

Light Metals 2011

ALUMINA and BAUXITE

**Precipitation, Calcination and
Properties**

SESSION CHAIR

Hans-Werner Schimdt

Outotec GmbH

Oberursel, Germany

EFFECT OF TECHNOLOGICAL PARAMETERS ON PSD OF ALUMINUM TRI-HYDROXIDE FROM SEED PRECIPITATION IN SEEDED SODIUM ALUMINATE SOLUTIONS

Yusheng Wu, Mingchun Li Yanping Qu

School of Materials Science and Engineering, Shenyang University of Technology, Liaoning 110178, P. R. China

Keywords: Bayer process, Sodium aluminate, Particle size distribution, Aluminum tri-hydroxide

Abstract

Periodic attenuation of particles, which interferes seriously with normal alumina production, is a characteristic of the Bayer process. In order to construct a mathematical model of total number of particles and particle size distribution (PSD) which is helpful to predict and control the PSD of aluminum tri-hydroxide for an alumina refinery, the PSD of aluminum tri-hydroxide from the seeded precipitation process with different technological parameters, has been investigated under industrial conditions modeled in the laboratory. The results show a move of PSD to smaller particle sizes with decreasing initial precipitation temperature and increasing molar ratio of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$. The volume fraction of particles below $44\mu\text{m}$ first increased and then decreased with increasing initial concentration of Na_2O . The PSD has the opposite variation tendency with different initial seed amounts, compared with the initial concentration of Na_2O .

Introduction

The most common industrial process for alumina production from bauxite is the Bayer process. Seed precipitation is the key stage, significantly affecting the yield and specification of product [1-3]. Lots of work has been done on the precipitation process for enhancing precipitation ratio in the sodium aluminate liquor, and improving product specifications [5-8]. However, the periodic attenuation of grain size, that mass percent of particle size above $44\mu\text{m}$ and attenuating periodic change from 50% to 90% and 3 to 5 months respectively, inherently exists in the Bayer process in China. During attenuation, the functioning of filters deteriorates and product quality drops. Meanwhile, the electrical consumption and the dust density in the exit of electrofilter increase [9-12]. Although some work has been done to solve the problem, such as using sandy alumina production technology invented by France and investigating the particle size distribution (PSD) of production alumina in some branches of China Aluminum Co., Ltd, the explosive attenuation of alumina particles can't be avoided.

In general, the precipitation of aluminium hydroxide from seeded sodium aluminate solutions (SAS) is comprised of crystal growth, nucleation, agglomeration, and attrition [13-15]. By the mechanism of crystal growth, dissolved alumina hydrate transfers from the aqueous phase to the solid phase, depositing on the surface of existing particles. Thus the growth rate determines the overall liquor/solid mass balance. Growth also impacts the particle population balance because particles will increase in diameter. The crystal growth rate in gibbsite precipitation has been shown to depend on three factors: the supersaturation of sodium aluminate in solution, the temperature, and the solid surface available for reaction. Agglomeration is essential in gibbsite precipitation because it is the only economically feasible method of producing particles with the required size, given the

extremely slow growth rate. Agglomeration consumes smaller particles and creates larger particles, representing the additive volume of the smaller particles consumed. Nucleation is the formation of new, minute particles which replenish the particle count of the system. Particles are continuously lost through agglomeration or product removal. Thus for efficient operation, nucleation must provide enough particles to maintain the total particle count, and to provide surface area for continuing growth. Particle size distribution of products mainly depends on all of the above four precipitation steps.

Most alumina plants nowadays have already developed, or are in the process of developing, their own precipitation model for the simulation of the aluminium hydroxide precipitation mechanisms. In order to construct a mathematical model of total number of particles and particle size distribution (PSD) which is helpful to predict and control the PSD of aluminum tri-hydroxide for alumina refinery, the PSD of aluminum tri-hydroxide from a seeded precipitation process under different technological parameters has been investigated under industrial conditions, modeled in the laboratory.

