

IMPROVED PRE-PASSIVATION OF MILD STEEL HEAT EXCHANGER TUBES

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Abstract

Unpassivated mild steel exposed to high temperature Bayer liquor can suffer catastrophic corrosion damage in relatively short periods. The formation of a durable passivation layer is therefore a key process in ensuring long heat exchanger tube life.

Based on accumulated observations and field data, a pre-passivation process was adopted at QAL in which mild steel heater tubes are exposed to low flows of low temperature spent liquor for a period of up to 12 hours.

In conjunction with a chemical supplier, a new acid inhibitor was developed which in laboratory testing showed a significantly reduced corrosion rate when acid cleaned metal coupons were subsequently exposed to hot synthetic Bayer spent liquor.

A series of plant trials have been undertaken to validate the laboratory results under plant operating conditions to determine whether a reduction in pre-passivation times could be implemented.

1 Introduction

Corrosion in heat exchanger tubes is an important issue for many process industries. At Queensland Alumina Limited (QAL), heat exchangers with mild steel tubes are used as part of the digestion process. These tubes are exposed to environments ranging from pH 1 to pH 14. In these extreme environments high corrosion is expected and finding suitable methods to substantially reduce corrosion are extremely important.

During digestion scale builds up within the tubes resulting in reduced heat transfer efficiency. To remove this scale, the tubes are periodically cleaned using sulphuric acid. It is normal practice to mix a small amount of corrosion inhibitor with the acid to reduce corrosion damage to the mild steel by the acid.

It is well known that, although mild steel should corrode rapidly when in contact with caustic, an oxide film develops that protects the steel from further attack by the caustic (Giddey et al., 1996; Lu et al., 2000; Wang and Stack, 2000; Giddey et al., 2001; Lu et al., 2001; Rihan, 2001; Gavril et al., 2003). An undesirable side effect of the acid clean is that it not only removes the scale but also any protective oxide film that may have formed during the previous liquor cycle. The result is bare, unprotected steel exposed to liquor (caustic) at high temperatures and flow rates when the liquor cycle commences. When the liquor is brought on line under these circumstances very high corrosion rates are expected until a stable, protective oxide film forms. This period of high corrosion is the so-called "liquor transient".

The growth and stability of the oxide layer depends on many factors. The main ones are: temperature, flow rate, available oxygen, and liquor chemistry (Giddey et al., 1996; Lu et al., 2000; Wang and Stack, 2000; Giddey et al., 2001; Lu et al., 2001; Rihan, 2001; Gavril et al., 2003). As the film forms there are competing processes that slow down its formation. In particular, some of the film may dissolve or be eroded away by the grinding action of particulates within the liquor (Giddey et al., 1996; Efirid, 2000; Lu et al., 2000; Postlethwaite and Nescic, 2000; Wang and Stack, 2000; Giddey et al., 2001; Lu et al., 2001; Rihan, 2001; Gavril et al., 2003). Hence a fresh oxide film must be encouraged to rapidly form following an acid clean, prior to the liquor cycle reaching full temperature and flow rate. Without this protection, catastrophic damage may occur as the liquor is brought on line or, at the very least, the high corrosion rates increase maintenance costs.

In an effort to reduce corrosion problems before the liquor is brought completely on line, a pre-passivation stage was introduced.

Pre-passivation involves exposing the mild steel tubes to spent liquor at reduced flow and temperature over a period of about 12 hours to allow an oxide film to form. Experience at the plant has shown that although pre-passivation may not eliminate the risk of initial high corrosion rates occurring, it is expected to reduce the problem. The main challenge is to reduce the transient or eliminate it altogether, since this is when catastrophic corrosion damage is most likely to occur. An ancillary challenge is to also reduce the pre-passivation period.

The sequence of events for a typical digestion liquor cycle described above can be summarised as follows.

1. The tubes are cleaned with acid to remove scale – the acid shot (AS).
2. The acid is then neutralised by flushing the tubes with diluted spent liquor, which is subsequently drained – the neutralising shot (NS).
3. The tubes may then be left standing for a period of time before liquor is introduced – the interim period (IP).
4. Liquor is introduced at reduced flow and temperature. This is the pre-passivation (PP) stage.
5. Lastly, the flow and temperature are brought up to plant operating levels and this is usually where the transient (T) is observed. A post transient (PT) period follows, where the corrosion rate stabilizes.

