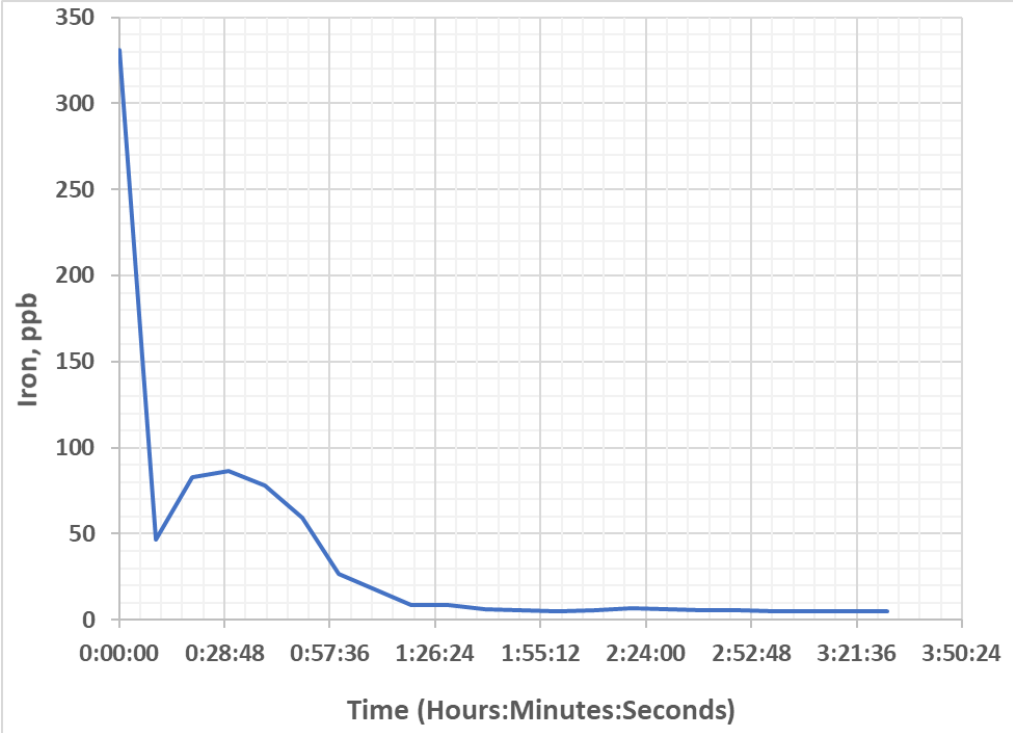


Figure 10. LP drum on-line iron residual logged during unit start 10 months after FFA treatment began

Figure 10 shows the overall iron reduction when treating the system with FFA. The comparison of on-line LPD data logged by a Swan nephelometer that reads iron concentration continuously can be observed in figures 9 and 10 after a unit start. After the unit start, iron residuals took a few weeks to drop below 500 ppb prior to FFA treatment, just over 6 days to drop below 80 ppb two weeks after FFA treatment started (Figure 9), and just under 10 minutes after 10 months of FFA treatment (Figure 10). These data indicate that boiler system iron residuals decreased as a result of FFA treatment.



Figures 11 and 12. Tiger stripe pattern in turbine exhaust duct hotbox area indicating two-phase FAC prior to FFA treatment (left). 10 months after FFA treatment, two-phase FAC pattern is absent (right).

Figures 11 and 12 show the two-phase FAC present in the hotbox area of the turbine exhaust duct was eliminated by the FFA treatment. The two-phase FAC is present in part because the low-pressure steam turbine exhausts to the hotbox area that guides the steam through a 90-degree turn. The water droplets impinge on the hotbox area, cracking the oxide surface and enabling the condensate droplet to penetrate to a less passive surface. Because the system is treated with ammonia only, the pH of the penetrating water droplet is low enough to be corrosive. The FFA was able to establish a coating that mitigated two-phase FAC as shown in Figure 12, which does not have the tiger stripe pattern observed in Figure 11. Reduction in iron returning in the condensate was also observed as the FFA passivated the air-cooled condenser. This also demonstrates that the FFA is thermally stable enough to passivate surfaces after the turbine (this is important to note, as the turbine receives superheated steam with a final temperature of $>1050^{\circ}\text{F}$).

