

MAX HT™ SODALITE SCALE INHIBITOR: PLANT EXPERIENCE AND IMPACT ON THE PROCESS

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Abstract

Development of a reagent to prevent sodalite scale formation in the Bayer process was presented at the 2005 TMS. Since that time this product has been run on many plants across the globe under various operating conditions and has demonstrated that it completely eliminates sodalite scale. This paper will cover the application experience gained and the benefits derived at these plants.

Introduction

Silica that is present in bauxite ores as silicates, primarily clay minerals, dissolves quickly under typical Bayer alumina digestion conditions. A majority of the silica subsequently precipitates as a sodium aluminosilicate – sodalite or DSP (desilication product). The Bayer liquor remains supersaturated in silica, however, and this supersaturation is greatest after the alumina precipitation step, i.e., in the spent liquor. As the alumina-depleted liquor is reheated, the rate of silica precipitation in the form of sodalite increases markedly with increasing temperature due to faster kinetics. This precipitation occurs as scaling on the inside of the heat exchange tubes and significant loss of heat transfer occurs. Besides the obvious costs of maintenance and labor required to clean the scaled up heat exchangers, the impact of scale may also be seen in increased energy consumption, increased caustic losses, reduced liquor flows, reduced throughput, reduced evaporation, and even reduced production.

Reagents developed for the successful elimination of sodalite scaling were presented in 2005 [1], followed later that year by initial plant results using these reagents [2]. After some background information, this paper will deal primarily with experience that has been obtained at a variety of plants.

The sodalite scale reagent MAX HT™ is commercial at five different plants across the globe. Trials are in progress or planned at an additional ten or more plants. None of these plants have experienced any negative downstream effects.

The original MAX HT reagent does not work in the presence of large amounts of solids as found in single stream plants. In single stream plants, fresh bauxite slurry is mixed with spent liquor before going through the heat exchangers on the way to digestion, as opposed to double stream plants where only the liquor flows through heat exchangers. However, even in single stream plants, benefits have been found in treating evaporator heaters with MAX HT.

In some double stream plants, there are low levels of solids present in the liquor. Under these conditions, the original MAX HT does not work effectively. A new product has been developed to address this issue.

Mechanism

The mechanism and chemistry involved is useful in understanding how the antiscalant reagents work.

The general chemistry of our antiscalant reagents has been disclosed [3, 4]. A generic structure for the sodalite scale inhibitors is shown in Fig. 1. There are several important features of this structure: 1) they have a silicon-containing group Si—O₃; 2) they are polymeric materials; 3) a wide variety of materials are possible for the polymeric backbone; 4) other substituents attached to the backbone may also cover a wide variety. In other words, there is not a single reagent, but in principle a large family of reagents.

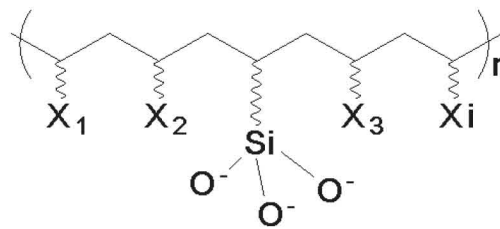


Figure 1. Generic structure for MAX HT antiscalants.

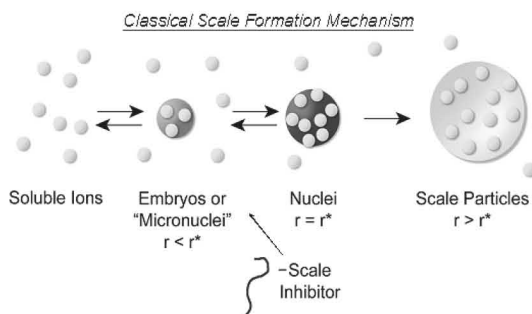
Most essential is the functional group that contains silicon. This Si—O₃ grouping occurs also in the sodalite structure and it is thought that this group is temporarily incorporated into the structure at a very early stage and prevents nuclei from reaching a critical size necessary to continue growth [1, 2]. Because of the amount of material attached to this group, however, the growing crystal cannot incorporate the large molecule into its structure and the crystal goes back into solution instead of growing larger. Small molecules containing this Si—O₃ grouping are not effective in preventing sodalite scaling. The bulky group attached to the Si—O₃ is essential in keeping the molecule from being incorporated into the growing sodalite. This mechanism is schematically illustrated in Fig. 2.