Experimental

The main apparatus for seed precipitation study included a blade-paddle mixer tank and a Model LB-801 super constant temperature bath. The supersaturated sodium aluminate solutions, with industrial concentration and initial gibbsite seed for all experiments, were provided by the Shandong branch of China Aluminum Co., Ltd. Precipitation time is 40 h. After adding seed, the precipitation commences by stirring at 260 rpm and the temperature decreased uniformly over the desired ranges.

Solution samples (precipitate suspension) were analysed by acid-base titration and complexometric titration methods. The precipitated aluminum tri-hydroxide samples obtained were washed with hot deionised water and dried at 60°C for 24h before observations with Scanning Electron Microscopy (S-3400N). The PSD and average size were measured by laser diffraction (Malvern Mastersizer 2000).

Results and discussion

Effect of technological parameters on particle size distribution

In our study, the question was to understand how all the experimental parameters usually considered in the Bayer process influenced PSD. In order to focus our attention on the effect of other parameters, only one parameter was varied in the experiments. The PSD of the products with different parameters are displayed in Fig. 3. It is found that all particle size distribution curves have a similar shape, but show different trends.

Figure 3(a) shows that PSD moves to smaller particle sizes with decreasing initial precipitation temperature. Fig. 3(b) shows that

PSD moves to coarser particle sizes with decreasing initial molar ratio $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$. The results we obtained are in agreement with

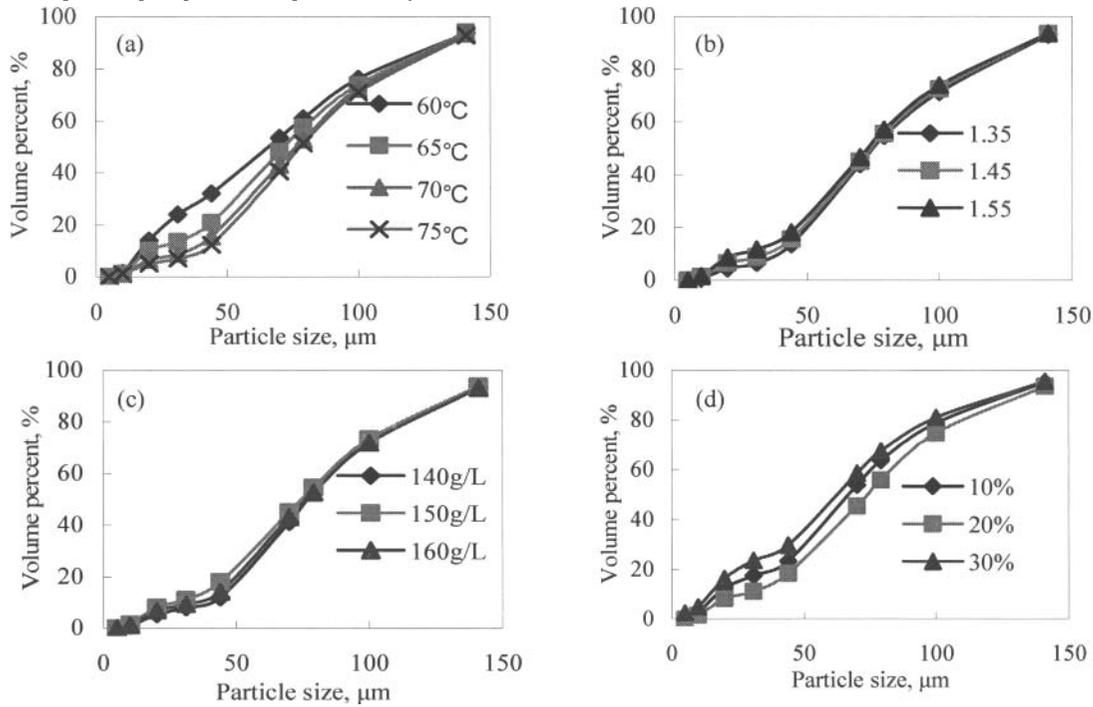


Fig. 1. Variety of Particle size with (a) temperature; (b) molecule ratio of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$; (c) concentration of Na_2O ; (d) fine seed content .