The FFA was able to establish a coating that mitigated both single- and two-phase FAC under system conditions that make it difficult for the iron oxide to establish a passive coating. This was done at an economical treatment concentration during normal unit operation.

Ammonia Plant

One ammonia plant maintains pH to develop a passive oxide layer and limit corrosion in the boiler and condensate systems. A congruent phosphate program is applied in the boilers, and high- and low-volatility amines are applied in the condensate system to maintain pH. A prior water treatment program used a copolymer for iron transport, but the polymer had difficulty transporting iron in this boiler system, which operates at 1850 psi. This resulted in the need to plug bundles in the waste heat boiler responsible for cooling the hot processes gases from the reformer (101C), reducing process efficiency. A new water treatment program was initiated using a polymer designed for high pressures: polyisoprenylphosphonic acid (PIPPA). The remainder of the program continued to use congruent phosphate to control boiler pH and a combination of amines to maintain condensate pH.

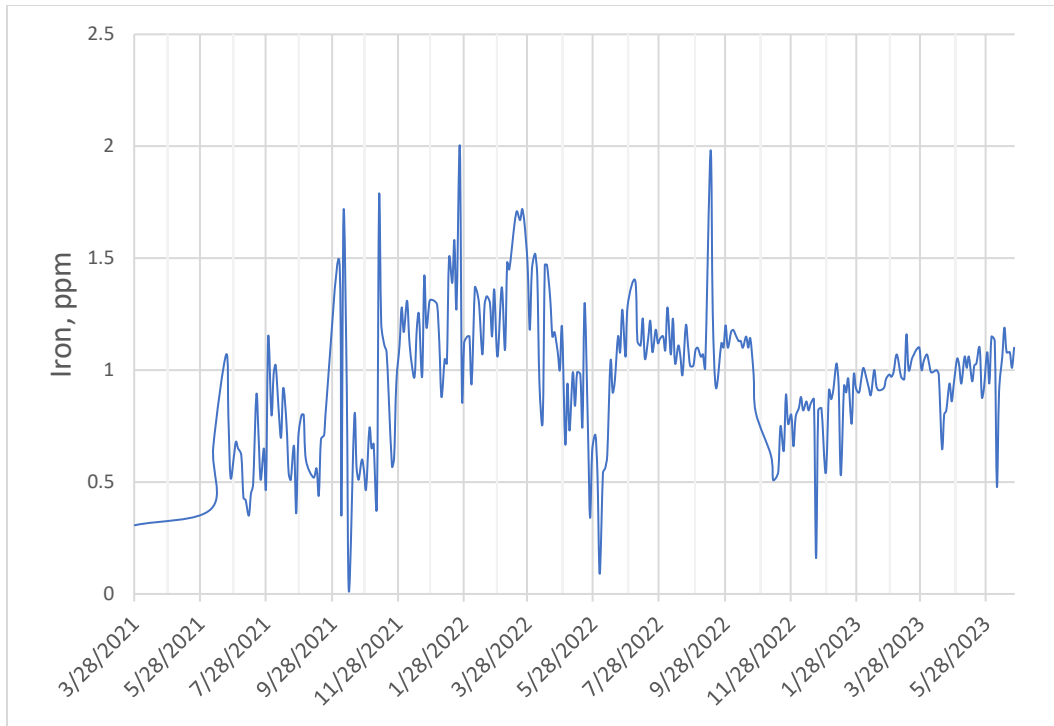


Figure 13. 101C iron residual at ammonia plant

PIPPA feed was started during the last week of May 2021, and iron residuals immediately increased from less than 0.5 ppm to over 1.0 ppm iron, indicating both the improvement of iron transport and the removal of previously deposited iron. Although well above the EPRI guideline for HP drums in a heat recovery steam generator (0.005 ppm), the boiler has been able to run effectively and efficiently for over two years since the program began, indicating that the PIPPA polymer is able to maintain iron transport.

In an advanced ammonia circuit, the 101C is fed by a steam drum (101F), which are both maintained at 1850 psi. Iron solubility at 1850 psi is very low, which makes it unlikely that there is a surface corrosion problem in either the 101C or 101F. The feedwater iron is averaging 5 ppb, but after the sample undergoes preheating, pH is at 9.4, maintaining a reducing environment by averaging 46 ppb DEHA. These feedwater conditions create an environment favorable for FAC; therefore, the feedwater is likely the source of the iron in the boiler circuit, although there may be other contributing factors.