An important consequence of the proposed mechanism is that the reagent is not used up as it would be, e.g., if it were adsorbed onto a large amount of sodalite surface. Surface adsorption is not required for this mechanism. Since the reagent is not used up, it can be added as early as desired in the heat exchange train, even before evaporators, and will work throughout the entire train. The reagent will ultimately be adsorbed onto the massive quantities of red mud present in digestion.

The proposed mechanism is consistent with why the reagents work at relatively low dosages. Typical silica concentrations are ~500-1000 ppm (0.5-1.0 g/l) while the concentrations of reagents required to prevent precipitation of this silica are far less, typically ~20-40 ppm. Conversely, it is also clear that if dosage is insufficient, some of the sodalite nuclei will continue to grow

rather than going back into solution and the result is scale.

Sodalite Scale Inhibitor - Concept



Scale formation occurs irreversibly once "nuclei" grow to a critical size (r^*)
Inhibition occurs because the scale inhibitor interferes with growth of embryos at early stages and prevents growth to stable nuclei size

Figure 2. Antiscalant mechanism.

Besides testing as sodalite antiscalants, a number of the reagents have been evaluated for adsorption onto sodalite, gibbsite, and other solids. Among materials that showed excellent performance as antiscalants, some adsorb rapidly onto sodalite, but others appear not to adsorb at all. That is, adsorption onto sodalite is definitely not a requirement for good antiscalant activity. Similarly, some of the reagents adsorb onto gibbsite, oxalate, etc. but again with no correlation to antiscalant activity. As indicated above, the polymeric structure to which the essential Si—O₃ group is attached can vary widely and it is the properties of this polymeric structure that primarily determine properties such as adsorption.

Liquor Chemistry

Problems caused by scaling in a particular plant will depend on a wide variety of factors related primarily to the type of bauxite used and details of the liquor chemistry. Although Bayer liquor chemistry is basically similar at all plants, the concentrations of the major components such as caustic, alumina, silica, carbonate, and sulfate vary significantly from plant to plant. Minor impurities vary even more. Temperature is also a major variable. The effects of most of these parameters on sodalite scaling are fairly clear.

Besides the obvious effect of increasing scaling with increasing temperature, the variables with the greatest effect appear to be the silica and alumina concentrations. With other variables held constant, the tendency of sodalite scale to form obviously increases as silica concentration in the process liquor increases.

Alumina has the effect of stabilizing the silica in solution so that the dependence on alumina concentration is opposite to that of silica; i.e., the tendency of sodalite scale to form increases with decreasing alumina concentration. Plants that operate with lower alumina levels in their spent liquor will generally have greater sodalite scaling problems. This could be especially important, e.g., if a plant pushes for more yield by going to lower A/C in the spent liquor. Use of MAX HT would eliminate the scaling problems caused by the lower A/C.

Increasing sodium hydroxide stabilizes silica in solution and makes sodalite scaling less likely. Carbonate, however, has the opposite effect; increasing carbonate causes sodalite to form more readily. Similarly, sulfate is incorporated even more readily than carbonate [5] and high sulfate levels in liquor increase the scaling problem. Somewhat surprisingly, most organics in the liquor, including oxalate, have relatively little effect on sodalite scaling [6].

Since the various components in the liquor have differing effects on the tendency for sodalite scale to form, the net scaling rate will be a balance of many factors and will clearly vary from one plant to another. At a given plant, changes in one of the variables, e.g., silica concentration or alumina concentration can be well anticipated. A potentially great advantage of MAX HT usage is that one no longer need be concerned about a change that might promote greater sodalite scaling. For example, some low silica bauxites that have been avoided in the past could now be comfortably be used with MAX HT.

Effects of solids in the liquor

During some of the initial plant testing, it was recognized that even in double stream plants the liquor may contain small amounts of solids. These can be gibbsite, "red mud", sodalite, or oxalate solids. Gibbsite is not really a problem because by the time the heaters get hot enough to experience significant scaling, the gibbsite will have dissolved. Red mud solids are typically <10 mg/l in most plants and this concentration presents no particular problems. In some plants, however, especially those that use sand filters, red mud solids may be 50-150 mg/l. These red mud solids have the effect of adsorbing some of the MAX HT and increasing the dosage required to eliminate sodalite scaling. In lab tests, MAX HT dosage can be increased by as much as a factor of two or three due to the presence of ~150 mg/l red mud solids.

