

PARTICLE STRENGTH OF BAYER HYDRATE

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Because of the proposed use of fluid bed calciners at the Kaiser Aluminum Baton Rouge Works, studies into the strength of alumina and alumina trihydrate from eight different alumina plants were initiated. It was found in the course of these studies that the particle strength of Bayer hydrate depended on the precipitation process conditions under which it was produced. A series of laboratory precipitation tests was conducted to determine the effect on particle strength of process variables such as seed charge, temperature, caustic concentration and seed recycle. It is concluded from these studies that relative particle strength of alumina trihydrate, as measured by a modified Forsythe-Hertwig Apparatus, can be predicted from a knowledge of the precipitation process conditions.

1. INTRODUCTION

Kaiser Aluminum & Chemical Corporation first began studying the strength of alumina to determine how to reduce handling losses and atmospheric dust emissions in both the Bayer plants and reduction works. The dusting properties of aluminas were studied and several processes for producing a non-dusty alumina were examined.

Because of the proposed replacement of the rotary kilns at the Baton Rouge Works with stationary calciners, the emphasis of the studies was changed from the control of dust after calcination to the control of fines generation prior to and during calcination. Using a modified Forsythe-Hertwig apparatus to measure relative strength, a survey of the friability of alumina trihydrate and calcined alumina from several alumina plants in the Kaiser and other systems was made. Results of this survey suggested that differences in hydrate strength could be related to differences in source precipitation processes.

A series of laboratory precipitation tests were made to study the effects of precipitation conditions and proposed process modifications on the relative strength of Bayer hydrate.

It is the purpose of this paper to describe the results of the survey and the test series.

2. EXPERIMENTAL METHODS

Two laboratory procedures were used in the survey and the subsequent laboratory test program.

LABORATORY ATTRITION TEST

In the modified Forsythe-Hertwig laboratory attrition test used to measure the strength of hydrate and alumina, samples are subjected to a high velocity air-jet, causing particle breakdown by autogenous grinding of the particles. Wet screen analyses made before and after the test give a measure of the strength of a sample relative to other samples.

Hydrates and aluminas usually are analyzed in the USA by a conventional dry sieving technique. Unfortunately, hydrate and, more particularly, alumina fines agglomerate before and after attrition and are not sieved properly in a dry procedure. Because of this, these samples were analyzed by wet screening before and after attrition.

In the plants survey in this report, both wet and dry sieve analyses are reported. Although methodology is not the subject of this report, when comparing this work with that of others, the influence of the method of analysis on results should be kept in mind.

The results in this paper are reported as Attrition Index. Attrition Index is defined as follows:

$$AI = \frac{(X-Y)(100)}{(X)} \quad (1)$$

X Where:

- X = % +325 Before Attrition Test
- Y = % +325 After Attrition Test

A sketch of the test apparatus is shown in Figure 1.

LABORATORY PRECIPITATION TESTING

A precision laboratory precipitation test was employed to study the effects of process variables on the strength of alumina trihydrate precipitated from plant and synthetic Bayer liquor. In this test, seed and pregnant liquor are weighed into 1-liter wide-mouth bottles. These bottles (up to 10) are placed in a water bath fitted with a temperature controller that provides a pre-programmed temperature change rate as desired. The samples are mixed by end-over-end rotation during the precipitation time. At the end of the desired holding time, the bottles are removed and rapidly cooled in cold water. The solids and liquor are separated by filtration and reserved for chemical and physical analysis.

Data from this laboratory precipitation test can be used to predict yield, product quality and precipitation curves with accuracy and precision. Replicate testing of a ten bottle series gave the following results:

TABLE I

REPLICATE SAMPLE VARIATION

PRECIPITATION BOTTLE - TEST

Liquor Final A/C	Hydrate Analyses			
	+200 M %	+325 M %	-325 M %	Surface cm ² /gm.
Average	0.394	39.0	89.4	1119
Standard Deviation	.002	.9	1.5	39

A comparison of results from a bottle-test with those from a parallel, full-scale precipitator is as follows:

