

# Mineral scale management, Part 1. Case studies

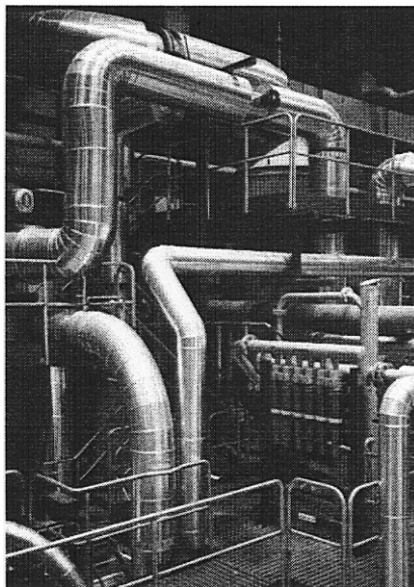
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**ABSTRACT:** Mineral scale increases operating costs, extends downtime, and increases maintenance requirements. This paper presents several successful case studies detailing how mills have eliminated scale. Cases presented include calcium carbonate scale in a white liquor strainer, calcium oxalate scale in the D0 stage of the bleach plant, enzymatic treatment of brown stock to eliminate oxalate scale, and control of barium sulfate scale.

**Application:** These case studies show how specific types of scale can be eliminated with improved process control and minor process changes.

Over the course of a single day of operation, thousands of kilograms of calcium pass through a pulp mill and bleach plant. If process conditions are not properly maintained throughout the entire pulping and bleaching process, relatively small amounts of these materials may precipitate onto various surfaces, resulting in upsets in operations and increased costs. Ironically, minor adjustments in operating conditions are often all that is required to substantially reduce or eliminate scale formation. It is possible to predict the process changes that are needed if one has a good understanding of the fundamental chemistry behind mineral scale formation. The ability to predict scale formation and identify operating conditions needed to eliminate the scale has been documented in at least one study.[1]

In addition to understanding the fundamentals of mineral scale formation, it is important to know the type of scale that is forming in various places. Typically, scale formation in the pulp mill area is calcium carbonate or lime scale. Scale formation in the acid stages of the bleach plant can be either calcium oxalate or barium sulfate. Under specific circumstances, lime scale can form on parts of acid stage equipment. Scale formation in the caustic extraction stages is typically lime scale. This report examines various case studies of scale formation and elimination from the digester through the bleach plant. In all of



**Chemithermomechanical pulp plant.**

these examples, the fundamental chemistry of why these process changes were successful is examined.

Most nonprocess elements enter the pulp mill with the wood [2]. About 2 tons per day of calcium enters the digester in this way. This calcium reacts with the white liquor to form calcium carbonate or lime scale [3]. The calcium precipitates on any readily available surface. By far the highest surface area present in the digester is the fiber. Thus, most of the lime scale forms on the fibers. Some specificity for heating surfaces exists [4]. The solubility of calcium carbonate decreases as temperature increases, and lime scale tends to form on liquor heaters

## CASE STUDIES

### *Calcium carbonate scale in a white liquor strainer*

Mill A was operating a continuous digester that had a frequent, reoccurring problem with lime scale forming in the white liquor strainer associated with the white liquor level tank. One of the strainers built up sufficient lime scale to require hydroblasting every 5 days. Extensive long-term maintenance costs resulted from the need to routinely isolate the scaled strainer, extract the internals, hydroblast the strainer to remove the scale, and put the system back together. Initially, the mill attempted to control the scale formation with commercially available crystallization inhibitors. The use of an antiscalant further increased the cost associated with scale formation, and the white liquor strainer still required hydroblasting every 5-7 days.

Normally, no chemicals are added to or mixed in the white liquor strainer. This ruled out a change in chemical concentrations as the cause of the scale. The solubility of calcium carbonate decreases with an increase in temperature, suggesting that heat intrusion might be occurring in the white liquor strainer. Careful observation of the strainer over several weeks showed that one side of the strainer was scaling faster than the other side. An examination of the strainer revealed a steam leak in the strainer housing on the side where the scale was forming most rapidly. Once the steam leak in the strainer was elimi-

steam leak in the strainer was eliminated, lime formation in the strainer stopped and the antiscaling chemical was eliminated. Understanding the inverse solubility of calcium carbonate was essential to determining why the scale was forming in this strainer and how to eliminate the problem.