As might be expected, sodalite solids in the liquor have more effect than red mud solids. As little as 10-20 mg/l can cause a significant increase in MAX HT dosage requirement. Particulate sodalite can result, e.g., if the liquor passes through an evaporator unit before entering the digester heat exchangers. In this case, the easy solution is to add MAX HT before the evaporator. It will then prevent sodalite formation in the evaporator and continue to prevent sodalite scaling in the heat exchangers. In some cases, a small amount of sodalite may form in the precipitators.

Oxalate solids can also have a significant negative effect on MAX HT dosage requirement. Fortunately, solid oxalate is not common in a plant that is running well. Note that oxalate in solution has no effect on MAX HT performance; it is only the solid that adsorbs some of the reagent and renders it ineffective.

The original MAX HT has the disadvantage that it does adsorb fairly readily onto red mud, sodalite, and oxalate solids, causing increased dosage requirements. To prevent these problems, a new antiscalant, MAX HT 500, has been developed that is much less sensitive to the presence of such solids. The reagent will still be adsorbed onto red mud when it reaches the digesters.

In principle, an antiscalant reagent that does not adsorb significantly onto gibbsite can be added even before precipitation.

Sodalite that co-precipitates with the gibbsite would be prevented, resulting in less silica in the product alumina. This appears to be a significant problem in a few plants.

Thermal Conductivity

Thermal conductivity of sodalite scale has been reported to be 0.3 W/m-°K [7], and this appears to be a widely accepted value. ~0.3 W/m-°K is also reported for a sodalite powder [8]. However, values of 1.2-1.7 W/m-°K have also been reported [9]. Thermal conductivity of dense sodalite has been reported to be ~3 W/m-°K, [10] similar to handbook values for dense silicates. Looking at an SEM photo of a sodalite scale from a plant (Fig. 3), it is clear that the limited number of good contact points between these particles would lead to poor heat transfer. If thermal conductivity of the sodalite is as low as ~0.3 W/m-°K, liquor trapped in this porous structure should raise thermal conductivity somewhat since thermal conductivities of concentrated salt solutions are similar to water, ~0.7-0.8 W/m-°K in the temperature range of interest. Resulting thermal conductivity for the sodalite/liquor layer should therefore be ~0.5 W/m-°K. If, instead of liquor trapped in the porous structure, there are polymeric materials such as humates or even MAX HT, which could result from poor mixing and inadequate dosage, the resulting thermal conductivity might be slightly lower than ~0.3 W/m-°K, since most polymers have thermal conductivities in the range of ~0.1-0.3 W/m-°K. Thermal conductivity for a sodalite/polymer layer might be ~0.2 W/m-°K. In sharp contrast, thermal conductivity of a clean steel is ~40. W/m-°K.

The clearest way to see the effect of poor thermal conductivity of the scale is to look at thermal resistivities [11]. The thermal resistivity of the clean heat exchanger is $(1/HTC_{clean})$, where HTC is the heat transfer coefficient of the clean exchanger. Thermal resistivity of the scaled heat exchanger is:

$$1/HTC_{scaled} = 1/HTC_{clean} + t/K, \quad [1]$$

where t/K (scale thickness/scale thermal conductivity) is the added resistivity due to the scale.

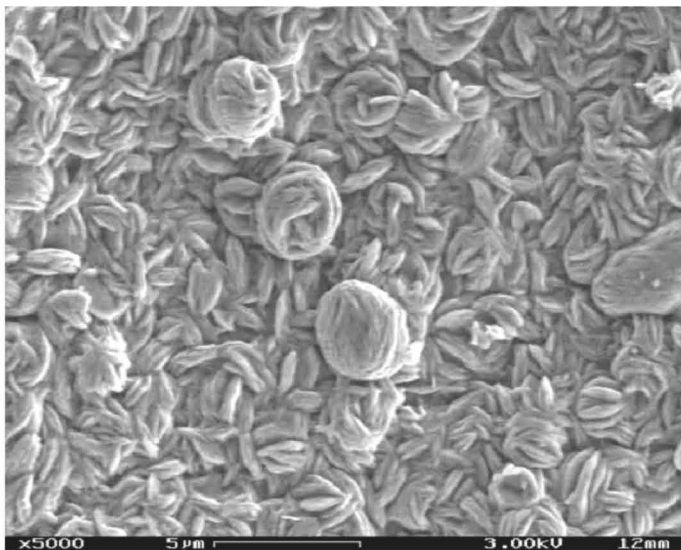


Figure 3. SEM photo of scale from an operating Bayer plant. Most plants clean heat exchangers when HTC is reduced to about half its starting value (thermal resistivity doubles). Using equation 1, one may readily calculate scale thickness required to