Specialized Polymers and Film-Forming Amines

FFA application at the ammonia plant illustrates how a specialized polymer provides sufficient iron transport for maintaining efficient boiler operation. However, the iron residual remains around 1 ppm, well above industry guidelines. At 800,000 pounds per hour of steam, one would

expect the removal of 175 pounds of iron over one year. The high iron residual showed the need for further treatment.

The current treatment program at the ammonia plant relies on traditional amine feed to maintain condensate system pH and a congruent phosphate program to maintain boiler pH. The feedwater is maintained at pH 9.4 and 50 ppb DEHA, while the 101CA is maintained at pH 9.4 using a congruent phosphate program. The phosphate is maintained at 5 ppm.

The water meets the specification for high-purity water, which means the feedwater and reducing conditions make magnetite soluble and less likely to form. This is because the corroding surface gives off ferrous oxide, which needs to oxidize to ferric and combine with other ferrous oxide material to form magnetite. This process is slowed by the reducing agent. Once magnetite is formed, it is more soluble, increasing the concentration of magnetite necessary to create a particle, and leaves more soluble magnetite after the particle is formed. The increase in soluble magnetite after the magnetite particle forms enables the magnetite particle to grow through diffusion before settling on the surface. The larger magnetite particles do not form a passive layer as effectively as smaller particles, thus increasing the oxide layer. In addition, the particles are less uniform in size and do not adhere as effectively, leading to oxide thinning in sharp angles, creating the conditions for classic single-phase FAC. Because the temperature in the 101C and 101F is high, magnetite solubility is not significantly increased in these conditions, indicating that the iron in the 101C is coming from the feedwater system.

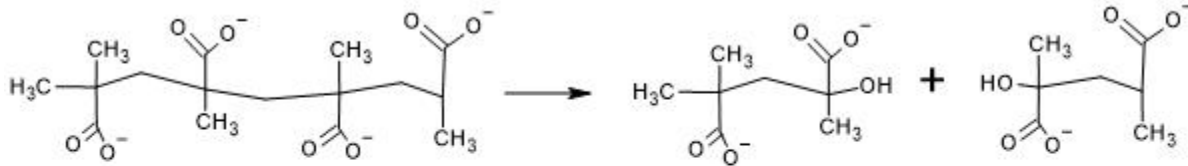
Dispersing iron in boilers typically involves high molecular weight methacrylic acid or a sulfonated acrylic acid copolymer. However, at operating pressures >900 psig, sulfonated acrylic acid polymers are not used, and, at >1200 psig, polymethacrylic acid (PMMA) is not used because of polymer breakdown. PIPPA is similar to PMMA and shares the same backbone, but instead of carboxylate functionality, it has phosphate functionality, which allows its breakdown products to transport iron.

Carboxylate polymers such as polymaleic acid (PMA), polyacrylic acid (PAA), and PMMA have been studied to understand the way they break down under pressure in boilers and lose functionality.⁷ The polymers break down by either chain scission or decarboxylation. Chain scission occurs when the carbon-to-carbon bond in the polymer backbone is broken and replaced by a hydroxyl group. This results in one fragment breaking into smaller fragments. Decarboxylation occurs when the active carboxylate group leaves as carbon dioxide and is replaced by an inactive hydroxyl group.

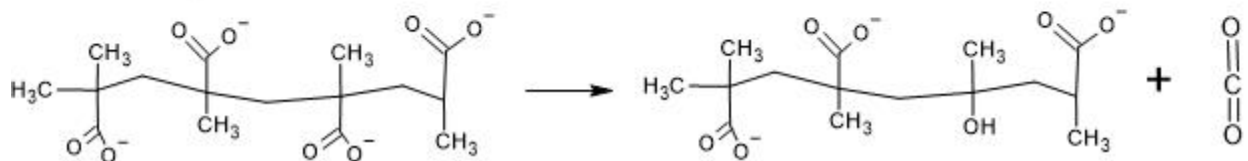
The Masler paper⁷ found PAA, PMMA, and PMA when heated at 575 psig/250°C for 18 hours showed decarboxylation of 9.5, 16, and 26%, respectively. At 1250 psig/300°C for 16 hours, PAA showed decarboxylation of 16% (the other polymers were not tested.) The same paper also looked at molecular weight and found that PAA, PMMA, and PMA lost 23, 13, and 27% of their molecular weight (Mn: number average molecular weight), respectively, while PAA lost 51% of its Mn at 300°C. Using linear regression, PAA is projected to have 17% decarboxylation and 55% loss in molecular weight at 1,400 psig and 18% decarboxylation with 59% loss in molecular weight at 1500 psig.