### **Lime scale on caustic extraction stage washer**

Mill B routinely experienced lime scale formation on the face wire in the first caustic extraction stage. The scale started forming shortly after the mill migrated from a chlorine-based bleaching sequence to an elemental chlorine free (ECF) bleaching sequence using chlorine dioxide. When Mill B converted to chlorine dioxide bleaching, it also introduced DO stage filtrate recycle. The terminal pH of the chlorine bleaching stage had been maintained in the 2.1–2.3 range, but when the mill converted to chlorine dioxide, the pH of the first bleaching stage increased to 3.2. Fresh caustic for the first caustic extraction stage was applied to the pulp by a spray bar directed into the repulper of the D0 stage. In addition to fresh caustic, blow-down from the bleach plant scrubber was added to the D0 repulper. The caustic source for the bleach plant scrubber was white liquor.

One unintended result of the ECF conversion was that the calcium concentration in the caustic extraction stage increased substantially. The higher terminal pH of the first stage decreased the amount of calcium removed in the first bleaching stage effluent. D0 filtrate recycle also contributed to an increase in nonprocess elements (NPE) of the stock by reducing the volume of filtrate sewerer from this stage.

One of the features of wood pulp that fosters mineral scale problems is the ion exchange behavior of the acid-functional groups [5,6]. Electroneutrality requires pulp to collect enough cations to balance or neutralize the charged groups [7]. Below a pH of about 4, protons provide most of this charge balance; above pH 4, cations in solution must be absorbed by the pulp to maintain the charge neutrality of the fiber. In the tradition-

al chlorine-initiated bleach plant, the first stage is operated at a pH near 2.0, and 90% or more of the trace metals are freed from the fibers and available to be washed from the pulp [6,89]. Whereas the ideal pH for chlorine is about 1.5, the ideal pH for chlorine dioxide is typically closer to 4. At this pH, 50% of the trace metals are still "bound" to pulp [6,8,9] and will be carried further into the bleach plant. Typical limits on washing efficiency increase this value significantly.

The results of these changes were a substantial increase in the calcium

charge of the phenolic sites on the pulp. This could be about a 50% reduction in bound metals, but in an extraction stage the hulk of the bound metals consist of sodium. As stated in the introduction, the calcium in brownstock is calcium carbonate. Precipitated calcium carbonate (PCC) on a paper machine becomes unstable when the pH drops below 7. Likewise, some PCC in the pulp will dissolve and wash from the pulp in the final washer. Although this calcium probably precipitates again in the first or second stage washers, the

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content of the pulp entering the extraction stage of the bleach plant. The increased calcium content, in conjunction with substantial carbonate levels in the caustic and scrubber blow-down, resulted in scale formation on the extraction stage washer wire.

This bleach stage was not modeled, but the circumstances are consistent with a case we had evaluated [10]. The scale in that case can be attributed to an ion exchange chromatography effect that increases the concentrations of cations and anions in the bottom layers of a washer mat. However, this understanding is not critical to the resolution of the scale problem (see the cited research for a detailed explanation).

Mill B was in the process of installing an enzyme-bleaching stage between the third and fourth stages of brownstock washing. Enzyme bleaching required near-neutral conditions. The pulp pH was lowered to about 6.5 by mixing sulfuric acid into the water applied to the third-stage repulper. Enzymes were also added into the repulper water. The stock was held in a tower for at least 15 min, then subjected to a fourth stage of brownstock washing.

Lowering the brownstock pH to 6.5 freed all cations balancing the

reprecipitated calcium carbonate often remains suspended in solution and is more effectively washed from the pulp than is the lime that forms in the digester where it often forms inside the fibers.