reduce HTC by 50% vs. thermal conductivity of the scale. Fig. 4 shows that the required scale thickness is generally less than one millimeter, depending on the thermal conductivity. For the frequently quoted value of 0.3 W/m-°K for scale thermal conductivity, the required thickness is less than 0.2 mm!

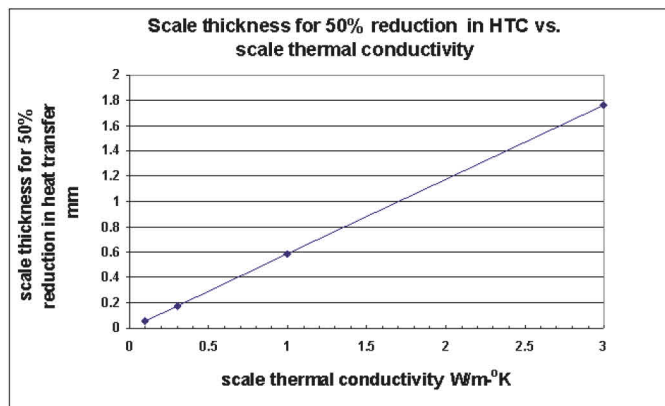


Figure 4. Dependence of sodalite scale thickness on scale thermal conductivity.

It is quite likely, however, that morphology and packing of the sodalite particles – therefore thermal conductivity - may vary significantly from plant to plant, and this may be one of the reasons why some plants have greater problems than others due to scale. In any case, the scale thickness required to decrease heat transfer by a factor of two is surprisingly small.

Regardless of the nature of sodalite scale that has formed in the past, use of MAX HT can completely prevent such scaling.

Plant Application

Dilution and Mixing of MAX HT

Since the start of plant testing, it has become clear that proper dilution and mixing of MAX HT with liquor are most important. First, the “as supplied” reagent is to be diluted with a relatively clean water source and this is readily accomplished by metering the reagent and water through a static mixer and several meters of one inch pipe. With the current MAX HT, dilution to 5 % is important – less dilution gives some loss in performance, but more dilution gives minor improvement (Fig. 5). The extent of dilution must be balanced against the cost of adding extra dilution water.

The reason for the poorer performance when MAX HT is added to liquor at high concentrations is that the MAX HT reacts with something in the liquor to form “insoluble complexes”. These insoluble complexes are obviously quite stable in the liquor since they do not go back into solution even in an overnight lab test. Only the MAX HT that remains in solution can be effective as an antiscalant, so the real concentration is reduced and performance is worse than expected.

These insoluble polymeric “blobs” which visually appeared very dark and tar-like (probably due to incorporated humates) were especially evident when 10% MAX HT was added to liquor. These polymeric blobs were identified by FTIR as being rich in

MAX HT. We specifically looked at whether any of the impurities in the liquor were concentrated in these polymeric blobs and saw no evidence at all for this. These blobs or complexes appear to be a consequence of the high ionic strength of the liquor; they will readily dissolve in clean water. This mixing problem is not necessarily unique to MAX HT, but will be characteristic of many polymer solutions when added to a solution of extremely high ionic strength, such as a Bayer process liquor.

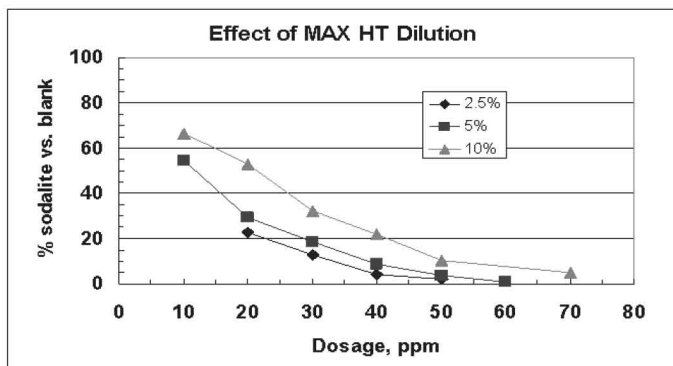


Figure 5. Effect of MAX HT dilution in preventing sodalite formation in a plant liquor with added silica.