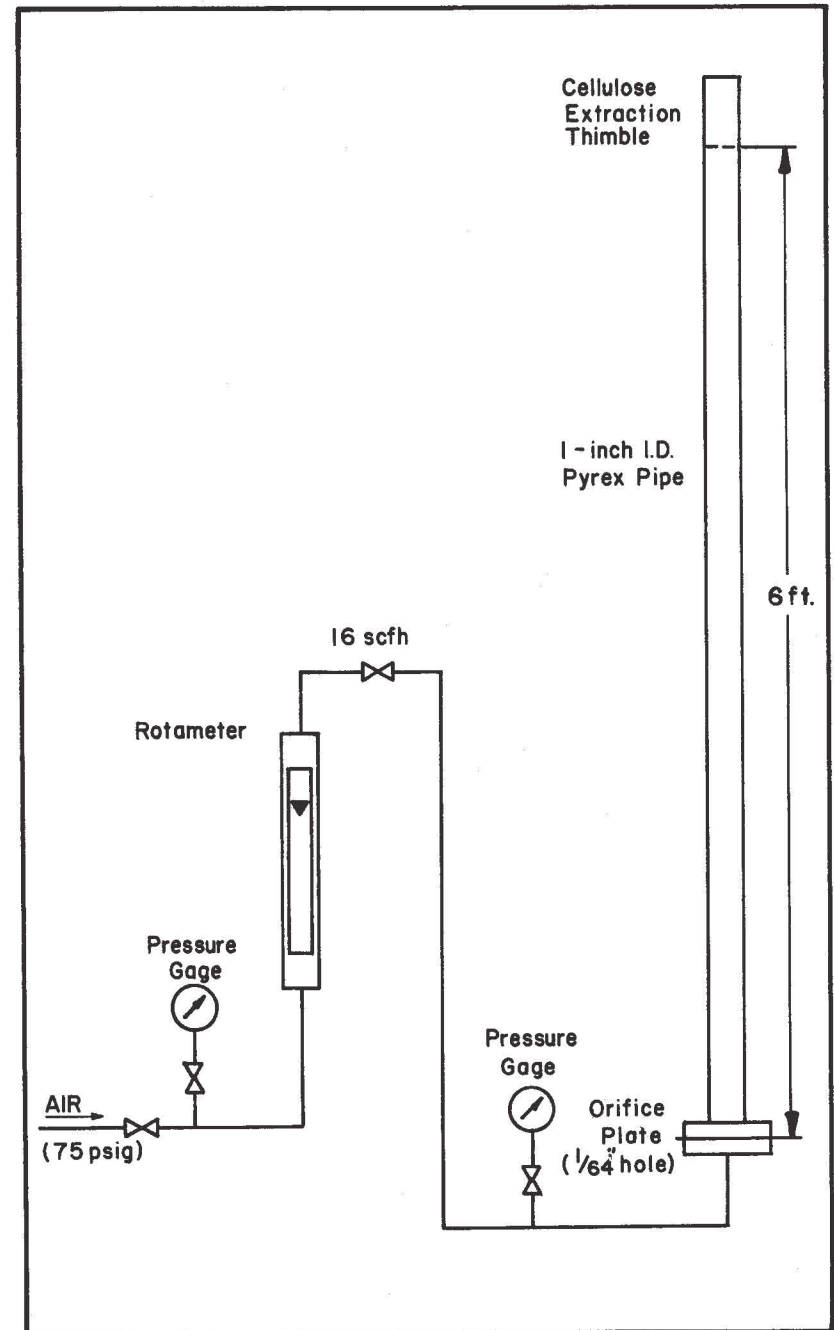


FIGURE I - ATTRITION APPARATUS

TABLE II

RESULTS COMPARISON

"PUMPOFF ANALYSIS"

PLANT AND BOTTLE-TEST

	Plant Precipitator 3	Laboratory Test
Solids Concentration, %	14.8	14.6
Liquor Final A/C	0.400	0.396
<u>Hydrate Properties</u>		
% +325 Mesh	82	81
Fisher Surface Area, cm ² /gm	775	738
Attrition Index	19.7	19.9

3. ALUMINA PLANTS SURVEY

Hydrate and alumina samples from various Kaiser and other Bayer plants were analyzed for particle size and attrition strength.