When an enzyme-bleaching stage with brownstock pH control was initiated, the sodium content of the bleach plant acid sewer dropped from about 16,000 mg/L sodium to an average of about 2500 mg/L sodium, with no significant change in effluent volume. Calcium was reduced enough that lime scale no longer formed on the extraction stage washer, and a small amount dissolved and disappeared from the washer.

### **Calcium oxalate scale in D0 bleach stage**

Lime scale is not the only nonprocess element mineral scale that causes problems in an ECF bleach plant. Calcium oxalate formation has also been documented to cause extensive lost time and increased operating costs. Once calcium is dissolved, it is capable of reacting with oxalic acid to form calcium oxalate and precipitate onto process equipment. Oxalic acid is found in wood and also forms in the pulping process. But the conditions in the digester strongly favor calcium carbonate formation [3] and the

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be removed by good brownstock washing. Most of the oxalate in the bleach plant is a by-product of the bleaching chemistry. All oxidizing stages generate oxalic acid, typically about 250-500 g/metric ton of brownstock pulp [11,12]. With filtrate recycle and technical washing conditions, this provides for an oxalate concentration in bleach plant filtrate in the range of 35-90 mg of oxalate/kg of material (filtrate and pulp) in the first stage of bleaching. Filtrate recycle can increase the concentration of calcium to as much as 3000 mg/kg in hardwood pulp.

A mill with a low oxalate concentration (10-20 mg/L) has the potential to encounter scale problems when the pH in the first chlorine dioxide stage is above about 3.5. In a more typical mill, where oxalate concentration is around 50-100 mg/L [13], the mill can still reduce oxalate scale by lowering the pH to about 2.5. For a mill experiencing calcium oxalate scale formation, simply lowering the pH of the bleaching stage may eliminate or substantially reduce the scale.

Another effect makes it more difficult to eliminate calcium oxalate scale by simply lowering the target pH. Calcium precipitates with oxalate in three crystal morphologies that differ in the number of water molecules included in the crystal. Under most bleaching conditions, the preferred form is the monohydrate, but when the pH is below 4, the less soluble dihydrate also begins to form [14]. This effect increases the amount of calcium oxalate that precipitates between pH 2.5 and pH 4, but it has little effect on the pH at which scale problems should cease.

Mill C started experiencing calcium oxalate scale formation about the time it converted to ECF bleaching. The scale was prevalent enough to extend the length of scheduled bleach plant outages by a couple of hours every eight weeks. As oxalate scale formed on the washers during the eight weeks between acid washes, the washing performance deteriorated and bleaching costs increased. After the scale was removed from the washer, bleaching costs would drop, then slowly climb again. The oxalate scale was responsible for increased operat-

ing costs and downtime.

When Mill C originally converted from chlorine-based bleaching to ECF bleaching, no adjustments were made to pH. The natural terminal pH of the D0 stage was about 3.0-3.4. In this pH range, about 10% of the oxalate is still in the divalent anion form. By reducing the pH to 2.5, the divalent form can be reduced to 2% or less. The mill piped a sulfuric acid line to the blend chest where brownstock pulp was diluted and fed to a chlorine dioxide mixer. A simple feedback control system and pH probe were installed approximately 30 s downstream of the chemical mixer. Manual tests were conducted to correlate the pH of the stock 30 s after chlorine dioxide addition to the terminal pH of the D0 stage. Simple proportional feedback control was used, maintaining the sulfuric acid flow to keep the D0 stage terminal pH in the 2.5-2.8 range. Calcium oxalate scale formation on the D0 washer was eliminated.

## ***Oxalate scale in a bleach plant rebuild***

Mill D had been experiencing exceptional levels of calcium oxalate scaling over the course of two years, again associated with conversion to ECF bleaching. Stock lines were depositing scale several centimeters thick, and the D0 washer was routinely being taken out of service to be cleaned. Bleach plant operating costs were exceptionally high because of excessive carry-over between washing stages. Every time the mill shut down or experienced large production swings in the bleach plant, scale would crack off the internal piping and send small particles of calcium oxalate to the paper machine for several hours.

