

AA18 - Importance of Chemical Cleaning for Precipitation Optimization

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Abstract

Precipitation circuits for alumina refineries such as Jamalco are responsible for producing the alumina trihydrate tonnage and alumina quality required to meet the organizations' contractual agreement. The successful operation of the precipitation circuit is dependent on the availability and optimization of critical equipment such as coolers, filters, and precipitators to attain the required yield and particle sizing. With the exposure of these equipment to varying hydrate slurry flows, liquor and solids concentrations, as corrosive materials, the ability to timely and effectively execute chemical cleaning is a key enabler for maintaining the circuit stability and effectiveness. This paper will explore the cleaning gaps identified and the strategies employed by Jamalco to optimize its caustic cleaning practices and the results attained. The impact of shell side acid cleaning of precipitation coolers will also be explored.

Keywords: Caustic cleaning, Acid cleaning, Free caustic, Precipitation, Scaling.

1. Introduction

The precipitation circuit within the Bayer process is known for its susceptibility to gibbsite and oxalate scaling [1] in the precipitator internals as well as in other key process equipment such as filters, heat exchangers, tanks, and lines. Scaling within these areas result in increased production losses due to lower precipitation yield and flow throughput capacity. Scaling mainly affects the precipitation yield by increasing settled solids within precipitators (reduced residence time), plugging of heat exchanger tubes and reduced equipment reliability affecting seed charge. The increase in settled solids for the precipitators result in equipment failures and increased downtime for descaling efforts [1].

It was recognized that the caustic cleaning practices within the precipitation circuit are critical to reduce the turnaround time for equipment on cleaning as well as prevent caustic embrittlement and the associated safety concerns. A revision of the precipitation caustic cleaning program was undertaken using the caustic soda service chart shown in Figure 1.

