Application of Specialty Chemicals during Precipitation for Production of High Quality Alumina Hydrate with Higher Productivity

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Abstract

Production of Alumina hydrate by the Bayer process involves the digestion of bauxite with caustic soda and precipitation of alumina hydrate from the saturated sodium aluminate liquor. The yield and quality of precipitated alumina hydrate are significantly affected by the process parameters like precipitation temperature, residence time, seed size, solid content and alumina to soda ratio in the pregnant liquor, etc. Alumina manufacturers optimize these parameters to maximize the yield and product quality. Alumina hydrate particle size is one of the key parameters that determine the quality of metallurgical-grade alumina. A large percentage of fines, particularly below 45 microns, is undesirable for the manufacturers. Also, strength of alumina hydrate plays a major role on the breakage of particles during calcination and alumina handling. A highly efficient crystal growth modifier has been developed which can be very effective to control the fines generation and to produce much stronger particles by agglomeration and cementing the smaller particles. It also helps to control foam formation in precipitation and removes oxalate from the process. Reduction of foam can improve heat transfer efficiency, reduce scaling in precipitation, minimize tank overflow and improve operation of alumina hydrate classification systems due to reduced alumina hydrate retention in foam. In this paper laboratory tests have been conducted to evaluate effectiveness of this highly efficient crystal growth modifier on nucleation rate, particle size distribution (PSD), particles surface area, attrition index, liquor productivity and oxalate removal capacity.

Keywords: calcination, alumina quality, nucleation, agglomeration, attrition index.

1. Introduction

In the Bayer process, bauxite digested with hot caustic soda to produce saturated sodium aluminate liquor and bauxite residue as insoluble. Bauxite slurry generated in this process is treated with flocculants to separate the red mud from the pregnant liquor and this liquor is then further purified in a filtration process to remove suspended fine solids. This clear saturated aluminate liquor is then cooled and seeded with alumina hydrate to precipitate the final product, alumina hydrate. The yield and quality of precipitated alumina hydrate are significantly affected by the process parameters like precipitation temperature, residence time, seed size, solid content, caustic concentration and alumina to soda ratio in the pregnant liquor, etc.

Many research studies have been carried out to quantify the effect of process variables on the yield and strength of alumina hydrate precipitated from aluminate liquor [1, 2]. Alumina manufacturers optimize these parameters to maximize the yield and product quality. All over the world in Bayer process, there are existence of two processes for alumina hydrate precipitation technology, so called nucleation and agglomeration technology. The precipitation of alumina hydrate in both the technologies involves three mechanisms i.e. nucleation, agglomeration and crystal growth [3]. Sometime attrition is also an intermittent stage which happens due to collision of hydrate crystals or surface vessels. Size of the alumina hydrate is one of the key parameters which determines the quality of metallurgical grade alumina. The control of granulometry is linked to the relationship between the number of smaller particles below 5 microns (apparent nucleation) and the physio-chemical condition at the precipitation. Nucleation is called apparent because, in fact, it results from two well-known elementary phenomena: true nucleation –- the creation of particles around 1 to 2 microns in diameter –- and the agglomeration of fine particles less than 15 microns [4].

The success of particle size distribution control is based on the accurate measurement of fine particles in the seed hydrate. Process with higher agglomeration leads to agglomerates with numerous elementary crystals at around 20 microns and the crystals are cemented by crystal growth which corresponds to mosaic structures. These mosaic structured particles are stronger than the radial structured particles which are generated by very fine crystals around 8 microns [4–6].

Bauxite invariably contains long chain polymeric organic materials, of which content depends on its source. During digestion, these organic materials are degraded into numerous smaller species. Some of them are insoluble in nature and filter out along with red mud, whereas others are soluble and remain in the liquor. The soluble organic materials consist mainly of sodium salts of compounds with carboxylic and/or phenol functionalities, as well as a mixture of high molecular weight compounds generally referred to as humate in the Bayer process [7].