of those of Yamada [15]. Yamada indicates that the agglomeration of $\text{Al}(\text{OH})_3$ particles, for similar supersaturations, is more liable to take place at higher temperatures. He supposes that, at high temperature, the dispersion of the growth species across the particle surface, is faster, and provides a higher probability for the successful cementation of the particles. At the same temperatures, higher initial supersaturations also favour agglomeration, due to the higher concentration of adsorbed growth species. We have worked with fine seeds which are more liable to participate in the agglomeration process. At the same concentration of Na_2O , as the supersaturation of sodium aluminate solution increases, this favours agglomeration as the initial molar ratio of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ decreases.

Fig. 3(a) shows that the PSD moves first in the direction of coarser and then to finer particle sizes with increasing initial concentration of Na_2O . Compared with the initial concentration of Na_2O , the PSD has the opposite tendency with different initial fine seed content. In a seeded precipitation process, the fine particles need more aluminium tri-hydroxide deposition from SAS to transform into coarse particles. However, the seed surface area must respond to the decreasing degree of supersaturation. If not, this is harmful to the agglomeration process. In the tests, the supersaturation of SAS favours agglomeration with Na_2O at 140g/L and that can't match agglomeration with Na_2O at 160g/L. However, with further increase in concentration, the precipitation solution has a higher viscosity, which increases the chance of collision between fine particles, thereby increasing the agglomeration efficiency and reducing fine particle levels. The sodium aluminate liquors in

three fine seed concentration loading tests have the same degree of supersaturation. That is, have the same driving force for seeded precipitation. Therefore, the secondary nucleation takes place with the fine seed content at 10%, and the agglomeration degree decreases with the fine seed content at 30%.

Table1 shows the effects of technological parameters on crystal growth. It is found that when the initial concentration of Na_2O was varied from 140g/L to 160g/L. The volume percentage of product particles below $44\mu\text{m}$ is increased by 6.05% and then decreases by 3.88%. The surface area is increased by $0.015\text{ m}^2/\text{g}$ and then decreased by $0.009\text{ m}^2/\text{g}$. However, the average volume size is decreased by $3.77\mu\text{m}$ and then increased by $1.828\mu\text{m}$. Compared with the initial concentration of Na_2O , the volume percentage of particles below $44\mu\text{m}$, surface area, and the volume average size have opposite variation tendencies with different initial fine seed content.

The volume percentage of particles below $44\mu\text{m}$ and the surface area, decreased with increasing initial temperature. This varied from 32.17% to 12.41% and $0.148\text{ m}^2/\text{g}$ to $0.107\text{ m}^2/\text{g}$ respectively. That is, higher precipitation temperatures favour particle size coarsening. For example, the average volume increased by $11.511\mu\text{m}$ with initial precipitation temperature increase from 60°C to 75°C . The product characteristics therefore show the opposite variation tendency with different α_k compared with initial precipitation temperature.

Table 1. Characteristics of product

Technological parameter		volume fraction of <45 μm / %	volume average size / μm	surface area / m^2/g
N_k	140g/L	12.03	78.171	0.104
	150 g/L	18.08	75.401	0.119
	160 g/L	14.20	77.229	0.110
α_k	1.35	13.65	78.047	0.111
	1.45	15.72	76.223	0.115
	1.55	18.27	75.740	0.120
T	60°C	32.17	66.890	0.148
	65°C	20.92	74.453	0.120
	70°C	15.54	77.016	0.110
	75°C	12.41	78.401	0.107
Fine	10%	23.24	64.683	0.111
	20%	18.38	74.611	0.115
	30%	29.65	62.130	0.120

Note: N_k - concentration of Na_2O ; α_k - molecule ratio of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$; T- temperature; Fine - fine seed content.