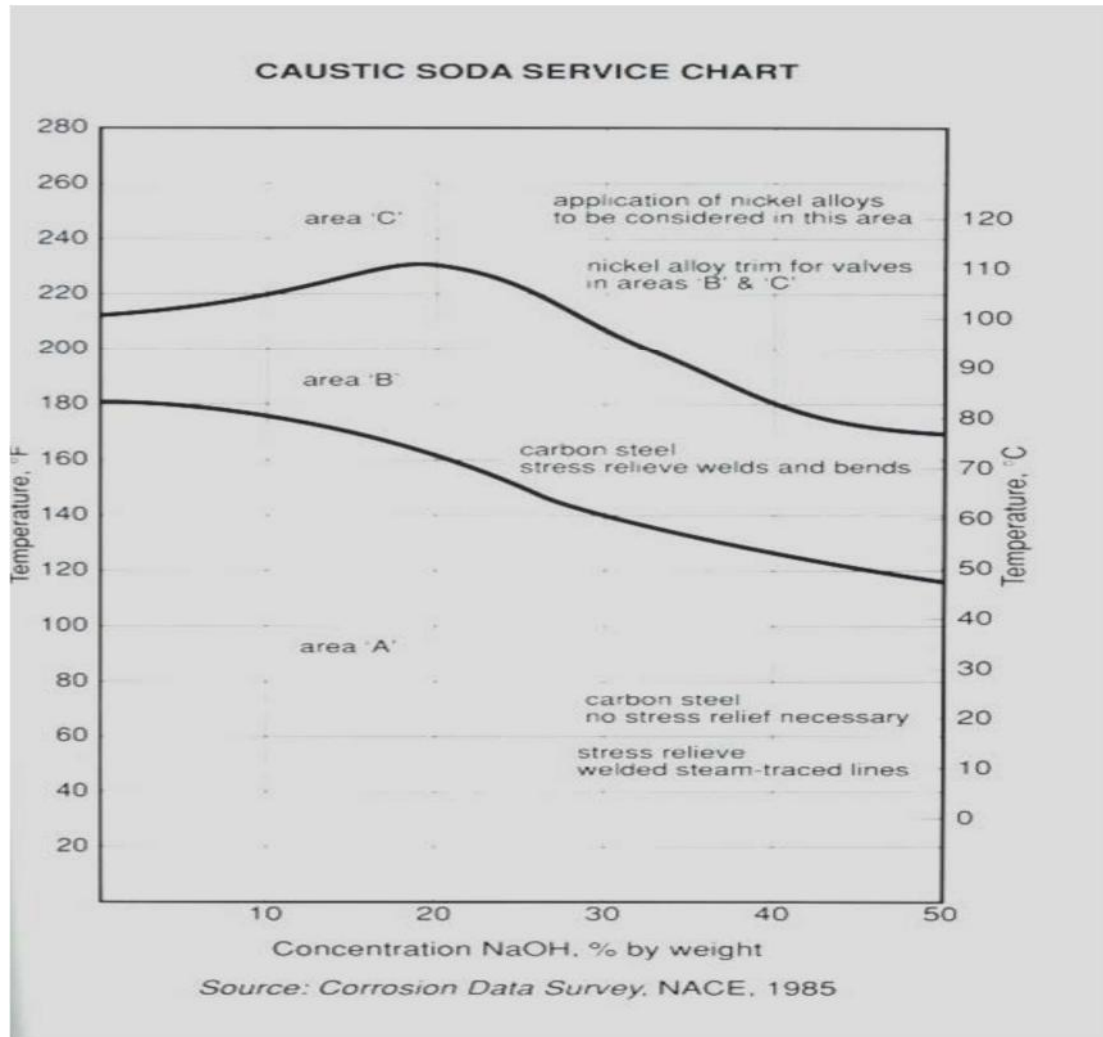
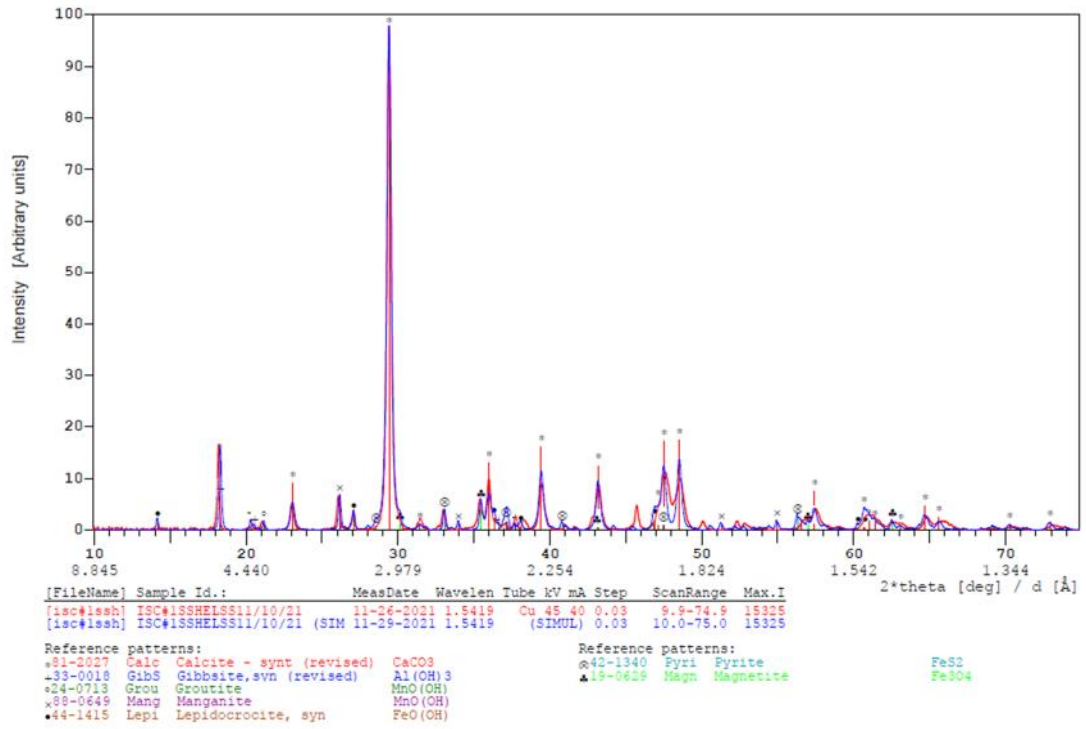


Figure 1. Caustic Soda Service Chart [2].

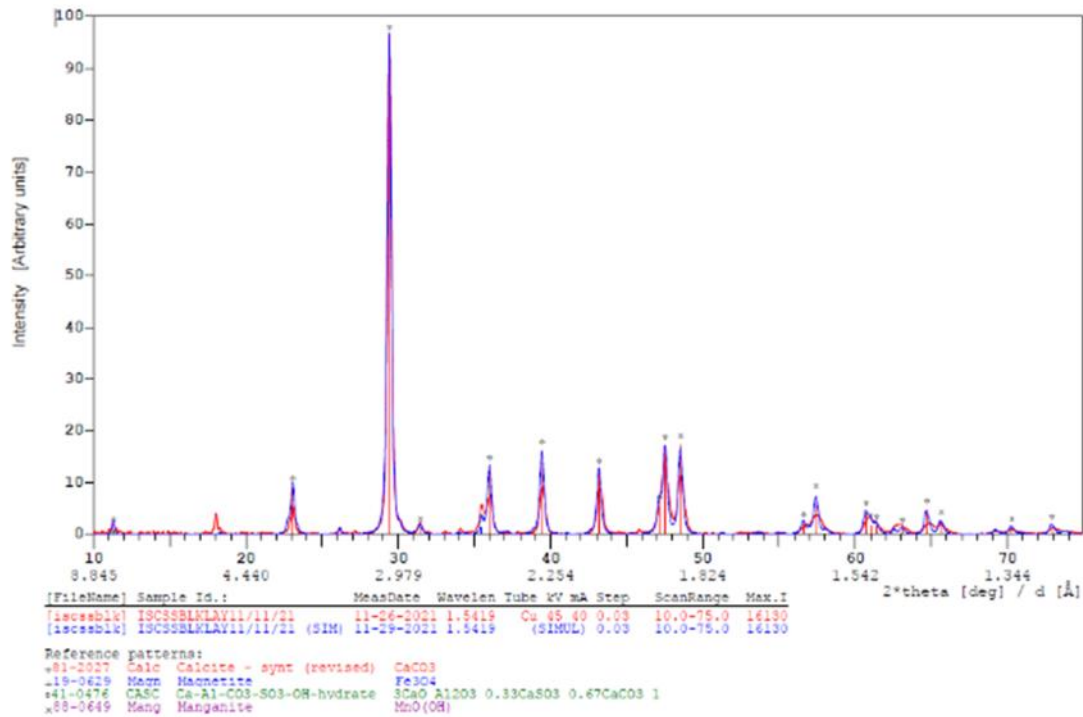
At Jamalco, a quality and yield critical parameter is the temperature of the 1st growth stage precipitators referred to as the 3rd tank temperature, these temperatures are affected by the performance of the Inter-stage coolers and volume management within the precipitation circuit (i.e. overflowing of the weir of the second seeded precipitators). One of the major limitations for the inter-stage cooler is shell side scaling due to the calcium carbonate levels within the cooling water stream. Scale composite analyses indicate up to 96 % calcium carbonate with the average level being ~64 % as shown in Figure 2.