Because of this organic content of Bayer liquor, it has a natural tendency to create foam. Foaming of the liquor is aggravated by the mixing steps in the Bayer process. Foaming is especially a problem at the stage of separation of the red mud and during precipitation. The amount of pregnant liquor cannot be maximized in vessels partly filled with foam, and therefore maximum product throughput cannot be obtained. Foam also poses a safety hazard in that overflow can expose workers to high levels of caustic, which can cause severe chemical burns. Since foam is an insulator, reduction in foam can improve heat transfer efficiency. Reduction of foam can reduce scaling in precipitation and improve operation of alumina tri-hydrate classification systems due to reduced alumina tri-hydrate retention in foam [8, 9]

Oxalate in pregnant liquor is a problem which can lead to scaling problem, fine generation and formation of fragile particles [10, 11]. With continuous oxalate inputs from ongoing bauxite digestion, the concentration of sodium oxalate increases in the circulating liquor inventory until it reaches a relatively balanced concentration in the liquor.

Nowadays most of the plants use a variety of additives to control the particle size of the precipitated alumina hydrate and to reduce foam during precipitation. Scientists have developed technology to use polysaccharide or poly-saccharide graft co polymer for agglomeration and/or crystal growth promotion. The advantages of applying an aqueous emulsion comprising an alkyl or alkyl succinic anhydride have also been studied. Methods for enhancing production of alumina hydrate has been widely studied by Scientists [12–16].

Kimberlite India private Limited is a leading manufacturer of specialty chemicals for alumina industries for application as grinding aid, crystal growth modifier, foam control solution, synthetic flocculants, dewatering aid, scale inhibitor, corrosion inhibitor, humate removal, iron removal, dust suppressant and water treatment chemicals. It has developed an indigenous highly efficient crystal growth modifier, AL Flow 301 for alumina plants, which can be very effective to control both fine generation as well as reduction of foam. It is very effective to produce much stronger particles through agglomeration and cementing the smaller particles, controls foam in precipitation circuit and removes oxalate from the process. In this paper, laboratory tests have been conducted to evaluate the effectiveness of Kimberlite's AL Flow 301, on nucleation number, particle size, attrition index, liquor productivity, and oxalate level.

2. Experimental

2.1 Preparation for Test Samples

In all cases reported herein, spent liquor (liquor after precipitation) sample was collected from a refinery plant and the pregnant liquor was prepared of target alumina ratio (Al_2O_3/Na_2O_c) by digesting the alumina hydrate in this liquor at 145 °C for 45 minutes and filtered through Whatmann 42 size filter paper. This pregnant liquor was analyzed for alumina and caustic concentration and used for each set of precipitation tests. Seed sample was collected from the refinery plant was washed thoroughly with hot water. To maintain homogeneity, the seed hydrate was mixed well by Retsch auto divider, stored in a sealed container and used for all experiments. The chemical composition of spent liquor and the analysis seed hydrate used for the precipitation test are given in Table 1.

Table 1. Characteristics of spent liquor and seed hydrate.

Spent Liquor		**Seed Hydrate**	
Na_2O (Caustic)	154.8 g/L	-45 micron, %	2.47
Al_2O_3	90.7 g/L	d50, micron	105.9
RP (Al_2O_3/Na_2O_c)	0.586	Surface area, cm^2/gm	278
Carbonates	19.6 g/L	LOI % at 1000 °C	34.5
Chlorides	0.58 g/L	SiO_2 %	0.010
Sulphates	0.12 g/L	Fe_2O_3 %	0.065
Oxalates	0.514 g/L	Na_2O(total) %	0.23
Silicates	732 mg/L	CaO %	0.026
Fe_2O_3 (total)	18 mg/L	K_2O %	0.0006
Fe_2O_3(soluble)	7 mg/L	ZnO %	0.0003
Organic Carbon	4.54 g/L	V_2O_5 %	0.0009
P_2O_5	70 mg/L	MgO %	0.0006
Ga_2O_3	112 mg/L	TiO_2 %	0.0014
V_2O_5	56 mg/L	P_2O_5 %	0.0010
		Ga_2O_3 %	0.0068

2.2 Precipitation Test

In the present investigation, all precipitation tests were conducted in an Intronics-made rotating water bath fitted with a timer and a precise temperature controller. The set-up consists of 24 plastic bottles, each with a capacity of 500 mL, which can be rotated continuously up to 100 rpm and 90 °C. For each set of experiments, freshly prepared pregnant liquor of known caustic concentration was used. A calculated amount of seed hydrate was added to maintain 600 g/L solids. Then, varying dosages of CGM samples were added. After thorough mixing, the entire hydrate slurry was transferred to 500 mL precipitation bottles, and the precipitation tests were carried out. To evaluate the effectiveness of the CGM, a reference CGM – collected from an alumina plant and currently in wide use across Indian alumina plants – was also tested. The details of the precipitation conditions are provided in Table 2.