Extrapolating to PMMA at 1,400 and 1,500 psig, we can calculate 29% decarboxylation with 31% loss in molecular weight at 1,400 psig and 30% decarboxylation and 33% loss in molecular weight at 1,500 psig. At 1,400 psig, 12.3% of the molecular weight loss came from the 29% decarboxylation with the remainder from chain scission. The results indicate that PMMA has a significantly lower chain scission rate and a slightly higher decarboxylation rate, which makes it the preferable high-pressure carboxylate polymer. However, the elevated loss of molecular weight and decarboxylation at >1,200 psig indicates the polymer may have difficulty maintaining its dispersive capabilities.

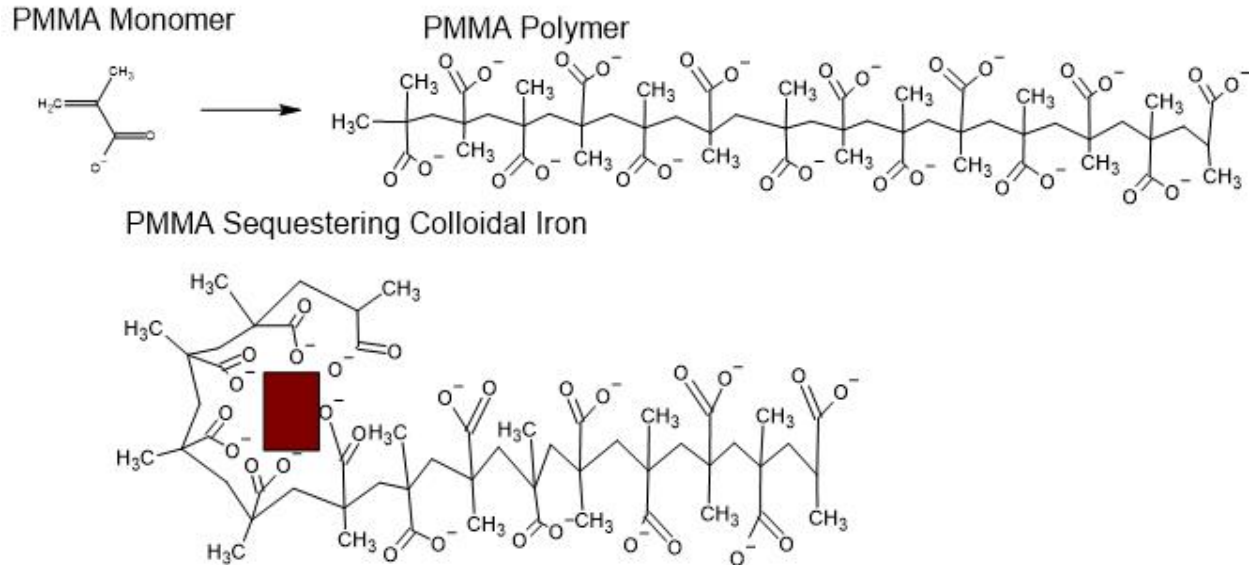
Chain Scission



Decarboxylation



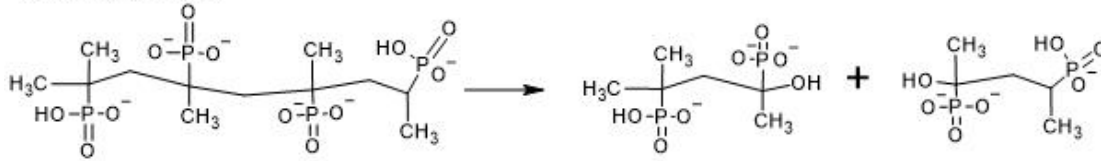
Most of the feedwater system iron coming from the condensate is particulate. The particles have charged portions that attract iron oxide and allow it to grow through diffusion. Anionic polymers are attracted to the charged areas of the particle. For the particle to sequester and transport, there must be enough anionic portions of the polymer to form electrostatic bonds with the iron particle and enough free anionic charge in the polymer to keep the polymer particle complex suspended in the water flow.