Research shows that utilization of 10 % hydrochloric acid with a commercial inhibitor can effectively remove calcium carbonate scaling [3].



[ISC#1SSHELSS11_10_21-1] ISC#1SSHELSS11/10/21

Phase%	SUM	Calc	GibS	Grou	Mang	Lepi	Pyri	Magn
100.00	69.44	9.84	2.31	4.63	7.18	3.70	2.89	



[ICSSBLKLAY11_11_21-1] ICSSBLKLAY11/11/21

Phase%	SUM	Calc	Magn	CASC	Mang
100.00	96.89	1.45	0.97	0.69	

Figure 2. Scale composition for inter-stage cooler shell side.

2. Acid Cleaning of Inter-Stage Coolers

The performance of the inter-stage coolers within the precipitation department is a critical parameter for achieving the precipitation yield and sizing. The scaling of the shell side of the inter-stage cooler as shown in Figure 3 is a major challenge to the performance of the system.



Figure 3. Shell side condition of inter-stage coolers due to poor cooling water quality and delayed maintenance.

The scaling of the shell side resulted in U values as low as $\sim 341 \text{ W/m}^2\cdot\text{C}$ ($60 \text{ BTU/h}\cdot\text{ft}^2\cdot\text{F}$) versus a design U value for clean coolers of $\sim 2215 \text{ W/m}^2\cdot\text{C}$ ($390 \text{ BTU/h}\cdot\text{ft}^2\cdot\text{F}$) as shown in Figure 4.

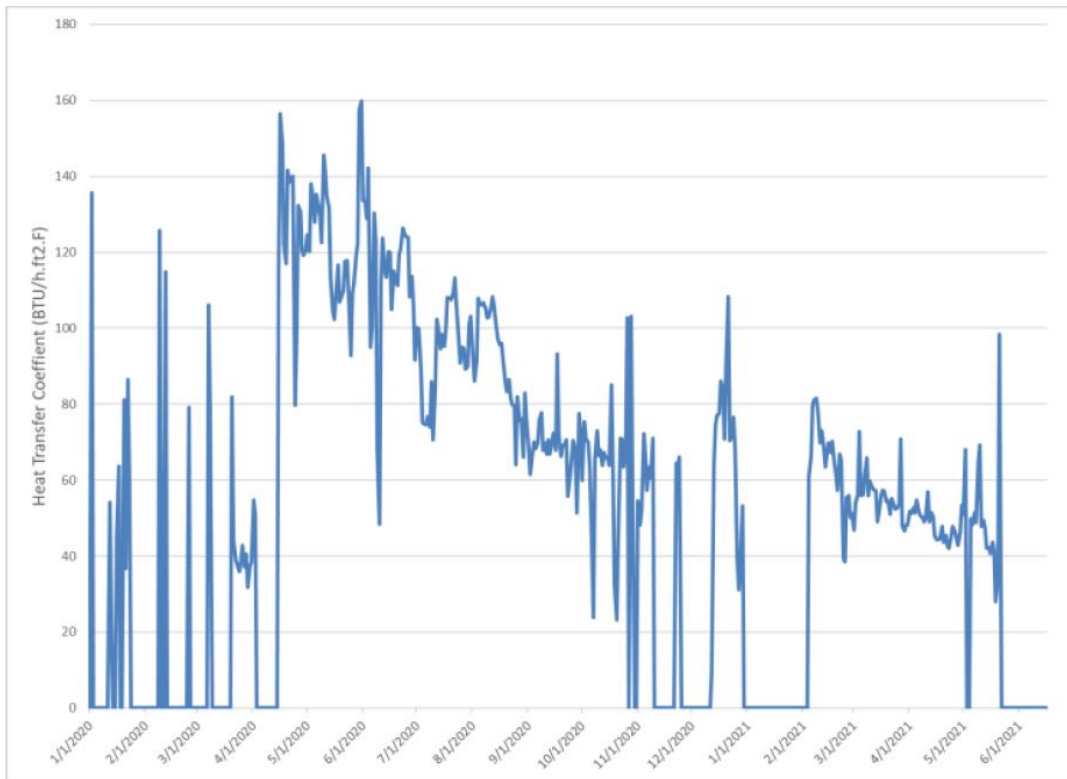


Figure 4. Example of heat transfer reduction for inter-stage cooler.