Table III shows the data from these eight alumina plants. Tentative conclusions drawn from comparison of the data are as follows:

- Hydrate and alumina strength varies between plants even with near identical process equipment. It is tempting to conclude from comparison of Plants "A and B" with "C & D" that the aluminas from batch precipitation plants are stronger than those from continuous precipitation plants. The apparent attrition strengths of their hydrates are similar. The product (aluminas) strengths, however, are quite different. On the other hand, Plants "E and H" produce strong aluminas from continuous precipitation. Plant "G" produces a strong hydrate (batch) and a strong alumina.
- Alumina strength is a function not only of hydrate strength but also is related to the complete size distribution of the hydrate and to the method and conditions of calcination as well. All plants use the coarsest hydrate that their operations can supply as kiln feed to minimize fines input. Usually kiln feed is at least 93-98% plus 325 mesh (45 micron) but kiln feeds with the same plus 325 mesh can have different size distributions and average particle sizes. How the strength of a hydrate is related to these factors is not known, but is a subject for further study.

TABLE III - HYDRATE AND ALUMINA STRENGTH
SUMMARY OF SOME SURVEYS

PLANT	PRECIPITATION PROC. TYPE	CALCINATION KILN TYPE	KILN FEED			PRODUCT ALUMINA		
			% +325 M	AI _D	AI _W	% +325 M	AI _D	AI _W
"A"	Batch	Rotary	98	5	10	95	23	32
"B"	"	"	95	6	12	92	17	24
"C"	Continuous	"	94	5	11	89	35	46
"D"	"	"	98	6	--	96	30	36
"E"	"	Fluid Bed	9	6	--	90	11	17
"F"	"	Rotary	97	6	--	93	35	44
"G"	Batch	Fluid Bed	98	1	3	92	11	17
"H"	Continuous	Rotary	--	-	--	90	12	17

AI = Attrition Index Sub D = Dry Screen Sub W = Wet Screen

Additional tests were made of hydrate strength within the precipitation operations in three of the eight Bayer plants. The results of these surveys are summarized in Table IV.

TABLE IV

HYDRATE STRENGTH WITHIN PRECIPITATION

Plant	Process	Made from Coarse (Secondary) Seed		Made from Fine (Tertiary) Seed	
		% +325	AI _D	% +325	AI _D
A	Batch	96.8	5.8	98.7	25.3
B	Batch	92.7	5.2	96.8	23.3
C	Continuous	92.5	2.1	83.3	11.7

It was found that the strength of the coarse hydrate produced from a fine (tertiary) seed was four to five times weaker than that of a hydrate produced from a coarse (secondary) seed. The laboratory precipitation tests that followed were initiated because of these results.

4. LABORATORY TEST PROGRAM

The alumina plants survey showed that a product from a precipitator charged with coarse seed was 4-5 times stronger than a product from one charged with fine seed. This indicated that hydrate strength may be related to the agglomeration of fine particles. The next phase of the work was to define the effect of precipitation variables such as seed type, temperature, caustic concentration and holding time on the strength of the precipitated product.

Since plant scale tests are difficult to control and perform, a series of laboratory tests was undertaken in conjunction with another precipitation project, using the procedures described in Section 2.

In this other precipitation project, strength was but one aspect of a much larger study of hydrate agglomeration and growth. Because of this, data were collected over a wide range of process conditions. These were:

Caustic Concentration	165 - 250 gpl as Na ₂ CO ₃
Initial Temperature	155 - 175 °F
Seed Charge	35 - 280 gpl Solids
Seed Specific Surface Area	500 - 1200 cm ² /gm (Fisher)
Holding Time	1 - 36 Hours
Cooling Rate	Isothermal to 1.50°F/hr
Liquor Contamination	Pure Caustic to Plant Liquor

Initial analysis of the data from the plants survey and laboratory tests indicated a strong relationship between the amount of agglomeration achieved in a precipitation cycle and the strength of its precipitated product.