Table 2. Parameters for precipitation test.

Sl.N°.	**Parameters**	**Set points**
01	Na_2O_c g/L of Pregnant Aluminate Liquor	151.94
02	RP or (Al_2O_3/Na_2O_c) of Pregnant Aluminate Liquor	1.060
03	Solids in Test Slurry (input)	600 g/L
04	Precipitation Temperature (water bath temperature)	60 °C
05	Precipitation Time (timer)	48 hours
06	Rotation Speed of Test Bottle	20 rpm
07	Test Sample Quantity	500 mL
08	CGM Dosage	0 (blank), 25, 50, 75, 100, 150 and 200 ppm

2.3 Particle Size and Population Analysis

In this present investigation, analysis of grain particle size distribution, surface area and particle count of hydrate samples were analyzed by Accusizer 730AD of Particle Sizing System, which is based on single particle optical sizing, a technique that uses light interaction with individual particle to determine size and count [17].

2.4 Attrition Index Test

Loading and unloading of alumina, transportation and dry scrubbing within smelter tends to breakup alumina particles [18]. Consequently, determination of the breaking strength of alumina has become an empirical parameter and thus the measurement of attrition index became important for aluminium manufacturer [19].

In the present work, calcined alumina samples were prepared by calcining alumina hydrate - obtained by precipitation - in a muffle furnace at 1000 °C for one hour. For the attrition test, the alumina samples were placed in a Pyrex tube 1500 mm long with an internal diameter of 25.4 mm. The samples were then subjected to a stream of compressed air at a pressure of 3.7 bar and a flow rate of 384 liters per hour, passing through a 0.381 mm calibrated orifice for 15 minutes. Particle size analysis (at 45 microns) was carried out both before and after attrition. The attrition index was calculated using the following formula in Equation (1).

$$\text{Attrition Index (\%)} = \frac{(+45\ \mu\text{m before test}) - (+45\ \mu\text{m after test})}{(+45\ \mu\text{m before test})} \times 100 \tag{1}$$

2.5 Oxalate Carbon Analysis

In the present investigation, oxalate carbon in the pregnant aluminate liquor and spent liquor after precipitation were carried out by ion chromatography method using Metrohm 925-IC.

2.6 Caustic and Alumina Analysis

In the present work, caustic soda and alumina were tested by potentiometer method by 905-Titrando, Metrohm using sodium gluconate method [20]. Calculation of RP is made by alumina (Al_2O_3, g/L) divided by caustic soda (Na_2O, g/L). The liquor productivity is calculated based on the Equation (2).

$$\text{Liquor Productivity (g/L)} = \text{Initial Caustic (g/L)} \times \left(\text{RP}_{\text{before precipitation}} - \text{RP}_{\text{after precipitation}}\right) \tag{2}$$

2.7 Anti-Foam Test

To observe the anti-foaming properties of CGM, a foam sample was artificially created by grinding 0.1 g of laundry detergent (or surfactant like sodium laureth sulphate) and 0.7 g of aluminium sulphate in a mortar into a fine powder with a pestle. Then this powder was dissolved in 50 mL of water in a conical flask. In another flask, 5 g of sodium hydrogen carbonate was dissolved in 50 mL of water. Then both the contents were mixed quickly and transferred the whole material to the 1000 mL test cylinder and top up with 500 mL synthetic pregnant aluminate liquor. The compressed air was passed slowly from the bottom through a small tube to generate foam up to 1000 mL mark and time taken is noted. Then into another cylinder with same condition 50 ppm of CGM mixed with aluminate liquor and test was repeated.

3. Results and Discussion

3.1 Effect of CGM on Population Number of Smaller Particles

Precipitation tests were conducted without and with addition of AL Flow 301 along with reference CGM (presently being used by the majority Indian refineries) with varying 25, 50, 75, 100, 150 and 200 ppm dose. After completion of precipitation test the product hydrate were filtered, washed and then analysed by Accusizer for particle population counts at 1.51, 1.84, 2.49, 3.40, 3.55 and 9.51 microns and the results are represented in Table 3.