8 % Rodine 213 SF inhibited HCl acid was used to wash the shell side of the interstage cooler in a closed recirculating loop for 16–18 h. Rodine 213 SF is expected to provide 99.92 % inhibition [4]. The acid cleaning setup allowed for the top up of the recirculating acid once the concentration fell to 2 %; the top up was done using a 32 % HCl stock solution. The iron concentration was tracked throughout the cleaning process to determine the inhibitor effectiveness. The acid was deemed completed after attaining three (3) consecutive concentration results with a gap of < 0.05 %. Following the acid wash, the residual acid neutralized with sodium metabisulphite and the inter-stage shell side flushed with condensate.

The acid cleaning the inter-stage cooler shell side, has proved beneficial in improving the attainable heat transfer coefficients by > 200 %. Visual inspection of the tube side also indicated significant scale reduction as shown in Figure 5.



Figure 5. Shell side condition post acid wash.

Though a single acid wash approach has successfully yielded the intended results where most of the scale composition is calcium carbonate, this does not hold true for blends with high gibbsite content such as the scale highlighted in Figure 6.

For these types of scaling a combined cleaning approach is required, that is a caustic wash followed by an acid wash of the unit.

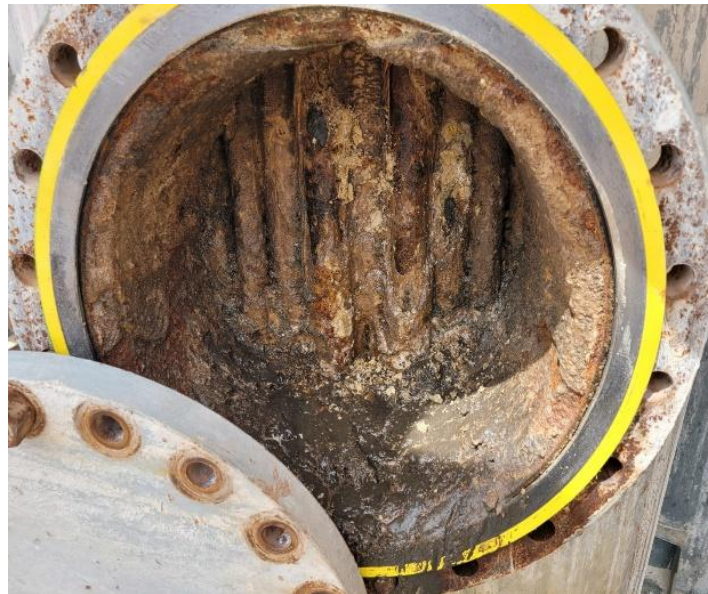
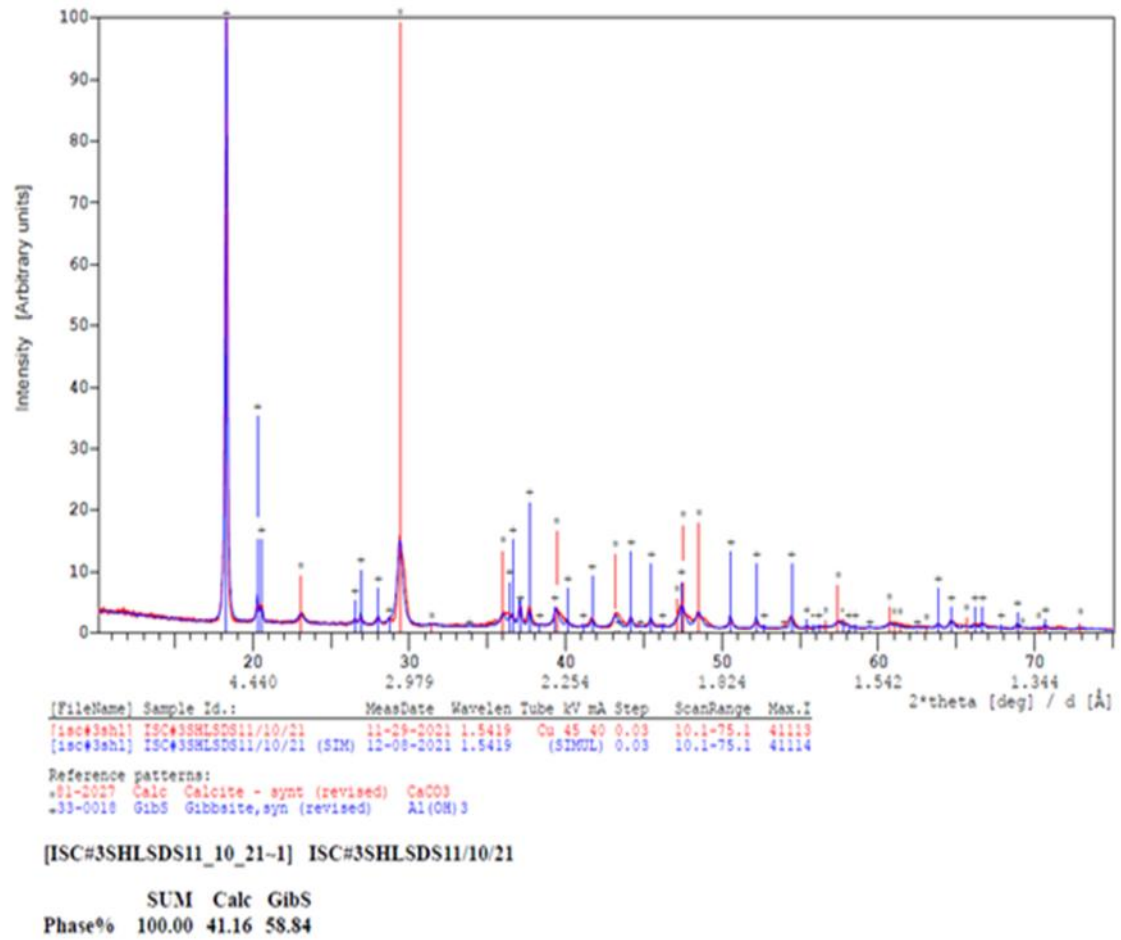