A schematic of the variation of corrosion rate during a low-pressure heater liquor cycle is shown in Figure 1. The peaks indicate periods of high corrosion. The main features are the peak corresponding to the acid/neutralising shot, the plateau corresponding to the pre-passivation stage followed by the transient due to full operating conditions being reached.

Ideally it is preferable to develop a solution that provides adequate protection as the liquor is being brought on line. However, the film formation process is complex and relies on a fine balance of the film forming at a faster rate than the rate at which it is removed by dissolution and/or erosion. One strategy for mitigating this problem, and the one that is explored in this paper, is the use of an inhibitor that forms a protective monolayer to prevent corrosion as the liquor comes on line. This treatment was designed to significantly reduce both the pre-passivation time and the magnitude of the transient.

Two inhibitors were compared in this study. Inhibitor A is currently used to protect the steel during the acid clean. It is not expected to provide any benefits following the acid clean. Inhibitor B is based on

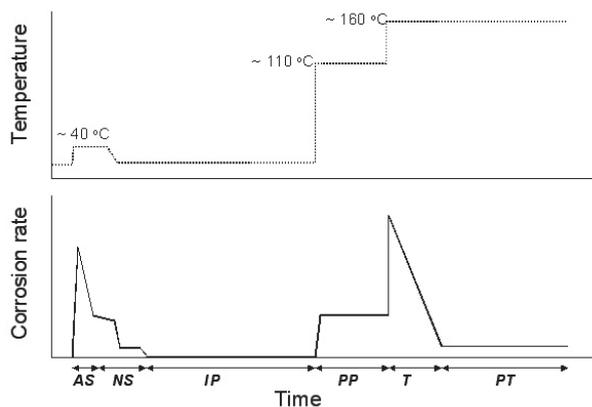


Figure 1 A schematic of typical corrosion behaviour and the corresponding variation in temperature during a liquor cycle. In practice the actual temperature profile depends on the heat exchanger position.
AS – Acid Shot; NS – Neutralising Shot; IP – Interim Period;
PP – Pre-passivation; T – Transient; PT – Post Transient.

Inhibitor A and contains additional components that are intended to provide protection following the acid shot when hot liquor contacts the steel tubes. Both inhibitors contain a mixture of anodic and cathodic inhibition agents and could be considered mixed inhibitors. In addition, Inhibitor B also contains high temperature amines that form a monolayer on the steel to prevent corrosion when the steel is exposed to Bayer liquor.

A laboratory comparison of Inhibitor A and the new, modified version (Inhibitor B) showed very promising results. As a result plant trials were undertaken to determine if this new inhibitor would perform as well under real process conditions. It was convenient to monitor the corrosion rate electrochemically and this is discussed in more detail in the next section.

2 Experimental

2.1 Electrochemical corrosion monitoring

Linear polarisation resistance (LPR) measurement, an electrochemical corrosion monitoring technique, was employed to evaluate the effectiveness of the inhibitors in the laboratory and plant. The LPR technique has the advantage that it is straightforward to implement and will not significantly affect the material (heater tube steel) being studied (Tait, 1994; ASTM Designation G 59–97, 2004).

It involves applying a small voltage perturbation to the test specimen and measuring the subsequent current flowing through the electrodes. Since the perturbation is small the relationship between voltage and current is assumed to be linear. A parameter known as the polarisation resistance (R_p) can then be calculated using Ohm's law,

$$E_{app} = IR_p$$

where E_{app} is the applied potential, I is the measured current and R_p the (polarisation) resistance. Corrosion rate is inversely proportional to the polarisation resistance and hence can be determined if required.

Since only relative corrosion rates were of interest in this study it was sufficient to leave the corrosion data in terms of R_p . In fact, conversion to actual metal loss (in mmpy) may be misleading, since the actual pipe corrosion is not being monitored directly, rather it is inferred from the corrosion of the electrode specimen. As will be seen shortly the geometry of the probes (in both the laboratory and plant studies) is significantly different to the tubes. The probes used in the plant are also positioned in a location where the flow is expected to be greater than for the tubes. Hence, it is the probe corrosion that is being measured, not the tube corrosion.

2.2 Laboratory evaluation

Preliminary laboratory tests, using a rotating cylinder electrode, were commissioned from the Department of Chemical Engineering

at Monash University. This information provided an indication of the effectiveness of the new inhibitor when subject to typical flow and temperature regimes found in the plant. Monash University is equipped with a dual autoclave rotating cylinder electrode system that enables electrochemical measurement to be conducted at the high temperatures and pressures found at the plant. The rotating cylinder arrangement provides a method of simulating plant flow conditions (Darby et al., 1999; Roberge, 2004). The cylinder electrode was rotated at a speed of 730 rpm during all of the laboratory measurements.