Table 3. Analysis of number of smaller particles with variation of CGM dose.

Number (N × 10^5) at micron size	**Blank**	**AL Flow 301 dosage (ppm)**					
	0	**25**	**50**	**75**	**100**	**150**	**200**
at 1.51 µ	23.592	20.146	12.474	13.633	13.142	14.012	13.043
at 1.84 µ	11.359	9.055	6.046	6.028	6.482	5.954	6.947
at 2.49 µ	3.488	3.044	1.816	1.793	1.848	1.827	1.807
at 3.40 µ	1.353	1.128	0.740	0.775	0.774	0.771	0.773
at 3.55 µ	1.144	1.065	0.641	0.674	0.659	0.639	0.662
at 9.51 µ	0.148	0.132	0.103	0.105	0.11	0.109	0.101
	Reference CGM dosage (ppm)						
		25	**50**	**75**	**100**	**150**	**200**
at 1.51 µ		23.433	22.433	17.321	18.863	18.244	17.038
at 1.84 µ		11.869	11.014	9.386	8.636	8.045	7.299
at 2.49 µ		3.544	3.046	2.378	2.567	2.384	2.764
at 3.40 µ		1.324	1.212	1.014	1.058	1.110	1.034
at 3.55 µ		1.140	1.124	0.969	0.846	0.844	0.836
at 9.51 µ		0.136	0.132	0.120	0.118	0.126	0.112

From Table 3, upon the addition of both AL Flow 301 and the reference CGM there was a sharp reduction in the number of smaller particles with respect to blank. This is because of the agglomeration of smaller particles. Lesser number of smaller particles indicate the effectiveness of CGM for agglomeration. From the table, it can be of CGMat the optimum dose for Al Flow is 50 ppm and 75 ppm for reference CGM. It can be observed that the number particles at 1.51 micron were 23.592 (×10^5) in blank sample (zero CGM dose) and on addition of 50 ppm of AL Flow 301, it was reduced to 12.474 (×10^5) and with 75 ppm of reference CGM it is 17.321(×10^5). This can be seen that Al Flow 301 works better in comparison to reference CGM at all sizes from

1.84 to 9.51 microns. Even after the addition of 200 ppm of Reference CGM, reduction of 1.51-micron particles is not better than the result achieved with only 50 ppm dose of AL Flow 301.

It is known that at low precipitation temperatures, productivity is high, but the generation of fines is greater. Similarly, at higher precipitation temperatures, productivity is lower, but the crystals are coarser. Manufacturers require both productivity and coarser hydrate. Nucleation number is generally considered as the number of the smallest particles per gram of seed hydrate, and these are the smaller sizes from where agglomeration and growth start. A high population of smaller particles means that fines generation will increase over time. For particle size control of the final product, effective control of nucleation is very important. A high nucleation number tends to produce more fines, if there is no agglomeration. Therefore, low nucleation number is desirable to get coarser material. From Table 3 control of nucleation can be done effectively by addition of a very small dose of AL Flow 301, in comparison to the existing CGM. Figures 1 to 4 presents a comparison between AL Flow 301 and Reference CGM.

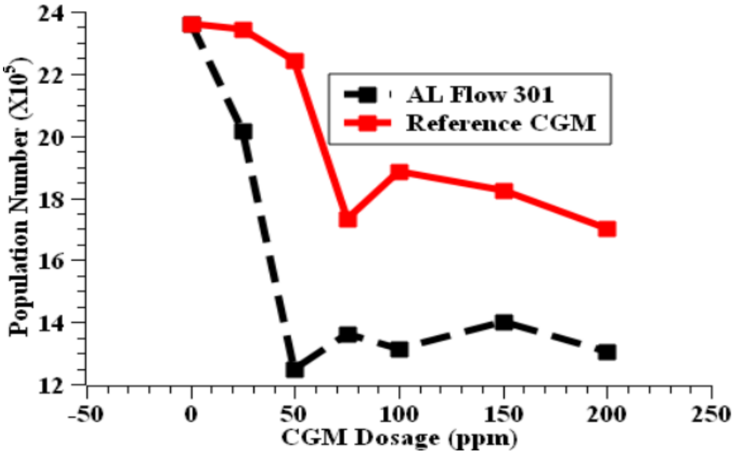

Figure 1. Effect of CGM on population number (10^5) at 1.51 micron.