Figure 6. Top: Scale composition, Bottom: Post acid wash result for inter-stage coolers with higher gibbsite content (result of tube leaks).

2.1 Improvement Opportunities for Acid Washing of Interstage coolers

The draining and flushing of the inter-stage shell side with lake water prior to acid washing of the unit is critical for removing excess sludge buildup within the cooler. This flushing will allow for easier flow of the acid through the unit preventing residual scales as shown in Figure 7.



Figure 7. Left: Sludge, Right: Residual scale post acid wash.

Bi-annual acid cleaning of the cooler shell side will reduce the likelihood corrosion due to calcium scaling as shown in Figure 8. This will increase the life of the tubes as well as aid in maintaining the coolers heat transfer coefficient.

To improve the overall health of the inter-stage cooler, Jamalco has revised its operating and maintenance practices as well as the practices for cooling tower treatment and management. This review includes trialing different water treatment chemistries as well as improving cooling water quality monitoring. The troubleshooting system around the cooling water quality has also been optimized using troubleshooting guides, and reagent inventory management; the quality it tracked by a performance score.



Figure 8. Signs of corrosion on inter-stage tubes.

3. Caustic Cleaning in Precipitation

Scale growth is common for the Bayer process in areas where supersaturated liquors such as green liquor meet solids surfaces [5]. This scale growth reduces the efficiency of the fluid systems resulting in increased drag forces, blockages, and pump wear [5].

The production demands and instabilities in conjunction with the scaling rate of the equipment within the precipitation circuit has contributed to higher settled solids within precipitators,

reduced precipitation equipment residual life to < 36 % versus a best practice range of 45–55 %, reduced filter throughput affecting circuit seed charge and spent liquor generation, and pre-mature equipment failure. With this reduction in the residual life of the equipment it is expected that the equipment performance will decline as shown in Figure 9.

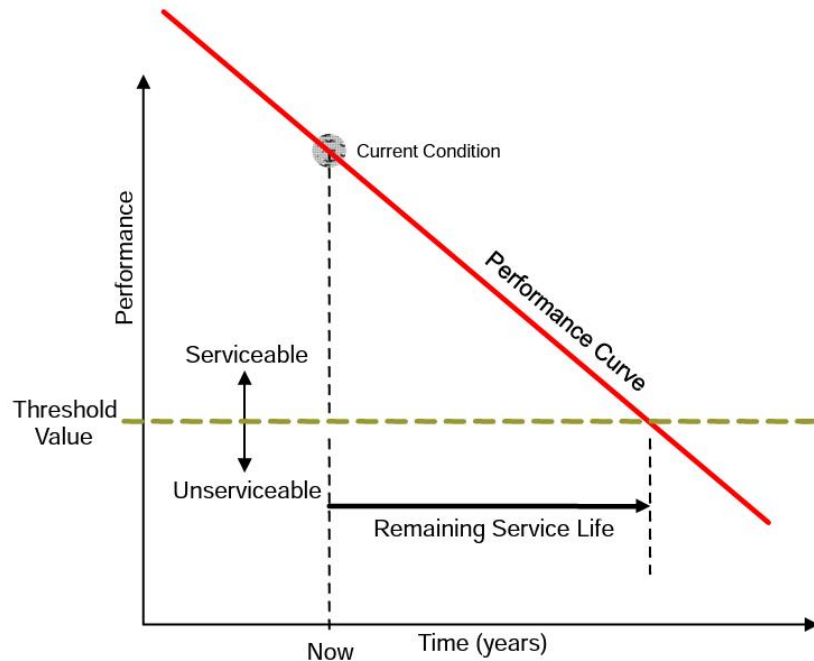


Figure 9. Residual life projection for assets [6].