Two test procedures were implemented to measure the corrosion rates of mild steel tube exposed to both acid and Bayer liquor environments. The first involved measuring the corrosion of the steel specimen in 6% vol/vol sulphuric acid mixed with a small volume fraction of inhibitor added (0.1% or 0.2% vol/vol). This test was conducted at 60°C and the corrosion rate was determined by iron solution analysis. The second procedure was used to measure the corrosion of the steel specimen in synthetic Bayer liquor. LPR measurements were used to monitor corrosion rate of the steel specimen in liquor. The procedure described by Monash University for the Bayer liquor measurements involved:

1. Washing the specimen with 6% vol/vol sulphuric acid, containing the desired volume of inhibitor, for one hour at 60°C.
2. Rinsing the specimen with 1 M NaOH for 5 minutes (to neutralise the acid) at 60°C.
3. Preheating the electrode to 100°C in a nitrogen environment.
4. Introducing synthetic Bayer liquor into the autoclave at 130°C.
5. Increasing the temperature gradually to 160°C and waiting 30 minutes before an LPR measurement is recorded.

Following preliminary plant trials, differences between the plant and laboratory results were observed. As a result, a number of scenarios were also provided to Monash University to determine whether plant procedures might adversely affect the effectiveness of the new inhibitor. The prescribed procedures were as follows and used 1 M NaOH to simulate dilute Bayer liquor:

1. Perform tests as per the normal procedure (outlined above).
2. Remove the neutralising stage from the procedure.
3. Remove the neutralising and pre-passivation stage from the procedure.

2.3 Plant trials

Alcan developed the LPR probe and instrumentation configuration used for the plant trials (Newton et al., 1998). The LPR specimen probe consisted of five rings positioned along the length of the probe. Four of the rings were constructed from the heat exchanger tube steel of interest and acted as working electrodes. A centrally located 5th ring, made from monel, was used as a counter electrode. All the rings were electrically isolated from one another by Teflon® spacers. This probe also housed a thermocouple wire to monitor the temperature of liquid flowing past the electrodes.

A Hg/HgO reference electrode patented and supplied by Alcan was housed in a separate probe. The body of the reference electrode probe was used as a pseudo reference electrode for the LPR measurement. A simultaneous measurement of the potential of the pseudo reference electrode with respect to the Hg/HgO potential was recorded independently using a data logger.

The electrochemical measurement was controlled by a potentiostat configured for three electrode measurements. The mild steel specimen rings (individual working electrodes) were connected to the potentiostat via a multiplexer. The multiplexer enabled each of the four specimen rings to be selected in sequence so that LPR measurements on individual rings could be made. A schematic of the measurement system is provided in Figure 2.

The probes were positioned in the process stream by inserting them into a pipe connecting two of the low-pressure heaters. The location of the probe between heaters and the geometry of the probe arrangement in the pipe is shown in Figure 3. Note that a third, mass loss, probe is also shown.

The mild steel rings were simply off cuts of new heater tube, and apart from mounting them on the probe no further preparation was performed. This was thought to be reasonable since the normal acid

cleaning procedure would provide a fresh surface to study. Fresh probes were used for each trial (acid shot liquor cycle combination) so that additional, history dependent effects were not introduced into the measurement. The field data comprises 6 baseline trials, using Inhibitor A, and 3 trials using Inhibitor B conducted over a 6-month period.

Initially, LPR measurements were taken at 30-minute intervals; however later measurements used 15-minute intervals to increase the data density. A single LPR measurement actually consisted of four LPR measurements performed in sequence. As a result, by combining the data it was possible to achieve a maximum effective sampling rate of approximately one measurement per three minutes. This data was automatically logged throughout the duration of the trial by a personal computer connected to the electrochemical instruments.