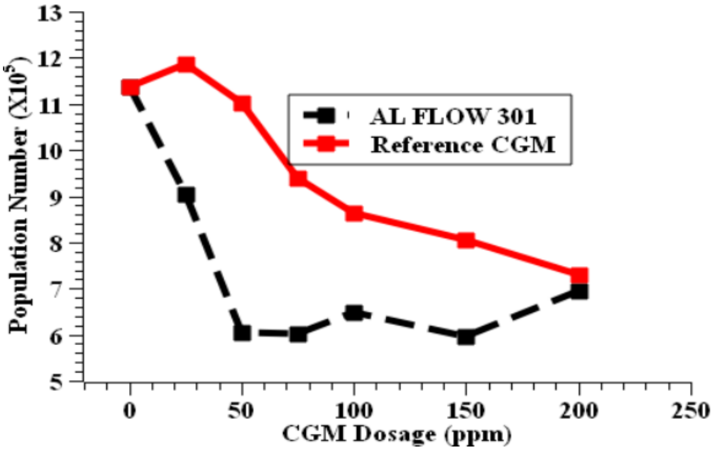

Figure 2. Effect of CGM on population number (10^5) at 1.84 micron.

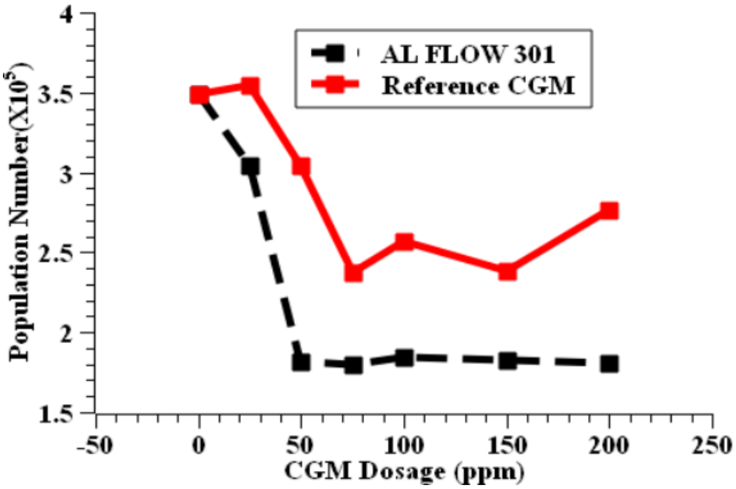

Figure 3. Effect of CGM on population number (10^5) at 2.49 microns.

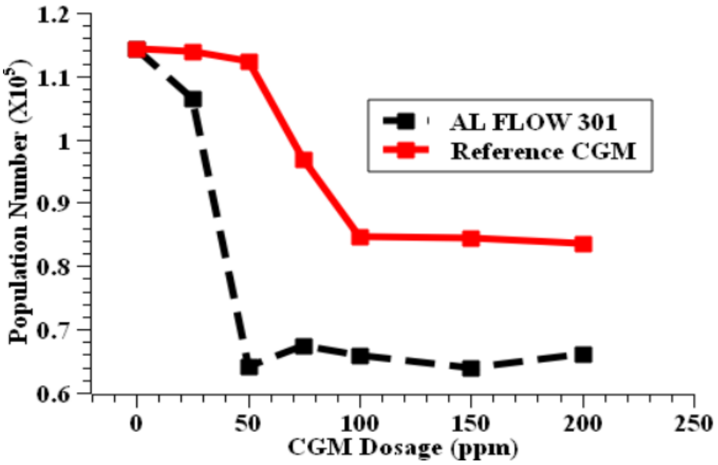

Figure 4. Effect of CGM on population number (10^5) at 3.55 microns.

From figures 1 to 4, effectiveness of AL Flow 301 presents better performance than the reference CGM on reduction of particle counts at almost all levels from 1.51 to 9.51 microns size.

3.2 Effect of CGM on Surface Area

Analysis of seed surface area of precipitated hydrate with addition of different dose of AL Flow 301, reference CGM and blank were carried out by Accusizer, and results are presented in Figure 5.