Lab Simulation of Poor Mixing

Poor mixing of diluted MAX HT solution with liquor has much the same effect as using a solution that is too concentrated. Poor mixing will give local areas that are high in MAX HT concentration, which result in formation of the insoluble polymeric blobs.

It is, of course, impossible to reproduce plant mixing conditions in the lab. We were able to show, however, that if 5% MAX HT solution contacted the liquor for a short period of time before it was thoroughly mixed in, results could be far worse than when initial mixing was good. Test results are summarized in Fig. 6 Depending on mixing, performance can be much worse than the case with good mixing (bottom curve) (details of mixing are not important for the various curves, only that they represent a range from very good to poor). In most cases, performance seems to be leveling off and is still well above zero sodalite even at 60 ppm dosage.

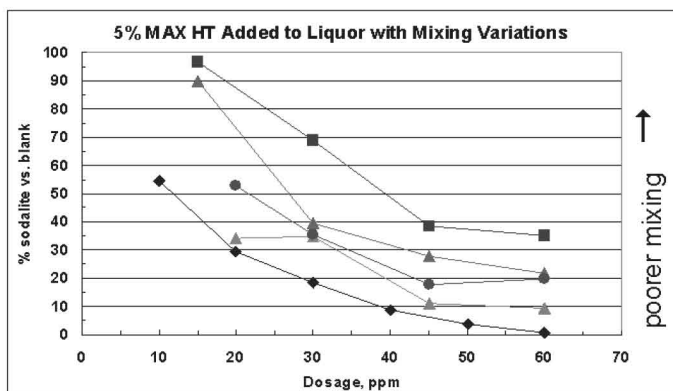


Figure 6. Effect of mixing on MAX HT performance (in a plant liquor with added silica).

The above results emphasize the fact that the diluted MAX HT must be very quickly dispersed after contacting liquor. It is essential that diluted MAX HT can not be added to an unstirred tank

Obviously, one cannot draw quantitative conclusions from such simplistic experiments, but the important point is that the results can be quite sensitive to mixing, in this case the mixing of the 5% MAX HT solution with the spent liquor. By varying only this mixing, we get a wide range of results, some of them much worse than the case of good mixing.

Conclusions based on Mixing

A clear conclusion from all of these mixing experiments is that any situation that results in high local concentrations of MAX HT in liquor will result in formation of insoluble polymeric blobs. This can result either from insufficient initial dilution of the MAX HT or from poor mixing of the diluted MAX HT when it contacts the liquor.

Consequences of poor mixing are: 1. Performance (at a given dosage) is not as good as it should be because part of the MAX HT has been rendered inactive by formation of the insoluble complexes. 2. The problem of formation of insoluble complexes gets worse as MAX HT dosage increases because of higher local concentrations of MAX HT. Complete elimination of scaling will not be possible even at high dosages. 3. The insoluble complexes that are formed may show up as dark, tar-like deposits, especially in areas where liquor flow is relatively low.

Recommendations for mixing are now to first dilute the MAX HT to 5 %, or less if the dilution water is not a problem. Secondly, the dilute solution must be rapidly dispersed into the liquor. This would generally be done by injecting the MAX HT solution through a 1" line into the typically 16 or 20" feed line. Flow rate of the MAX HT solution through the 1" pipe should not be less than 30 cm/sec, which is turbulent flow (at a dosage of 30 ppm of 5% solution, flow rate through the 1" pipe would be 34 cm/sec). Flow rates in the liquor feed pipe are typically 60-120 cm/sec and also turbulent flow, so mixing of the two streams should give rapid dispersion of the MAX HT. Also, the mixing point should be at least thirty pipe diameters before the first treatment point, if possible.

Plant Experience

Benefits from using MAX HT previously reported [2] are summarized in Figs. 7-9. Scaled heater tubes and declining heat transfer are changed into clean tubes and constant heat transfer when MAX HT was used.

Sodalite scale inhibitor MAX HT is used commercially to eliminate and/or minimize scaling in evaporator and digestion heater tubes at dosages ranging from 20-40 ppm. Without use of MAX HT plants have minimal control on the rate of scaling in these heater tubes. Heater cleaning cycles vary from about 5 days to 60 days depending on chemistry of the liquor, silica in bauxite, desilication, and operating temperature in heaters, etc. Current practice is to acid or mechanically clean heater tubes on a regular cycle to maximize benefits realized for operating with clean

heaters. MAX HT has allowed plants to gain control or completely eliminate the formation of sodalite scale in heaters.