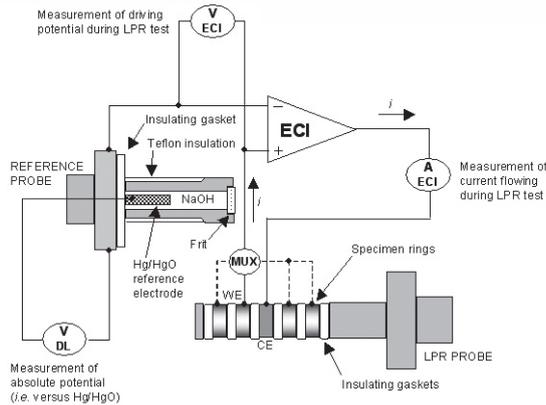


Figure 2 A schematic of the LPR monitoring system employed by QAL (Newton et al., 1998). The main features of this arrangement are: the two recording instruments – a potentiostat or electrochemical interface (ECI) and a data logger (DL); connection between the specimen rings (shaded rectangles) and the potentiostat via a multiplexer (MUX); and two reference electrodes – a Hg/HgO cell and the monel electrode housing that provides the pseudo reference for all potential measurements. The dashed lines indicate the other potentiostat-multiplexer connections. (Reprinted with permission from Alcan International)

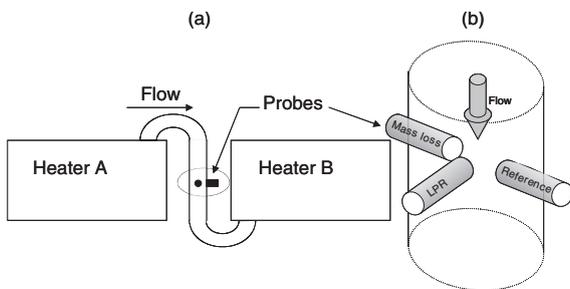


Figure 3 The relative positions of the monitoring probes inserted into the pipe connecting two heater exchangers. (a) Shows the location between two heaters and (b) indicates the probe arrangement in the pipe. All of the probes are located on the same plane normal to the downward flow direction as indicated by the arrow.

3 Results and discussion

3.1 Laboratory evaluation

A summary of the results for the laboratory tests is provided in Figure 4. The original results (Ao – Inhibitor A and Bo – Inhibitor B) were certainly encouraging and indicated a substantial improvement in protection of the mild steel under simulated process conditions. However, after noticing considerable variability in the plant data (discussed later), subsequent tests were commissioned. It is clear from the test results for each of the different batches that the variability in performance noted in the plant was also reflected in the laboratory. It is also interesting to note that the percentage error in the acid test results

was approximately 12%, indicating that Inhibitor B performs reasonably consistently in an acid environment. However, as can be seen from inspection of the data, the error in the liquor results ranges from 50% up to 85% if individual batches are considered. If all of the data for the tests in liquor are combined (for Inhibitor B) the percentage error is approximately 35%.

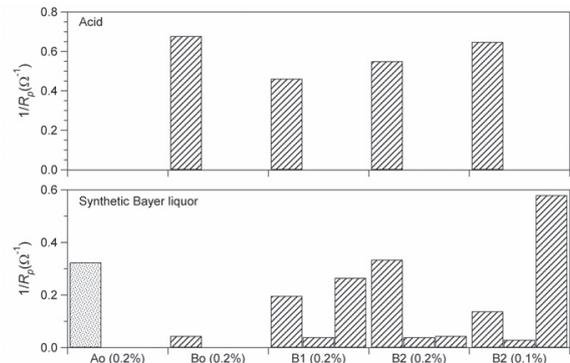


Figure 4 The laboratory results for the corrosion of mild steel specimens exposed to 6% vol/vol sulphuric acid only (top graph) and synthetic Bayer liquor (lower graph). Ao and Bo are the original test results for Inhibitor A (speckled pattern) and Inhibitor B (diagonal lines) respectively. The numbers 1 and 2 following the letter B indicate batch numbers for subsequent tests using Inhibitor B. The percentage of inhibitor (vol/vol) added to the sulphuric acid is given in the brackets.

3.2 Plant trials

The results of a typical LPR measurement within the plant are shown in Figure 5, where $1/R_p$, which is proportional to the corrosion rate, is measured as a function of time. The first peak corresponds to the acid and neutralising shot whilst the plateau followed by a peak is a result of introducing the liquor. The plateau corresponds to the pre-passivation procedure and the peak to the liquor transient once the liquor cycle is at full operating conditions. Once the transient has decayed the corrosion rate remains low and relatively constant until the liquor cycle has completed.

Figure 6 summarises all of the field trial data in terms of the average corrosion rate per event per trial and the corresponding duration per event per trial. To generate this data each corrosion event (i.e. during the acid shot, pre-passivation period, liquor transient and post transient) in each trial was analysed to determine the total corrosion resulting from the event. Total corrosion is calculated by integrating the area under the $1/R_p$ as a function of time curve. By dividing the total corrosion per event by the event duration an average corrosion rate for a particular event can be determined.