A simple agglomeration measure, analogous to the Attrition Index, was developed to correlate the data. This relationship is:

$$\text{Agglomeration Index} = \frac{(A - B)(100)}{(B)} \quad (2)$$

Where:

- A = % +325 mesh of precipitated product
- B = % +325 mesh of the seed

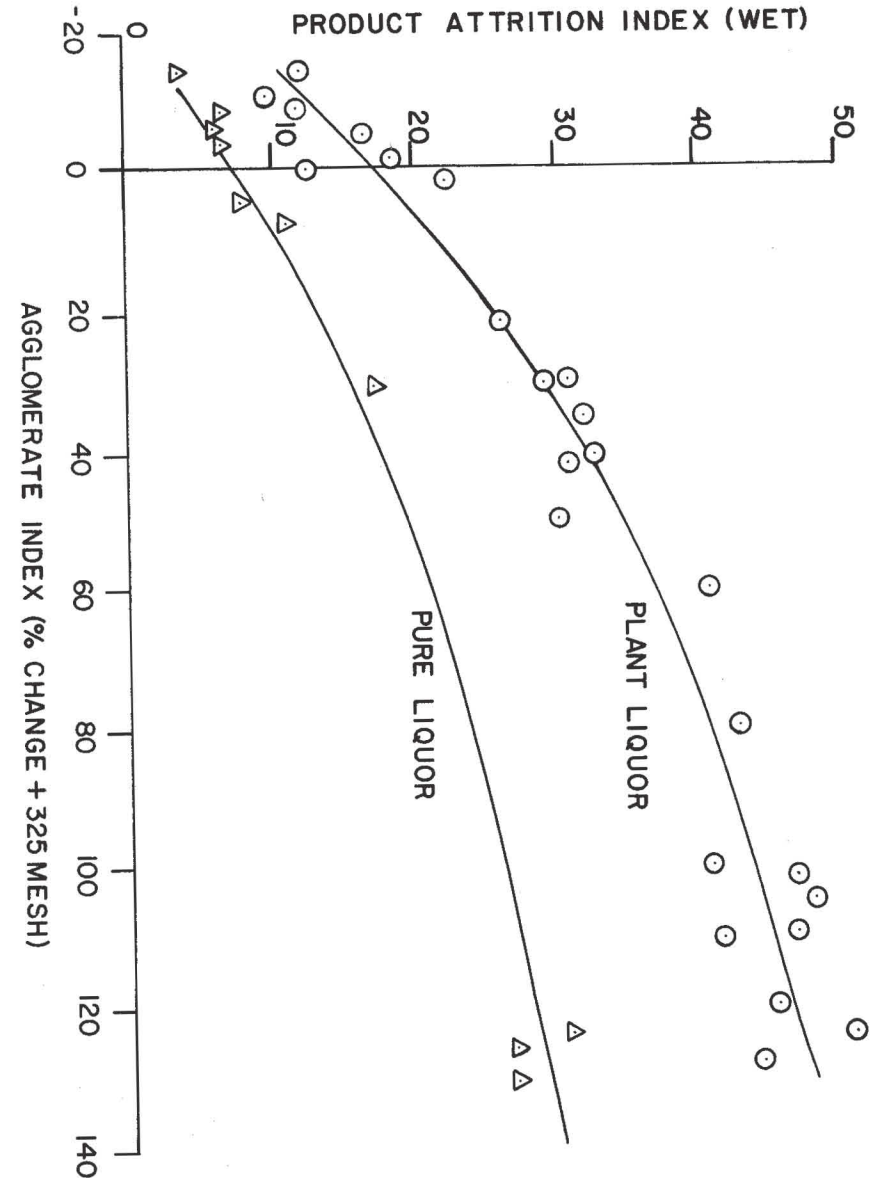
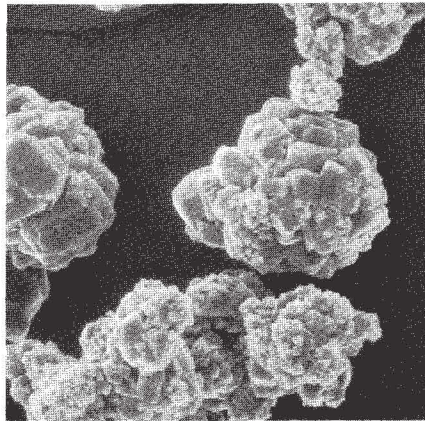
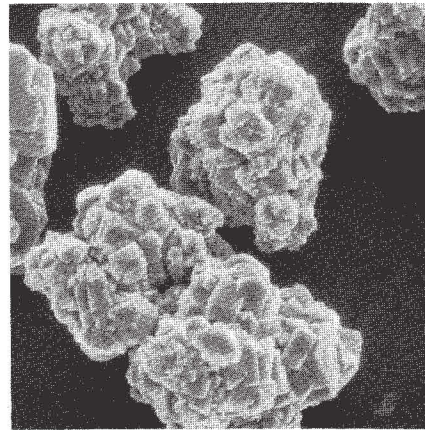