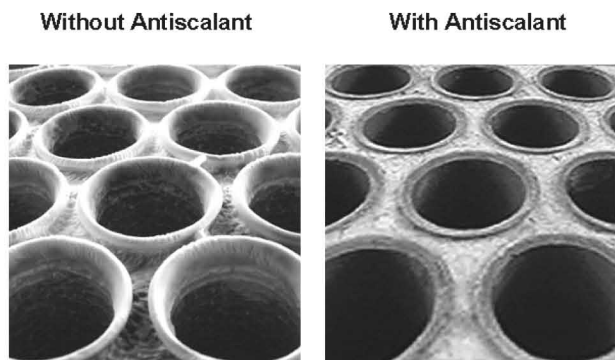


Figure 7. Dirty and clean heat exchangers from operating without and with MAX HT antiscalant, corresponding to the heat transfer curves in Figs. 8 and 9, respectively.

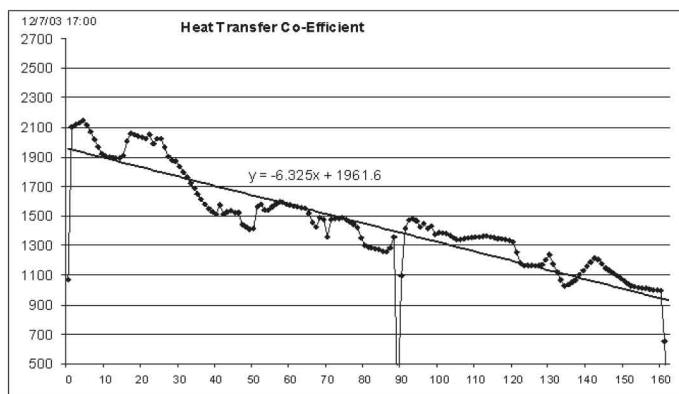


Figure 8. Typical heat transfer decay during ~7 days when no antiscalant was used.

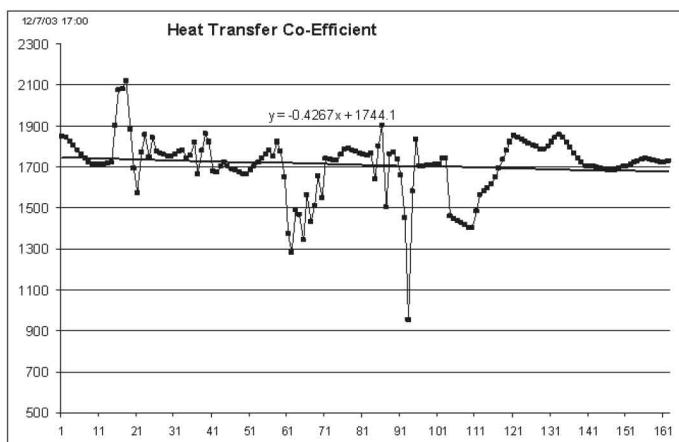


Figure 9. Constant heat transfer coefficient resulting from use of MAX HT.

Many plants do not have all the instrumentation required to collect data to calculate the heat transfer coefficient (HTC) and rely on selected process data which is readily available such as flows, temperatures, pressures, amp draw on pumps, along with visual inspections of tubes and piping to set desired heater cleaning cycles. In one plant the live steam flow to an evaporator

heater is used as a guide to set the desired cleaning cycle. As shown in Fig.10 the on stream time for this evaporator increased from 30 days to 155 days with use of MAX HT. This unit was taken off line after 155 days to inspect the tubes for scale and repair tube leaks which were present from the beginning of the trial. Normal capacity of this unit is reduced some 25% after 30 days at which time the unit is taken out of service from some 2-3 days for cleaning and maintenance.