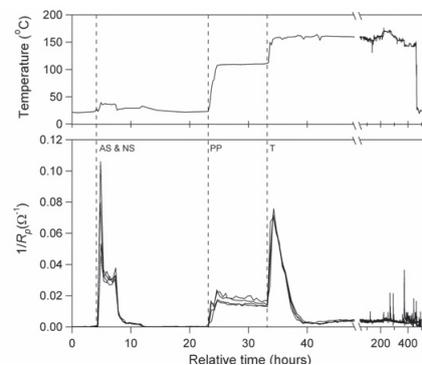


Figure 5 An example set of corrosion rate ($1/R_p$) data for trial 1 (lower graph). The vertical dashed lines indicate the start of the acid/neutralising shot (AS & AN), pre-passivation (PP) stage and acid/liquor transient (T). The upper graph provides the corresponding temperature profile.

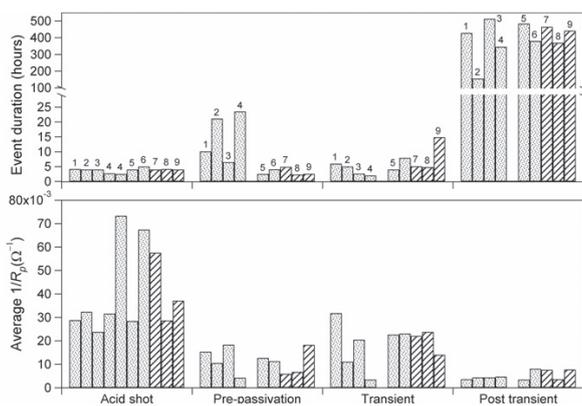


Figure 6 A comparison of the performance of Inhibitor A (speckled) and Inhibitor B (diagonal line) for each individual trial. The lower graph summarises the average corrosion rate (in terms of $1/R_p$) for each trial as a function of liquor cycle event. The corresponding event durations for each trial are provided in the top graph. Numbers above each duration bar are provided to identify the trials (1 to 6 are trials using Inhibitor A and 7 to 9 are trials using Inhibitor B). Note that trial number 4 consisted of a double acid shot, hence the gap between 4 and 5 for the remaining events.

Average corrosion rate on its own is not particularly helpful when trying to compare the two inhibitors in the plant since event times and average corrosion rates vary considerably between trials as indicated in Figure 6. This is a consequence of the variable nature of plant trials as opposed to laboratory test where conditions can be more carefully controlled. Hence, any criteria developed to describe the performance of an inhibitor in the plant environment should take into account the variability of the plant process. To address this problem the event data can be averaged across all trials associated with a particular inhibitor. The result is a numerical figure that encapsulates the average overall behaviour of each of the inhibitors.

To arrive at a suitable numerical figure it is desirable to combine the time per event and the corrosion rate (either the average or overall corrosion rate). As a result this figure can be expressed in a number of ways (e.g. as an average total corrosion or time weighted average corrosion). The average total corrosion per event for each of the inhibitors was used here since it provides some indication of the relative amounts of corrosion expected during different stages of the liquor cycle. It is calculated by multiplying the average event time by the average corrosion rate for that event. For example, the average event time of an acid shot using Inhibitor A was calculated by averaging all of the acid shot durations of trials using Inhibitor A. Likewise, an average corrosion rate for the corresponding corrosion event was also calculated. The two results were then multiplied together. The resultant data set is presented in Figure 7.

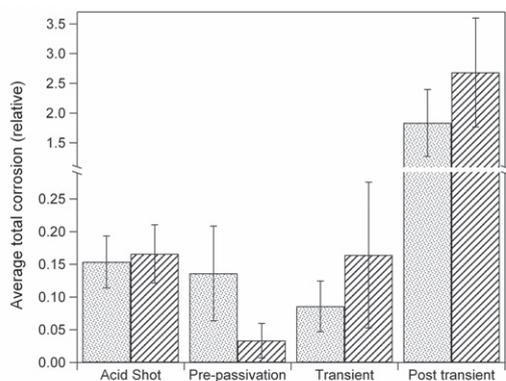


Figure 7 The average total corrosion as a function of corrosion events for Inhibitor A (speckled pattern) and Inhibitor B (diagonal line). Error bars are presented as the standard error.