As a result, caustic cleaning of critical areas such as lines, pumps, filters, and vessels is integral in maintaining optimal flow throughput within precipitation and by extension the refinery. The inability to maintain these flows result in increased production losses and downtime.

The effectiveness of caustic cleaning is tracked by the available caustic (also known as free caustic, FC) as indicated in Equation (1) and the alumina to caustic ratio change.

$$FC = TC - 1.04(Al_2O_3) \tag{1}$$

where:

- FC* Free Caustic, g/L
- TC* Total Caustic, g/L
- Al₂O₃* Alumina concentration, g/L

Data acquired from lab and process cleanings indicate an inverse relationship exists between these critical parameters as shown in Figure 10.

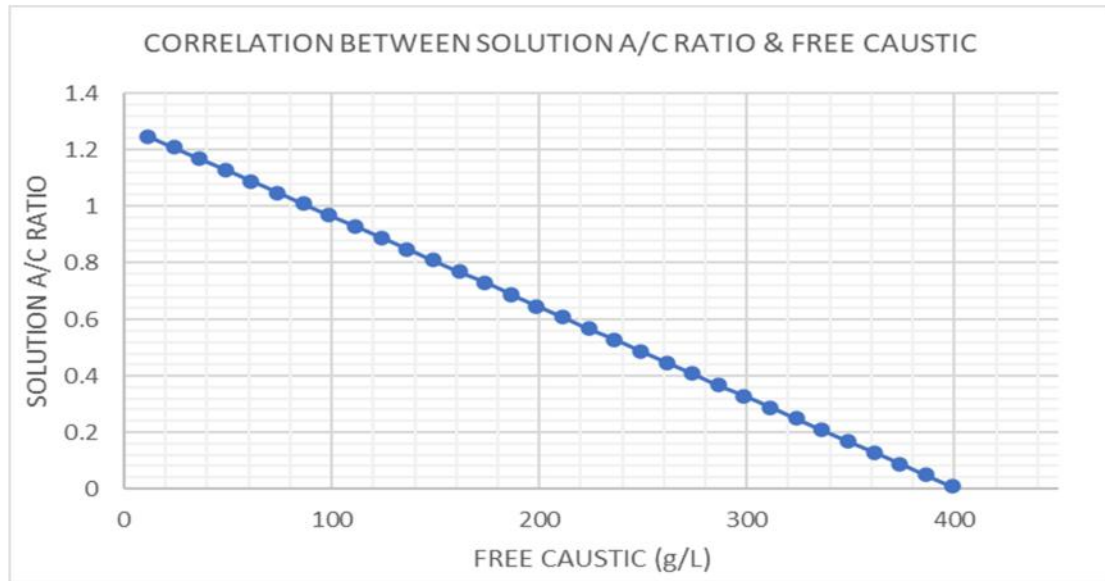


Figure 10. Correlation curve for free caustic and alumina to caustic ratio.

3.1 Caustic Cleaning Management

In the Precipitation department at Jamalco, a standard operating procedure is used for the caustic cleaning of vessels, pump suctions, lines, and filters. Deviation from this procedure can result in caustic ingress resulting in oxalate breakouts, damage to filter media as well as potential caustic embrittlement. The gaps identified within the caustic cleaning procedure surrounded the use of the total alkalinity, TA to track caustic strength, the cleaning temperature and concentration combinations used and well as the solution makeup and sampling protocol and the lack of a data tracking system for caustic cleaning.

For caustic cleaning of filters, it is recommended that concentrations of 200–450 g/l and temperatures of 80–100 °C [7]. Utilizing these industry standards as well as the caustic soda service chart, the following guidelines were implemented as a part of the caustic management system:

- Free Caustic Target: ≤ 400 g/L
- Total Caustic range:
 - o 400 to 420 g/l when making up solution with lake water
 - o 480 to 500 g/l when making up with a spent liquor and lake water combination
- Starting temperature: 70 ± 2 °C at maximum free caustic

This information was built into a database known as the Caustic Decision Sheet as shown in Figure 11. This database indicates to the user the target temperature for the caustic solution being used as well as requisite action to be taken for optimizing the caustic cleaning process thus reducing the troubleshooting time and streamlining troubleshooting efforts. An opportunity exists to increase the minimum starting temperature to 80 °C at maximum free caustic to reduce the turnaround time of the equipment.