According to Figure 5, AL Flow 301 results in lower seed surface area products compared to the reference CGM. A lower seed surface area indicates overall coarser particle sizes. The seed surface area of the blank sample was 266.8 cm^2/g, but upon the addition of 50 ppm of AL Flow 301, it was reduced to 246.5 cm^2/g. This reduction is due to a decrease in the number of fines due to the agglomeration of smaller particles, as shown in Table 3. In contrast, the reference CGM resulted 256.4 cm^2/g with optimum dose of 75 ppm, and it achieved a minimum surface area of 252.5 cm^2/g at a 150 ppm dose, but it was still not as effective as AL Flow 301. A lower surface indicates the presence of less fines which means agglomeration is more, which happened in case of Al Flow 301.

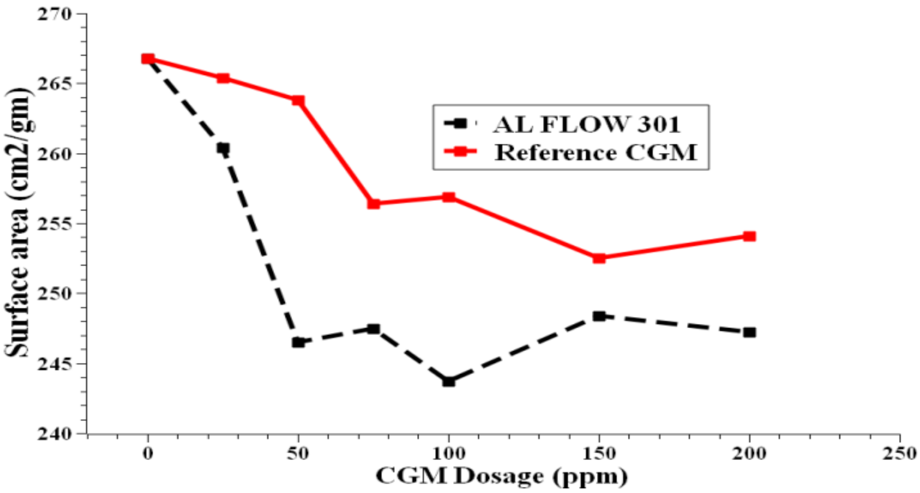

Figure 5. Effect of CGM on surface area, cm^2/g.

3.3 Effect of CGM on Grain Size of the Particles

Particle size analysis of the precipitated hydrate was carried out using an Accusizer, and the results are presented in Table 4. Table 4 shows that the effects of both AL Flow 301 and the reference CGM on granulometric weights are not highly noticeable, although there are significant changes in the number of fine particles, as seen in Table 3.

Table 4. Effect of CGM on particle size (under size %).

	AL Flow 301 dosage (ppm)						
micron	**0**	**25**	**50**	**75**	**100**	**150**	**200**
23	0.51	0.50	0.38	0.42	0.46	0.39	0.41
30	0.88	0.88	0.73	0.77	0.78	0.75	0.76
45	2.56	2.53	2.34	2.38	2.27	2.29	2.15
60	6.73	6.66	6.57	6.48	6.08	6.50	6.37
80	19.97	19.86	18.90	19.02	18.35	19.33	19.75
110	58.49	58.11	57.54	56.60	53.2	54.89	55.65
125	73.69	74.54	75.73	77.05	79.23	76.85	75.99
160	93.04	93.28	93.33	93.28	94.49	94.01	93.43
200	100	100	100	100	100	100	100
	Reference CGM dosage (ppm)						
micron	**0**	**25**	**50**	**75**	**100**	**150**	**200**
23	0.51	0.51	0.51	0.42	0.45	0.46	0.49
30	0.88	0.89	0.90	0.88	0.83	0.86	0.89
45	2.56	2.57	2.58	2.50	2.37	2.44	2.41
60	6.73	6.74	6.78	6.70	6.47	6.58	6.39
80	19.97	20.02	20.99	18.90	18.91	19.55	17.58
110	58.49	59.42	57.69	58.94	57.34	56.36	56.99
125	73.69	74.20	73.55	75.24	76.03	77.11	77.70
160	93.04	92.16	94.89	92.08	92.37	92.19	93.97
200	100	100	100	100	100	100	100

This is because in the present investigation, precipitation was carried out only for 48 hours. While calculating the weight %, the weight of fines (below 9.51 microns) has a little impact on total weight since sizes are spread over from 1.5 to 200 microns and median size is approximately 105 microns. However, from Table 4 a little decrease in fines is seen below 60 microns, which may be due to some agglomeration effect during the precipitation test. Internationally 45 micron size in calcine alumina is considered as the measure of the fineness and below 45 micron size in higher side is considered as fines. In the present case, the effect on coarsening will be seen in the plant with continuous cycle operation of about 30 to 40 days considering rate of growth 1 micron per day [2].