FIGURE 2 - AGGLOMERATE FRIABILITY ATTRITION INDEX (WET) VS AGGLOMERATE INDEX



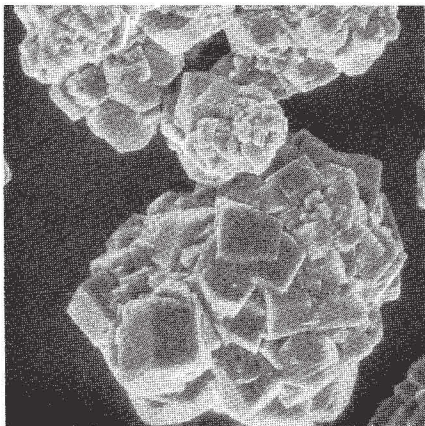
AGGLOMERATED PARTICLES
AGGLOMERATION INDEX = 50
ATTRITION INDEX = 30

A



STRENGTHENED PARTICLE
ATTRITION INDEX = 12

B



FINAL PRODUCT
ATTRITION INDEX = 3.5

C

20 MICROMETERS

FIGURE 3 - LABORATORY PRECIPITATED HYDRATE 500X

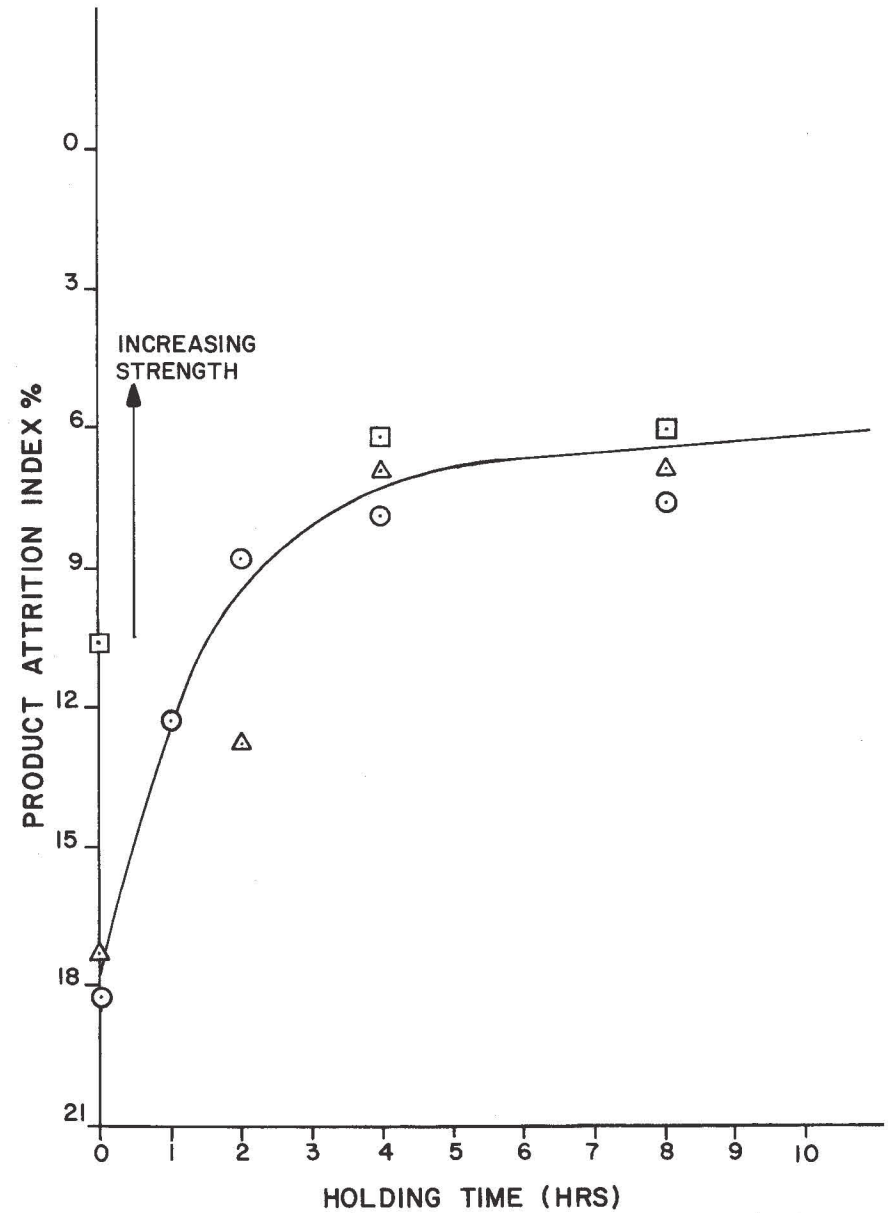


FIGURE 4 - STRENGTHENING BY GROWTH ATTRITION INDEX (WET)
VS
HOLDING TIME

Figure 2 is a plot of Attrition Index versus Agglomeration Index.

Although an equation of relationship was not fitted, it can be seen that hydrate attrition increases with increasing agglomeration over a wide range of agglomeration conditions. Data from all these tests fall near one curve for all conditions except one. A significant difference was found between the attrition of hydrate produced from plant (contaminated) liquor and that produced from pure liquor. In all cases the hydrate produced from pure liquor is stronger than that from plant liquor. Additional studies are required to define the relationship between liquor purity and agglomerate strength.

Scanning Electron Microscope (SEM) photos of large agglomerated particles give a qualitative insight into particle strength. Figure 3A shows typical agglomerates with an Attrition Index of 30 from an eight-hour precipitation test designed to produce large particles. The Agglomeration Index for this hydrate was 50 (96% +325 Mesh). As can be seen the particles are large but are also angular and contain voids. It is not difficult to visualize that these particles would be relatively easy to break during handling and calcination.

The hydrate shown then was used as seed in another precipitation test. The conditions of this test were such that growth of the crystal was the predominant reaction. Figure 3B shows the product after one hour of precipitation time. The Attrition Index had dropped to 12. The angularity has been reduced and the voids filled in. Figure 3B shows the product after an additional 23 hours of growth. The final Attrition Index was 3.5.

The Attrition Index of the hydrate from precipitation tests designed to promote growth (as opposed to agglomeration) were plotted versus holding time in the precipitators. Figure 4 shows that over 80% of the later strength of the particle will be achieved in the first 4 - 6 hours of the precipitation growth cycle.

5. CONCLUSIONS

Conclusions which can be drawn from the plant survey and Laboratory Test series are as follows:

1. Alumina strength is a function not only of the strength of hydrate but is also related to the size distribution of the hydrate and the method and conditions of Calcination.
2. Hydrate strength can be related to the precipitation process conditions under which it was produced. This strength is inversely related to the amount of agglomeration of fine particles achieved in the precipitation cycle.
3. Hydrate produced from pure liquor is inherently stronger than that produced from contaminated plant liquor.
4. Hydrate can be strengthened in 4-6 hours by establishing the precipitation conditions in which growth is the dominant reaction.