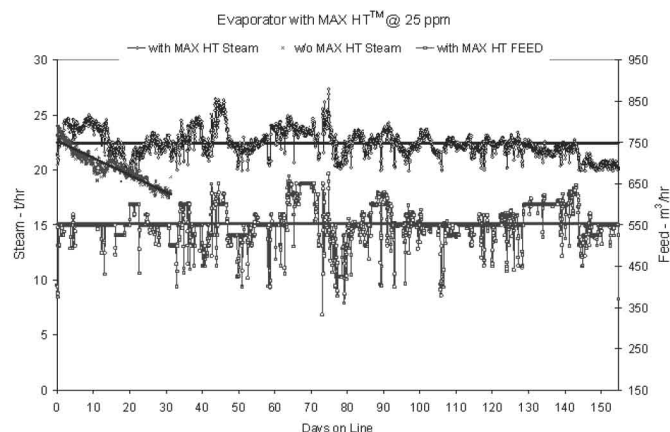


Figure 10. MAX HT used on an evaporator

In another plant, the fouling rate (1/HTC) is used as a guide to set heater cleaning cycles. As shown in Figure 11, the fouling rate is significant whereby heaters are cleaned every 5-6 days. Many times a heater and its associated spare are returned to service before being properly cleaned as shown by the data in Figure 11. Visual inspection before start of the test showed that some 15-18% of the tubes in the heater were plugged with scale after cleaning. Heater cleaning schedules become a real problem during drilling of tubes (by outside contractors), contributing to flow cuts and production losses.

A 53 day test conducted on this heater with MAX HT showed the fouling rate of the heater remained constant requiring no heater changes and the feed rate to the unit averaged 20% higher. No scaling is observed at a dosage of 20 ppm and plans are to continue testing on this heater with a lower dosage.

MAX HT has been used successfully to control or eliminate scaling in evaporator and digestion heaters at temperatures ranging from 80°C to 210°C. In general, dosage requirements increase with increasing operating temperatures. Plants normally adjust dosage to prevent scaling in the highest temperature heater, whereby others find it more economical to operate at a dosage

with no scale in the low temperature heaters such as in the evaporators with minor scaling in the higher temperature digestion heaters. Typically the on-stream time for a heater is increased from some 8-10 days to 45-60 days for digestion and 20-30 days to >150 days for evaporators.

In the only published report from a plant using MAX HT [12], conclusions based on a short initial test were that "...the effects of scaling were reduced by a factor of 10 from 4 to 5% per day down to 0.5% per day." Calculated reductions in energy savings and alumina and soda losses were given.

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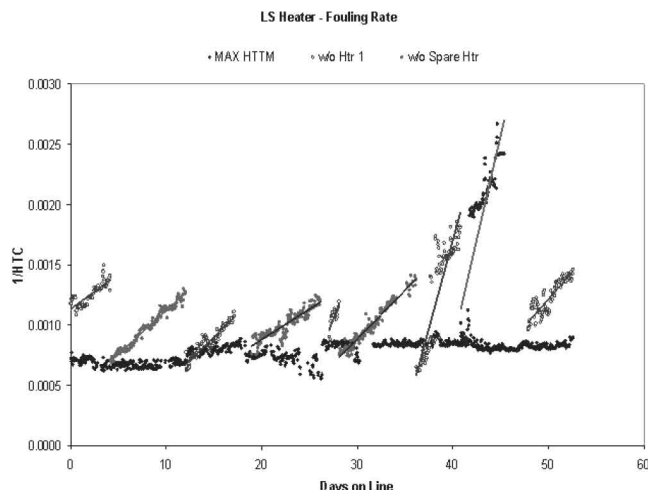


Figure 11. Heater fouling rates with cleaning every 5-6 days without antiscalant, vs. no fouling with MAX HT even after 53 days.

With use of MAX HT, reactive silica in the bauxite, control of pre-desilication time and temperature, heater cleaning, scheduling of manpower for cleaning and maintenance become less crucial in control and sustaining operation of the plant. As a result, plants have realized higher average liquor and steam flows, more stable operation and higher production.

Conclusions

1. MAX HT reagent is not used up, so it can be added early.
2. MAX HT can be effectively used on evaporators as well as on digester heaters.
3. Adsorption properties can be varied.
4. Scaling problems are directly related to liquor chemistry.
5. MAX HT removes constraints on plant operation, e.g., bauxites that were avoided in the past because of silica problems could be used.
6. Small amounts of solids interfere with MAX HT, but a new MAX HT 500 is less affected by solids.
7. Proper dilution and mixing of MAX HT are essential
8. Effective dosages are generally 20-40 ppm.

Ultimately, MAX HT has resulted in lower production costs in the plants at which it has been used.

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