Caustic Cleaning Decision Sheet												
Fi											Auto Calc is Currently: ON	
Date	Temp SF	Temperatu	Hydrometer SG	Al Resu	TC Result	Free Caus	TA Result	A/C Resu	Load Readi	Solids (Ea	Solids (West	Suggested Action to Take
23-Jul-21	164	173		78.7	399.3	317.5	436.1	0.197				check for caustic ingress Temperature is too high! Reduce temperature to match setpoint!
24-Jul-21	164	0		79.4	400.8	318.2	437.2	0.198				Caustic cleaning complete! Visually inspect, and determine the next step
26-Jul-21	166	143		87.3	386.0	295.2	400.8	0.226		52	38	If you're not deliberately adding caustic, check for caustic ingress
26-Jul-21	166	171		88.2	388.5	296.8	403.6	0.227				Caustic cleaning complete! Visually inspect, and determine the next step
27-Jul-21	165	200		92.9	396.9	300.3	411.3	0.234		56	48	Keep Cleaning Temperature is too high! Reduce temperature to match setpoint!
27-Jul-21	166	200		95.7	397.4	297.9	413.4	0.241				Keep Cleaning Temperature is too high! Reduce temperature to match setpoint!
27-Jul-21	212	141		48.8	223.3	172.5	251.6	0.219				No action. Sample next shift

Figure 11. Overview of caustic cleaning decision sheet.

After completion of laboratory tests to determine the correlation between the solution specific gravity (SG) as indicated by a hydrometer and A/C ratio. A correlation was developed and programmed into the database. This affords the user the ability to input a solution SG to determine the estimated free caustic and A/C ratio as shown in Equation (2).

$$A/C \text{ Ratio} = (-0.0032 \times FC) + 1.284 \quad (2)$$

where:

A/C Ratio Alumina to Caustic Ratio

FC Free Caustic, g/L

The use of the hydrometer will reduce the reaction time during the caustic cleaning process by eliminating the need to Metrohm analyses. Metrohm analyses will be used to confirm the starting and ending concentration for the cleaning process.

An additional feature of the caustic cleaning decision sheet is the caustic cleaning make-up calculator as shown in Figure 12, this calculator indicates to the user the solution volumes to be used for achieving a target free caustic. The calculator affords the user the option of selecting the dilutor used, equipment being cleaned and empty space for make-up.

CAUSTIC SOLUTION MAKE-UP CALCULATOR FOR SEED FILTER, TRAY THICKENER, INTERSTAGE COOLERS & PRECIPITATOR CLEANING

Note: The last solution data has to be inputted for the calculation to be done if the tank is not empty.

HOSE & SPENT LIQ. PUMP CLEANING TANK EQUIPMENT TO BE CLEANED: 36

VESSEL VOLUME: 454000 GAL

CAUSTIC CLEANING SPACE: 4/4

CLEANING VOLUME: 454000 GAL

EMPTY SPACE: 45%

DILUTION SOURCE TO BE USED: WATER

CURRENT VOLUME: 249700 GAL

REQUIRED VOLUME: 204300 GAL

TARGET FREE CAUSTIC: 300 gpl

CAUSTIC VOLUME: 128000 GAL

WATER VOLUME: 76300 GAL

LIQUOR VOLUME: GAL

LAST SAMPLE RESULTS:

Al2O3	TC	FREE CAUSTIC
		0

You may enter the Alumina and TC results, or click: Pull Last Sample Results

LIQUOR FRACTION: 10%

INSTRUCTIONS:

When making up the solution with WATER:

- Add up to 3 rings of raw caustic, i.e. 128000 gallons
- No liquor is to be added
- Fill the remaining volume with water, i.e. ~ 76300 gallons

Figure 12. Overview of caustic make-up calculator.

Caustic cleaning of the precipitators using these tools has resulted in a reduction of the total settled solids across the circuit as shown in Figure 13 leading to an estimated 4057 tonnes of recovered hydrate. It is estimated that for every meter of settled solids within an agglomerator, 0.02 g/l of

yield is lost and for growth precipitators 0.009 g/l of yield is lost. With the revised strategy, approximately two growth tanks are caustic cleaned simultaneously within 14 days or one agglomerator. This is compared to one growth tank for >14 days. Further trials will be done with agglomerators to determine the effective turnaround time.