Mill D attempted to implement a pH control scheme similar to the one described for Mill C, resulting in a significant decrease in the amount of scale formed in the piping system. The oxalate scale associated with the washer did not decrease, however, and the amount of lime scale in the extraction stage increased. Except for the reduction in calcium oxalate scale formation in the D0 piping system, the attempt to control scale formation by pH control was not successful.

Mill D used atmospheric diffusion

washers in the bleach plant. Filtrate obtained from a washing stage following the caustic extraction stage was used as wash water in the D0 stage. The pH of this wash water WAS slightly alkaline. Pulp washing was inefficient in this bleach plant. To simulate mill bleaching conditions in the laboratory, the D0-EOP bleach sequence required at least 45%-55% filtrate carryover from the D0 stage to the EOP stage. The poor washing conditions in this bleach plant and high wash water pH were responsible for the failure of pH control to substantially reduce or eliminate calcium oxalate scale formation. Even though the mill eliminated oxalate scale formation in the D0 stage piping and tower, the alkalinity of the wash water, in conjunction with the inability to remove the dissolved calcium in the washer, resulted in extensive oxalate scale formation. The calcium that had been scaling onto the D0 piping system was simply carried through the D0 stage and formed calcium carbonate in the extraction stage. To eliminate the excessive downtime associated with hydroblasting scale off of the atmospheric diffusion washers, and to substantially reduce the operating cost of the bleach plant, the mill decided to replace the D0 and EOP stage washers with twin-roll presses.

Shortly before the mill started engineering the press retrofit project for the hardwood bleach plant, it had been engaged in a project to model the nonprocess element behavior of the hardwood bleach plant. A WinGEMS model of the mill was connected through dynamic data exchange to an Excel equilibrium calculation developed at the Institute of Paper Science and Technology (Atlanta, Georgia, USA). The operating bleach plant and the model were in agreement. Details of the model development, calibration, and performance are documented in the literature [1]. The mill was interested in predicting the effect on the nonprocess elements and NPE scale formation of replacing the D0 and EOP stage washers with presses. The existing washer blocks were replaced in the model with split-blocks that split off filtrate to model the press discharge consistency of 30%. Laboratory work, in con-

junction with vendor recommendations, was used to determine the initial operating conditions of the new bleach plant. These were used in the NPE model for the mill. The model results suggested the mill would be operating at a risk for calcium oxalate scale formation under their planned start-up conditions, but that controlling the D0 pH to 2.5 would be sufficient to eliminate the calcium oxalate scale.

As the mill had previously been unsuccessful with pH control as a method of controlling scale formation, the initial suggestion was discarded, and the mill used the initial start-up conditions suggested by the project vendor. Excellent chemical reduction and good washing were obtained immediately upon start-up. Then, calcium oxalate scale began to form on the D0 press rolls. After a couple of months of operation, the mill had to hydroblast the press rolls to remove the oxalate scale. The mill then initiated pH control and scheduled a second maintenance outage for eight weeks out. When the press was opened up for hydroblasting, no scale was found. Good washing and pH control were sufficient to eliminate calcium oxalate scale formation in this bleach plant. The mill data collected during this period are used in the equilibrium calculation presented in the third paper of this series [15].

### ***Calcium oxalate scale in piping to a caustic extraction stage tower***

Mill E was experiencing extensive calcium oxalate scaling in the piping and medium consistency (MC) pump going to the first caustic extraction stage bleaching tower. The pump needed to be hydroblasted every few weeks, resulting in excessive downtime and maintenance. The mill was operating the D0 stage within the desired terminal pH range of 2.5–2.8, and it did not have problems with oxalate scale on the washer. The wash water being applied to the D0 stage was a combination of D1 filtrate and paper machine whitewater. The paper machine operated at pH 5, and the whitewater contained a fairly high calcium concentration. Samples of the

pulp leaving the repulper of the D0 stage had a pH between 3.5 and 4.0. Oxalic acid that had not been washed out in the first chlorine dioxide stage was reacting with dissolved calcium in the paper machine whitewater and in the D1 filtrate to form calcium oxalate scale.