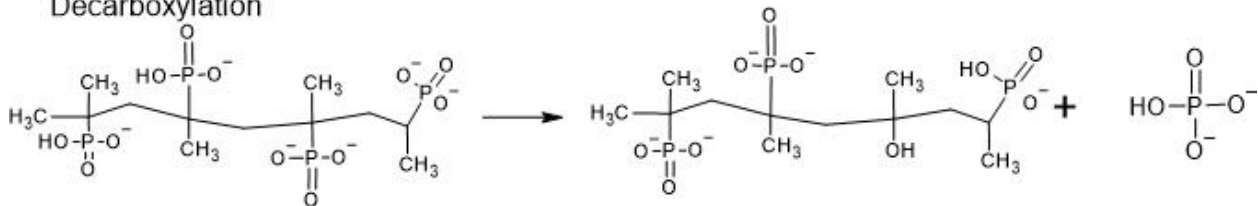
Because PMMA is fed neutralized with sodium at the ammonia plant, the degradation of PMMA led to significantly higher sodium-to-phosphate ratios because the carboxylate counter ion to sodium in the polymer is lost during degradation. The sodium-to-phosphate molar ratio reached as high as 4.6, which far exceeds the 2.6 target. Additionally, the boiler iron concentration during this period fluctuated between 3 and 7 ppb, independent of polymer feed rate indicating a loss in iron transport. These field results agree with the Masler study discussed earlier⁷.

As mentioned previously, PIPPA is comprised of a PMMA polymer backbone with phosphate attached. The polymer degrades by both phosphate leaving the polymer backbone (similarly to decarboxylation) and the carbon-to-carbon bonds breaking in the polymer backbone and being replaced by hydroxyl groups (similarly to PMMA decomposition). In the process of dephosphorylation, the leaving phosphate gains one acid group, decreasing the sodium-to-phosphate ratio in the boiler. Caustic soda is added to maintain the proper sodium-to-phosphate molar ratio, and phosphate feed is adjusted to maintain the phosphate residual at target levels. Since increased retention time causes an increase in dephosphorylation, PIPPA application may be a good option for units with a relatively constant load.

Chain Scission

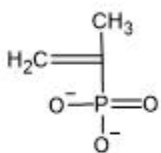


Decarboxylation

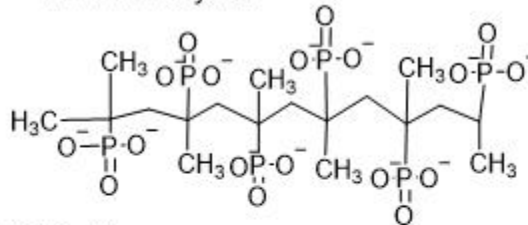


Applying PIPPA in a boiler operating at 1500 psig/315°C, the author has observed well over 75 to 85% degradation through dephosphorylation when running between 50 and 75 cycles. At elevated cycles (300), PIPPA has been known to transport iron through the boiler but contribute to deposit weight densities on evaporator tubes. For this reason, maintaining <100 cycles is recommended for PIPPA applications. Elevated iron transport has been observed with dephosphorylation as high as 95% at an operating pressure of 1800 psi, indicating enough intact polymer was available for iron transport. Literature⁸ reports the elevated transport capabilities of PIPPA in high-pressure boilers consistent with experience.

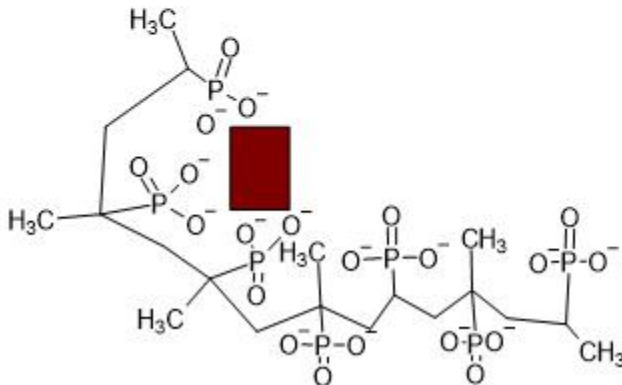
PIPPA Monomer



PIPPA Polymer



PIPPA Sequestering Colloidal Iron



PIPPA uses a phosphate functionality unlike the carboxylate of PMMA and PAA. The phosphate has an anionic charge in addition to the more polar portions of the molecule around the phosphorous molecule, allowing PIPPA to transport iron with smaller polymer fragments. The phosphate is also more adherent to iron oxide, making PIPPA a preferred polymer for boilers operating at >1,200 psig.