Conclusions

A systematic study has been made on the PSD of aluminum trihydroxide from a seeded precipitation process with different parameters. The PSD moved to smaller particle sizes with decreasing initial precipitation temperature, and increasing molar ratio of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$. The volume fraction of particles below 44 μm first increased, and then decreased with increasing initial concentration of Na_2O . The PSD has the opposite tendency with different initial seed amount compared with the initial concentration of Na_2O .

Acknowledgments

The authors are grateful to appreciate the financial support of National Natural Science Foundation of China (NO. 50804031) and the educational department of Liaoning Province of China (NO.L2010395).

References

1. R. Chester, F. Jones, M. Loan, A. Oliveira, and W. R. Richmond, "The dissolution behavior of titanium oxide phases in synthetic Bayer liquors at 90 °C," *Hydrometallurgy*, 96(20093), 215–222.
2. H. Watling, J. Loh, and H. Gatter, "Gibbsite crystallization inhibition 1. Effects of sodium gluconate on nucleation, agglomeration and growth," *Hydrometallurgy*, 55(2000), 275–288.
3. F. Farhadi, and M. B. Babaheidary, "Mechanism and estimation of $\text{Al}(\text{OH})_3$ crystal growth," *Journal of Crystal Growth*, 234(2002), 721-730.
4. N. Brown, "A quantitative study of new crystal formation in seeded caustic aluminate solution," *Journal of Crystal Growth*, 29(1975), 309-315.
5. Z. Wang, S. W. Bi, Y. H. Yang, and Z. F. Yuan, "Evolution of particle size and strength of hydragillite from carbonization in seeded sodium aluminate liquors," *Journal of Crystal Growth*, 274(2005), 218-225.
6. A. M. Paulaime, I. Seyssiecq, and S. Veesler, "The influence of organic additives on the crystallization and agglomeration of gibbsite," *Powder Technology*, 130(2003), 345-351
7. H. LI, J. Addai-Mensah, J. C. Thomas, and A. R. Gerson, "The crystallization mechanism of $\text{Al}(\text{OH})_3$ from sodium aluminate solutions," *Journal of Crystal Growth*, 279(2005), 508-520.

8. I. Seyssiecq, S. Veesler, D. Mangin, J. P. Klein, and R. Boistelle, "Modeling gibbsite agglomeration in a constant supersaturation crystallizer," *Chemical Engineering Science*, 55(2000), 5565-5578.
9. Y. S. WU, D. Zhang, M. C. Li, S. W. Bi, and Y. H. YANG, "Periodical attenuation of $\text{Al}(\text{OH})_3$ particles from seed precipitation in seeded sodium aluminate solution," *Transactions of Nonferrous Metals Society of China*, 20(2010), 528-532
10. C. B. Zhang, and P. Zhao, "Studied on polarizing particle size of aluminate hydroxide in Bayer process," *Light Metals*, (1999), 17-19. (In Chinese)
11. G. Zhang, and J. N. Yang, "Studied on particle size fluctuating of aluminate hydroxide in Bayer process," (2002), 9-12. (In Chinese)
12. B. Garner, B. Cristol, and A. Soirat, "Precipitation particle size control," *Light Metals*, (1999), 71–76.
13. B.K. Satapathy, and T. Padhi, "Determination of grain-size distribution of sandy alumina using electron sensing zone method," *Light Metals*, (1990), 185-191.
14. T. K. Hunter, G. M. Moody, and S. E. Sankey, "Advances with chemical additives for the alumina industry," *Light Metals*, (1991), 159-162.
15. K. Yamada, "Nucleation and agglomeration during crystallization of aluminium trihydroxide in sodium aluminate solution," *Light Metals*, 32(1980), 720-726.