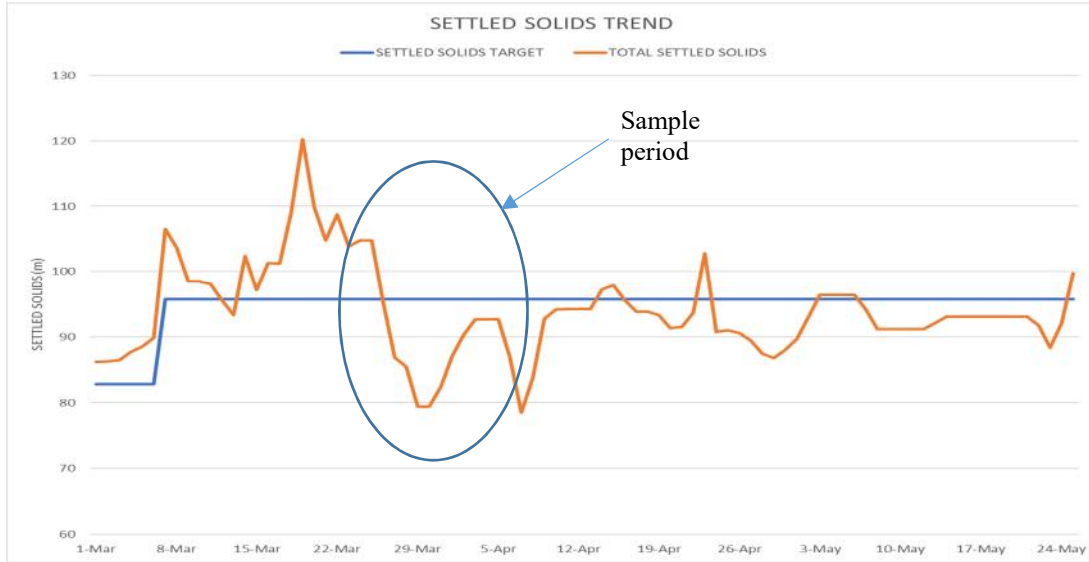


Figure 13. Total precipitator settled solids.

These gains can be sustained by stabilizing the green liquor flows and suspended solids within the circuit. Data acquired during plant trials indicates that scales with high oxalate concentrations versus gibbsite such as the sample highlighted in Figure 14.

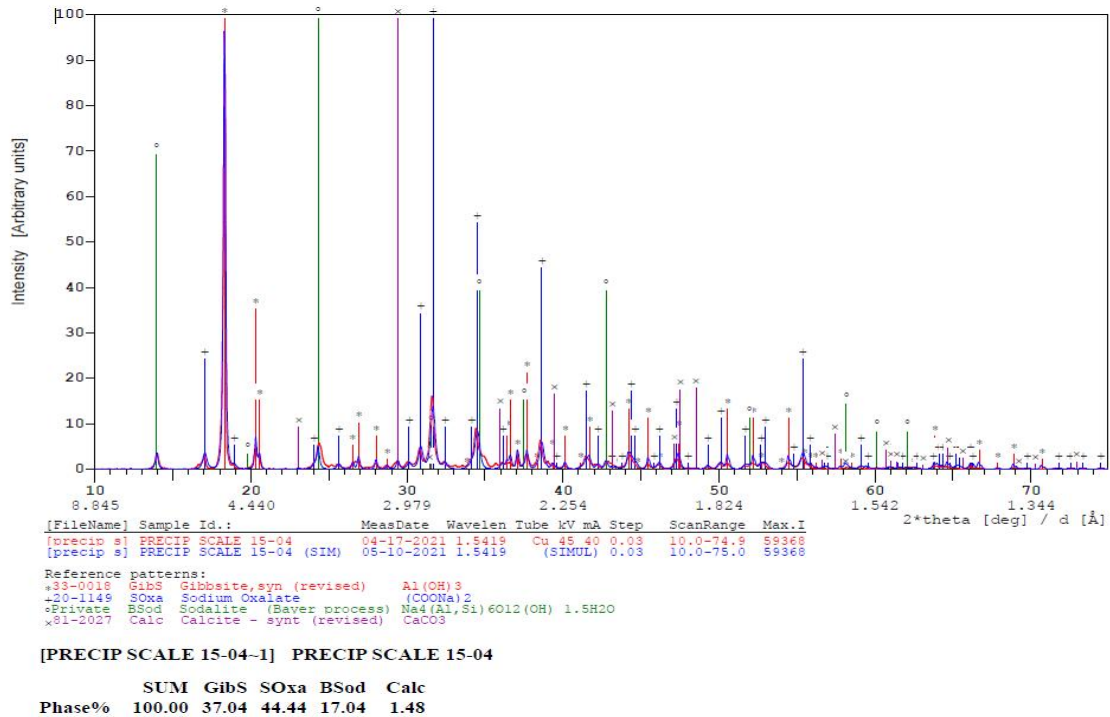


Figure 14. Precipitator scale sample analysis.

It is recommended that scales with these compositions be removed via a dual cleaning methodology; two proposals to be tested are as follows:

- Caustic Cleaning followed by mechanical cleaning;
- Caustic cleaning followed by hot water wash.

Jamalco utilizes the chemical and mechanical cleaning dual methodology to remove this type of scaling within precipitators.

4. Future Works

To further improve the chemical cleaning practices in precipitation, the installation of a clean-in-place system for acid cleaning is being considered and well as improving the caustic heater management through instrumentation and controls. The additional instrumentation for the caustic heaters will be used to improve the predictions for the Caustic Cleaning Decision Sheet.

Optimization of the precipitator injection systems will be undertaken to aid in sustaining lower settled solids within the precipitators.

5. Acknowledgements

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6. References

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