However, care should be taken when applying PIPPA because of the increased iron transport, especially when taking into account that <20% of the iron entering a boiler leaves it through blowdown. At 20 ppb iron and 50 cycles, 200 ppb iron is leaving the boiler, and a steaming rate of 500,000 pounds per hour every day all year long would leave 70 pounds iron or 97 pounds of magnetite per year building up in the boiler. A boiler operating for 5 years without cleaning would contain close to 500 pounds of magnetite. Some of the iron built up in the boiler will agglomerate and fuse into larger masses. Applying PIPPA to a boiler in this situation could result in dislodging of a large mass of magnetite, which could impact a boiler tube, potentially causing tube clogs or failures. In addition, iron residuals in the boiler will increase significantly beyond the concentrations deemed acceptable by industry guidelines, which may cause fouling of a variety of valves or other equipment, impeding proper operation. For this reason, we recommend carefully evaluating a boiler before applying PIPPA and monitoring dosage closely to allow for consistent and efficient boiler operation.

FFA feed can also be considered in conjunction with polymers or as a standalone program for transporting iron and reducing the overall corrosion byproduct by developing a passive film. Applying FFA in the ethanol plant illustrated how, after one month, the iron residual in the condensate decreased significantly. Reducing iron residual in the economizer took more time because the corroding surface made it harder for the FFA to establish a film. However, once the film was established, the iron residual was almost 70% less than it was without FFA, indicating the ability of FFA to reduce FAC, though it took more time.

The power plant application was also able to overcome FAC conditions. However, because the water balance was too low and the blowdown was not increased, iron residuals were maintained at approximately 1 ppm. Subsequently, product feed had to be increased by 50% to overcome the additional surface area introduced by the iron residual so the FFA could develop a film and arrest FAC.

FFA application at the ethanol plant economizer and power plant took a little longer to reduce iron residual because the FFA needed to establish a solid surface that could withstand a film. FFA develops a film by establishing a bond with the iron within the iron oxide, creating a torque on the surface. This torque results in the removal of the loose iron oxide and continues until the torque is no longer strong enough to remove the iron oxide on the surface and develop a film. The density of this film develops until there is a uniform monolayer. As the surface demand lowers in one region, more FFA residual is available for binding in areas of higher demand. When enough time has passed and a high enough residual is established after surface demand for FFA is decreased through monolayer development, a FFA bilayer is established. This usually occurs in cooler operating areas initially and is observed through the development of water droplets on metallic surfaces.

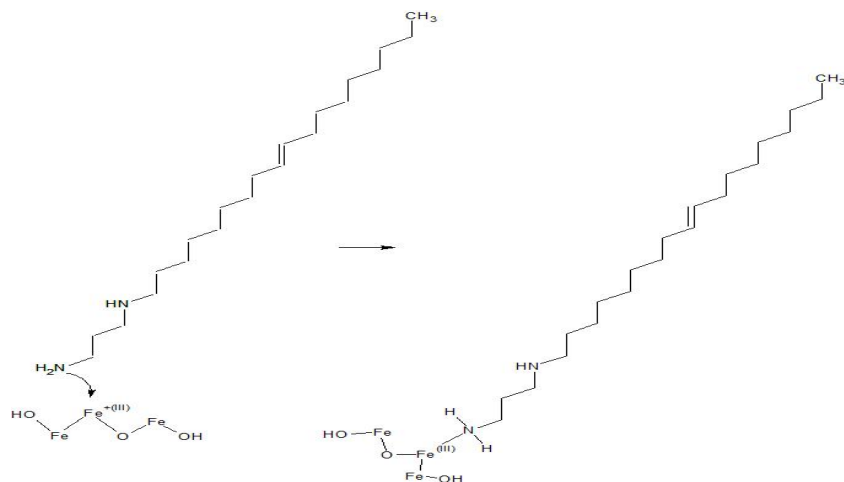


Figure 14. FFA binding to surface oxide by donating the lone pair of electrons on the nitrogen to an acidic iron