3.4 Effect of CGM on Liquor Productivity

Analysis of caustic soda and alumina concentration of liquor samples after precipitation test with addition of different dose of Al Flow 301, Reference CGM and blank were carried out by potentiometer and liquor productivity was calculated by equation (2). The results obtained from calculation are presented in Figure 6.

Results presented in Figure 6 show that with optimum dose of 50 ppm AL Flow 301 and 75 ppm reference CGM, the liquor productivity was 75.7 and 75.5 kg/m^3 respectively which is not widely visible. However, there is about 0.8 kg/m^3 increase in liquor productivity in addition of 50 ppm of AL Flow 301, in comparison to blank.

Productivity increases with decrease in precipitation temperature and AL Flow 301 is more effective to control fine generation at lower temperature. In plant condition higher productivity can be achieved with the addition of AL Flow 301 by targeting nucleation number at lower precipitation temperature.

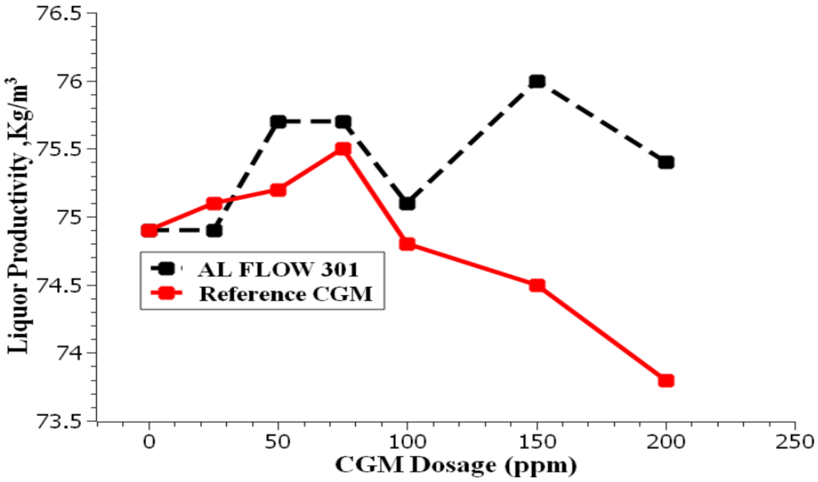

Figure 6. Effect of CGM on liquor productivity.

3.5 Effect of CGM on Oxalate Solubility

Analysis of soluble oxalate concentration in liquor samples after precipitation tests, with different doses of CGM and without CGM (blank), was carried out using ion chromatography. The results are presented in Figure 7.

From Figure 7, it can be observed that the oxalate concentration in spent liquor after precipitation is low in all cases with AL Flow 301 and in some cases with the reference CGM. Overall, AL Flow 301 appears to be highly effective in controlling soluble oxalate in the aluminate liquor.

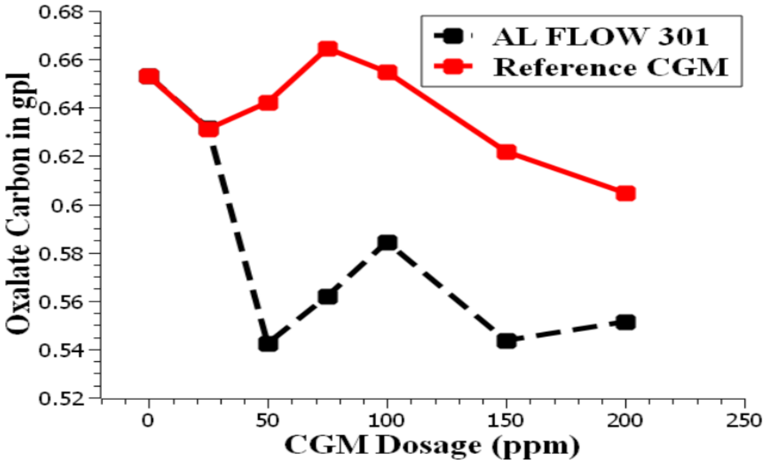

Figure 7. Effect of CGM on soluble oxalate.