The mill added caustic for the extraction stage at the suction of the MC pulp. This convened all the oxalic acid that remained after D0 washing into the active divalent anion, and scale growth in the pump was rapid. The mill was not able to alter wash

problem (calcium oxalate) for a worse scale problem (barium sulfate). The barium sulfate was forming in the weave of the D0 washer face wire. The face wire developed a shiny finish and the scale sealed the wire, drastically impeding production and increasing bleach plant operating costs.

The barium sulfate could not be removed with a simple acid wash. The washer required hydroblasting with a caustic detergent and chelant, adding substantial cost to the operation. Faced with the prospect of dealing with barium sulfate with the pH con-

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water sources or improve their existing washing capabilities. The only remaining option was to relocate the caustic addition source. The mill decided to move caustic addition from the MC pump to the repulper of the D0 stage. They understood that this change might just move the scale formation to the D0 repulper, but this area was at least easier to maintain. The hope was that by adding the caustic through a shower bar across the entire length of the washer repulper, most of any precipitate would collect on the material with the highest amount of surface area, the pulp fibers themselves. To date, the mill has not experienced any significant scaling problems in the D0 repulper, and the oxalate scale formation in the piping and in the bowl of the MC pump has not reappeared.

### ***Barium sulfate scale on the D0 washer face wire***

Mill F was having difficulties with calcium oxalate scale formation on the face wire of the D0 washer. Shortly after another mill explained their success in scale control by regulating the terminal pH of the D0 stage, Mill F decided to try the same thing. After several months of operation, the mill decided to stop using pH control because it had simply traded one scale

trol system or working with the more manageable calcium oxalate scale, the mill abandoned pH control of the D0 stage.

The mill had been controlling the D0 stage terminal pH to 2.0–2.5. If a little was good, more was better, and the mill attempted to operate at the lower end of the pH range to ensure elimination of the calcium oxalate scale. Frequently, the terminal pH of the D0 stage would swing as low as 1.9. By adding enough sulfuric acid to drive the pH into this lower range, the mill was establishing excellent conditions for the formation of barium sulfate scale. Several fairly subtle effects also occurred on washers to increase the probability of scale formation. The second pKa for sulfuric acid is about 1.9, so at this pH, half the acid is dissociated to the sulfate dianion and half is still associated as the bisulfate monoanion. The D1 wash water at the higher pH of 3.9 shifts this equilibrium to produce more sulfate. Secondly, both the pKa and the solubility product are temperature sensitive. Lowering the temperature decreases the solubility and increases the acidity, moving the acid-base equilibrium to produce more sulfate and the precipitation equilibrium to favor precipitation. The high sulfate concentration and shift in equilibria associated with

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a cooler and higher pH wash water was enough to initiate precipitation of barium sulfate on the washer.

Eventually, Mill F reimplemented pH control for the D0 stage with a significantly higher target and tighter range. The mill now operates its pH control in the range of 2.4–2.8, with a target of pH 2.6. With this operating

raise the pH of the D0 stage slightly, with no effect. Finally, they replaced the spent generator acid with sulfuric acid, which solved the barium sulfate scaling problem.

The second paper in this series, on fundamental chemistry, provides a calculation of the sulfate generated in pH adjustment using sulfuric acid and

ful solution in one mill may not work at another because of variations in process conditions and equipment layout. It is always important to understand exactly what type of scale is in question. Just because scale is forming in an extraction stage does not mean the scale that is forming is lime scale. As one case study demonstrated, it is possible for calcium oxalate to form in portions of the extraction stage.

By studying and understanding the fundamental chemistry of mineral scale formation, mills can minimize the effects of NPEs on the bleach plant. In some cases, it may not be possible to eliminate scale formation without radically altering the bleach plant water sources or operating conditions, but modeling and first principle predictions may be used to minimize the number of changes required to obtain minimal scale formation.