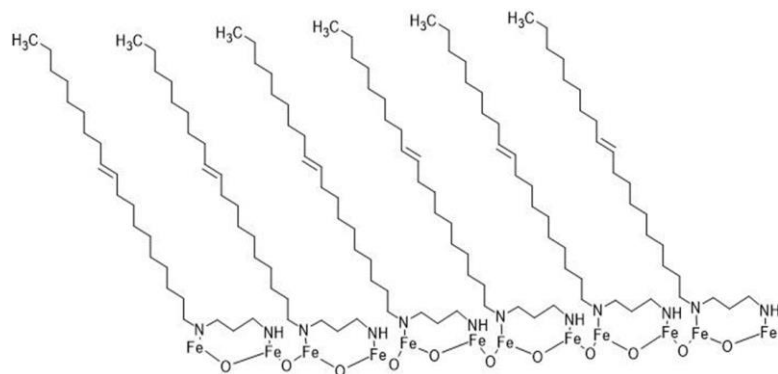


Figure 15. FFA bound to the surface iron oxide in a monolayer

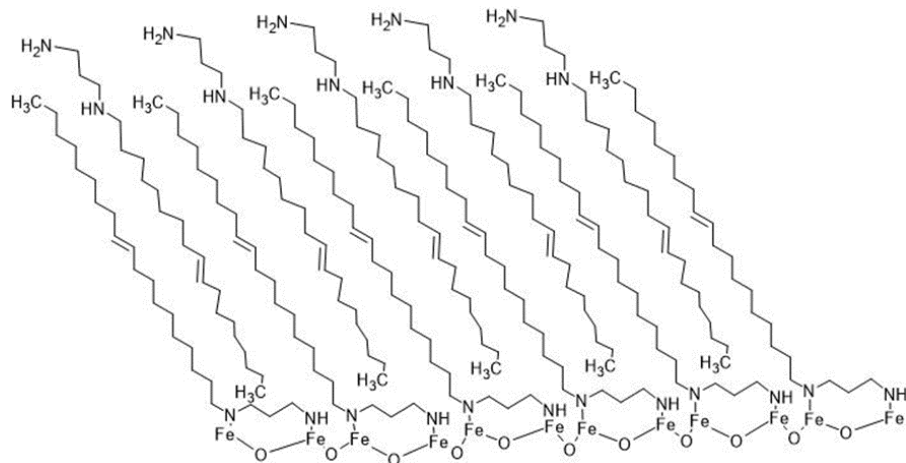


Figure 16. FFA forming a bilayer on the mild steel surface

The bilayer is labile and elutes off the surface if the FFA residual is not maintained. However, the monolayer is persistent because of the bond. The monolayer provides significant corrosion protection, and the bilayer further protects surfaces from corrosion.

A carefully maintained FFA program uses the proper feed rate based on iron residual levels and is actively monitored through colorimetric dye tests on-site or through chromatography testing in the laboratory. Theoretical concentration modeling is performed through applying the known distribution coefficients to the mass transport formulas.⁵

Deviations in the expected residuals are monitored and may require troubleshooting for a more thorough understanding of system conditions that could affect the maintenance of the passive FFA film. Persistent iron residuals are not only a frequent sign of active corrosion but represent a significant increase in surface area for FFA to establish a bond and subsequently reduce the available FFA to prepare the surface to develop a film. Increasing boiler blowdown rates can decrease iron residual to ASME or EPRI specifications. Once the passive film is established, the blowdown rate can be reduced. In rare cases where blowdown is not an option, we recommend increasing FFA residual so there is sufficient residual to establish a passive film.

Conclusion

Both the ethanol and power plant applications benefited significantly from the application of FFA, reducing iron concentrations under FAC conditions. At the ammonia plant, FFA application should be considered in conjunction with PIPPA application to arrest corrosion and reduce the overall iron residual closer to the EPRI guidelines. The application of FFA would both assist in iron transport and reduce the corrosion rate during operation, as noted by Lensun.⁶

The applications and results discussed in this paper are examples only; they are not guaranteed, and results may vary. Each system is different and has unique treatment needs, and due diligence is necessary for determining the feasibility for utilizing these methods. Always consult your equipment manuals and guides and contact a water treatment professional before making changes to your systems and treatment processes.

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