In the blank test, soluble oxalate was 0.6555 g/L, but upon the addition of 50 ppm AL Flow 301, it decreased to 0.5424 g/L, representing a reduction of about 17.3 %. Oxalic carbon, which is responsible for fines generation and causes operational problems such as scale formation and precipitation of fragile crystals, can be effectively managed by adding AL Flow 301. The equilibrium of sodium oxalate is dynamic, responding to changes in bauxite inputs, temperature and liquor caustic concentration and other factors of refinery. As the liquor flows through the process, it precipitates at a certain point or multiple points in the process. This can be on the walls of the precipitation tank, on the surface of the alumina hydrate, seed filters at the evaporator or can be precipitated as a discrete particle mixed with hydrate. Hui-bin Yang et al. [10] have reported a variety of sodium oxalate removal method such as red mud washing, aluminium hydroxide seed wash with water, lime Causticization, crystallization from spent liquor, Bayer liquor calcination, wet oxidation etc.

From the above experiments, it can be concluded that sodium oxalate present in the aluminate liquor in soluble form can be removed from the circuit as a precipitate along with the precipitated hydrate, which is finally burnt during calcination.

3.6 Effect of CGM on Attrition Index

A portion of the alumina hydrate obtained after the precipitation test was filtered, washed, dried, and calcined in a muffle furnace, and attrition tests were conducted. The test results are presented in Figure 8.

Low attrition index indicates less fragile and stronger particles. From the above results the CGM are effective towards attrition, but AL Flow 301 is more effective in producing stronger crystals. Attrition index of blank sample was 13.4 % and on addition of 50 ppm AL Flow 301, it decreases to 6.4 %, which indicates generation of very strong crystals.

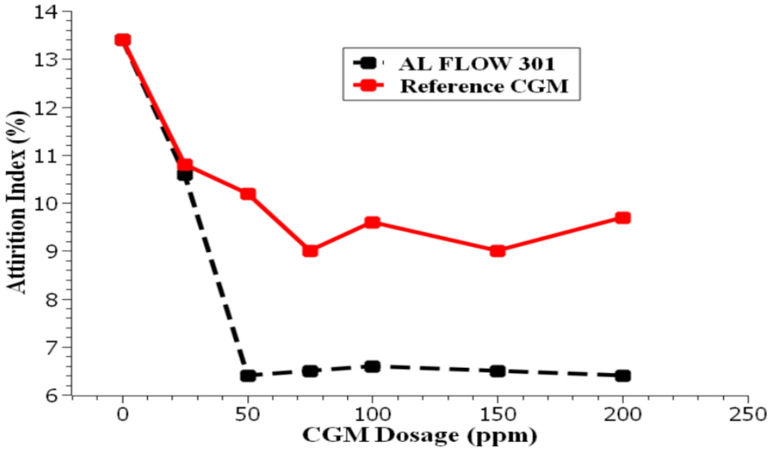

Figure 8. Effect of CGM on attrition index %.

3.7 Effect of CGM on Reduction of Foam

An anti-foam test was carried out in the laboratory to evaluate the tendency of AL Flow 301 to generate foam. According to the method, artificial foam was created in a 1000 mL glass cylinder. In another cylinder, 50 ppm of AL Flow 301 was added along with the sample, and foam was then attempted to be generated. The experiment showed that very little foam was produced, and it was unstable disappearing as soon as the airflow stopped. These results suggest that AL Flow 301 has inherent anti-foaming properties, which are highly beneficial for plant operations.

4. Conclusion

Based on the experimental results, the specialty chemical AL Flow 301, developed by Kimberlite India Private Limited, demonstrated effectiveness in reducing fines generation, promoting the precipitation of stronger crystals, and controlling soluble oxalate levels. When compared to the reference CGM, AL Flow 301 showed superior performance even at lower dosages. These findings suggest that increased plant productivity may be achieved by maintaining lower precipitation temperatures combined with the addition of smaller doses of AL Flow 301. Furthermore, due to its observed anti-foaming properties, the use of additional anti-foaming agents may be unnecessary in the precipitation process.

5. Acknowledgements

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