The data as presented in Figure 7 gives a good indication of the overall performance of each of the inhibitors. Both inhibitors have similar total corrosion during the acid clean. This is to be expected since the chemistry of Inhibitor B is based upon Inhibitor A. Inhibitor B outperforms inhibitor A during pre-passivation. Again this is to be expected for two reasons. The first is a consequence of the pre-passivation times being shorter when Inhibitor B was used. Secondly, the average corrosion rates for trials using Inhibitor B appear to be lower than for trials using Inhibitor A as indicated by Figure 6. The latter is probably due to the monolayer protective film provided by Inhibitor B preventing corrosion in the pre-passivation Bayer liquor.

However, following pre-passivation, liquor cycles that used Inhibitor B displayed considerable variability. It was this variability that prompted commissioning of further laboratory tests. As noted earlier these tests also confirmed the variable nature of corrosion rates when Inhibitor B is employed. Although the data indicates steel treated with Inhibitor B suffers from more corrosion, the statistics for this are limited and the variability is large. A more conservative conclusion is that Inhibitor B provided no significant benefit over Inhibitor A in terms of overall corrosion during the transient. The variability is interesting since it may indicate a lack of robustness of the monolayer when plant conditions are not ideal. From a practical view point this is significant since minimisation of corrosion damage is required under the best and worst case scenarios.

There are a number of possible reasons for Inhibitor B's failure to protect the steel as expected. Perhaps the other plant procedures, prior to the liquor being brought on-line, are affecting the characteristics of the protective monolayer that is thought to adsorb to the steel. It may be, for instance, that the use of dilute spent Bayer liquor to neutralize the sulphuric acid is detrimentally affecting the bond between the monolayer and steel. If this occurs, and the bond is not as strong, the monolayer may be removed once the flow rate is increased.

To test the effect of neutralising sulphuric acid with sodium hydroxide (not real Bayer liquor) a set of scenarios were supplied to the Department of Chemical Engineering at Monash University. It was found that removing the various stages in the test procedure increased the corrosion rate as follows.

- The corrosion rate (in terms of $1/R_p$) was $0.093 \Omega^{-1}$ for the normal procedure.
- The corrosion rate increased to $0.32 \Omega^{-1}$ when the neutralising stage was removed.
- When both the neutralising and pre-passivation steps were removed the corrosion rate increased to $0.38 \Omega^{-1}$.

Hence, at least according to this set of laboratory data, a sodium hydroxide neutralising step does not damage the film. In fact it appears that removing this step may lead to increased corrosion. Of course this system is very simple compared to real Bayer liquor, which is complex and variable. Though helpful, these tests do not completely rule out the possibility that the neutralising process detrimentally affects the monolayer. At most, they suggest that sodium hydroxide alone is not the culprit. It may be possible that sodium hydroxide is acting in synergy with other species present in real Bayer liquor to damage the film.

Alternatively, it may be speculated that the monolayer is formed correctly but interferes with the natural formation of oxide layer. If this happens then there is a question about what remains once the monolayer breaks down as a result of the high temperatures during the liquor cycle. If the monolayer had been functioning correctly prior to bringing the liquor up to full flow and temperature then it may have also prevented the protective oxide film forming. Hence, once the monolayer is removed, under full process conditions, bare metal would be exposed to the caustic. This coupled with the historical observation that the formation of a stable passivating oxide film is more difficult to achieve under full flow and temperature conditions would also be expected to lead to a higher corrosion rate.

4 Conclusions and recommendations

Plant trials have been used to compare two inhibitors. This also provided a good opportunity to compare laboratory measurements with field data. Both sets of data also indicated variability in the inhibitor

performance. However there are many difficulties associated with validating plant trials using laboratory studies since the ideal conditions in a laboratory cannot be replicated in the field.

Preliminary laboratory data indicated that a new inhibitor designed to provide protection during the early stages of a liquor cycle would lead to a significant reduction in corrosion rates throughout the liquor cycle. In addition, the use of this inhibitor would require much shorter pre-passivation times. The tested performance of Inhibitor B in the field did not show any definite improvements.

A comparison of the inhibitor performance, using the average total corrosion, indicated that although the new inhibitor appears to do its intended job during pre-passivation there is a chance that corrosion in subsequent steps will be higher. Hence, the new inhibitor in its present form is not a viable alternative to the current pre-passivation procedure

even though shorter pre-passivation times will result from using this inhibitor.

The corrosion rates resulting from treatment with inhibitor B are quite variable over the range of conditions encountered in the plant. Whereas, inhibitor A has performed consistently in the task it was designed to do – to protect the metal during an acid shot.

Acknowledgements

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