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## If a mill has any amount of barium entering with the wood basket, it should avoid using spent generator acid for pH control.

target, the mill has been able to eliminate the calcium oxalate scale and avoid the barium sulfate scale encountered at the lower pH.

### **Sodium sesquisulfate for pH control in D0 stage**

Mills G and H decided to reduce bleaching costs by substituting chlorine dioxide generator-spent acid for the sulfuric acid normally used in pH control of the D0 stage. These mills operate an Erco R8 process to produce chlorine dioxide. Another mill, operating an Eka SVP process, had similar cost-reduction thoughts. Both of these types of generators produce an acid salt by-product, a bi-salt of sodium sulfate and sodium bisulfate known as sodium sesquisulfate. The approximate chemical formula for this generator by-product is  $\text{Na}_3\text{H}(\text{SO}_4)_2$ . The three mills that used this spent acid for pH control in the bleach plant developed recurring problems with D0 stage face-wire plugging. The face wire of the D0 bleaching stage developed a shiny appearance and stopped draining, similar to the deposits described for Mill F. The drainage could be re-established by hydroblasting the face wire with a detergent/chelant material.

Using experience gained at other mills, we evaluated the pH control system used for the D0 stage. The mills were found to have been operating in a pH range that was considered “safe” for barium sulfate scale. After several months of difficulties, the mills finally got a sample of the scale material large enough for analysis and found it to be barium sulfate. The mills tried to

sodium sesquisulfate. By using spent generator acid for pH control, the mills increased the sulfate ion concentration in the bleaching stage by a factor of four. When using sulfuric acid, the mills add half a sulfate ion for each proton added. When spent generator acid is used, two sulfate ions are added for each proton. This fourfold increase in sulfate ion often results in barium sulfate scale, even at higher pH values. In general, if a mill has any amount of barium entering with the wood basket, it should avoid using spent generator acid for pH control.

### **CONCLUSIONS**

This report has presented several mill case studies in which some form of nonprocess element mineral scale resulted in increased operating costs, extended downtime, and substantially increased maintenance requirements. Mineral scale can form in the digesters, chlorine dioxide stages, and caustic extraction stages. Typical forms of scale include calcium carbonate or lime scale, calcium oxalate, and barium sulfate. In all cases, an understanding of the fundamental principles of scale formation helped minimize or eliminate the scale. In one reported case study it was possible to apply first principle understanding to a material and energy balance model. The mill could then predict that proposed operating conditions would cause scale and develop alternate conditions that would be relatively scale-free.

The fundamental aspects associated with scale formation and success

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### INSIGHTS FROM THE AUTHORS

The substantial increase in mineral scale problems resulting from changing to ECF bleaching has resulted in extensive cost increases and increased unscheduled downtime. These problems must be mitigated in order for mills to maintain profitability.

The ion exchange equilibrium model was developed as a Masters laboratory bleaching project at IPST (presented at the 1999 TAPPI Pulping Conference), segued into a mill WinGEMS analysis (presented at the 2003 TAPPI Fall Conference), and that became the basis for these papers on Mineral Scale Management.

Scale formation frequently results from subtle changes in process chemistry; that presented the most difficult aspect of this work. Small process details at each mill location result in different types and amounts of scale. In addition, mills with scale problems often have scale buildup in several locations. Researchers must focus on one problem at a time

Mineral scale increases the operating cost of a mill. It also results in an increased frequency of scheduled shutdowns, unscheduled shutdowns and lengthens planned shutdowns resulting in poor availability. The cumulative cost is very high;

thus, making process changes that remove scale as a periodic maintenance problem is sound management. We were glad to discover that we do know enough about the fundamental chemistry, and about how mills work, to be successful using sound chemistry and engineering to solve these problems.

The next step is a detailed equilibrium-mass and energy balance model to better understand methods for managing barium sulfate scale. Our second paper in this series (scheduled for publication in the July, 2006 issue of TAPPI JOURNAL) presents the solubility product equilibria for calcium carbonate, calcium oxalate and barium sulfate, and the acid equilibria for the precipitating anions.



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