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PITCHBLEND PROCESSING FILTRATION DATA

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3-14, 1945

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Object

To collect filtration data on relatively large batches so that the type and size of filtration equipment needed for plant scale operations of the 42-15 process can be decided upon.

General

Examination of the filtration data collected by Yale personnel before April 3 disclosed this information to be incomplete and subject to large variation.

The filtration rates on gangue from digestions 212 to 215 varied from 18.1 to 3.3 gallons per square foot per hour for hot filtrations (about 80°C) and 0.48 to 2.45 gallons per square foot per hour for cold (about 20°C) filtrations. These experiments were carried out on Nuches with vacuums ranging from 26-60 centimeters of mercury.

In filtering the lead sulphate cake, the practice of decanting supernatant liquor and filtering separately was followed in the above digestions. The rates varied from 12.4 to 133 gallons per square foot per hour for the supernatant fluid and 16.1 to 48.3 gallons per square foot per hour for the slurry. These filtrations were carried out at room temperature on Nuches. The vacuums ranged from 40-60 centimeters of mercury.

Filtration data on barium sulphate on digestions 212 to 215 was very sketchy. The decantation practice was followed. The rates varied from 0.621 to 39.4 gallons per square foot per hour. No vacuum or temperature data were recorded. After considering the above Yale data and other factors, a 100-pound 2x batch was decided upon. The data accumulated on this digestion and a subsequent 1.3x 60-pound digestion are given here.

Discussion

From the data collected to date, it appears that large areas will be required to filter gangue within a reasonable length of time and not adversely affect the My distribution.

With the fixing of the process it may be possible to experiment and determine methods of speeding up the rate of gangue filtration by varying digestion conditions. It must be kept in mind that the majority of filtration problems are solved by varying conditions in the tanks before the filter.

The lead sulphate filtration was very rapid and probably could be easily carried out within a bottom discharge centrifugal.

The barium sulphate filtration was relatively easy and offers no great problems. Our own experience indicates a rotary driven filter will prove satisfactory.

If it becomes necessary to include a sulphide precipitation, the consequent filtration could easily be done with an Oliver Precoat filter.

Program

Following the fixing of the process, the following steps will be taken:

- A. Determine how the gangue filtration rates can be increased and what type of filter to install. Indications are that the temperature greatly affects the filtration rate.
- B. Observe whether an Oliver can be used for lead sulphate filtration by increasing the turbulence within the Oliver casing.
- C. Obtain Oliver rate data on Barium Sulphate and determine what type of filter will be best suited to this operation, possibly an American Continuous Rotary Filter.

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PROBLEM: Filtration - Gangue

Experiment No. XVI

Date: 4-5-45

One hundred pounds of ore were digested for four hours with 2 x nitric acid. The liquor was allowed to boil down and water was not added until the end of the digestion. The digestion temperature was 95° C and the final volume 30.3 gallons.

Since no data was available on the cake density under the conditions of this experiment, the Sperry S.S. press was set up with five frames. Available volume ~~4.7~~ ^{0.26} ft³, area ~~6~~ ^{6.18} ft².

Glass cloth was used, the edges being sealed with Permatex No. 2 gasket material. (Style Q189 from Filter Media Corporation.)

Hot slurry (85° C) was allowed to enter the press by gravity and the liquid went through the press rapidly giving a brilliant filtrate. When the centrifugal was turned on, the filtrate clouded and would not come clear. The press was drained and opened. The cake formation was uniform, slimy in character, and about 1/8 to 1/4" thick. The cloth was ruptured on the edges. This cloth had been weakened previously by testing with water in the press using no gasketing material to prevent crushing action. The press did not leak during the run.

The press was fitted with duck and a second attempt was made to filter. The filtrate did not come brilliant even with recycling. Cake formation was good, about 1/4" thick and appeared fairly dry. The liquor was allowed to flow in by gravity for 3 minutes and then the centrifugal was turned on. Head pressure 10 psig. General small defects were found in the cloth, but the reason for the failure was not apparent.

- a. One frame was fitted up with cotton duck and the ore slurry was stirred with a 1/4 HP stirrer. The liquor was admitted to the press by gravity, but no clarification was observed even after prolonged recycling.

The press was broken but no defects were found in the cloth.

- b. One frame was fitted up with glass cloth, the edges being sealed and protected with Permatex No. 2 gasketing material. This filtration was successful and the filtrate was brilliant. The frame was full when opened. This frame was allowed to remain in place and four more frames were fitted up with glass cloth and Permatex for the next experiment.

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The five frames used in the previous filtrations were used to filter the balance of the slurry. The gravity feed was not used since the cloths were already coated.

Time Min.	Total Volume Gallons	Pressure lbs./in ²	Time Seconds	Volume Gallons	Flow GPM	Filtra- tion Rate Gal./hr 1 ft ²	
20		30	30	0.277	0.332	3.22	Recirculated 20 min. until filtrate cleared.
25	3.50	30	30	0.179	0.358	3.48	
30	5.25	30	30	0.150	0.301	2.92	
35	6.75	30	30	0.145	0.290	2.82	
40	8.00	30	30	0.132	0.264	2.56	
45							
51	10.00	30	30	0.145	0.240	2.82	51.15 pump stopped five minutes.
55	11.25	30	30	0.124	0.248	2.41	
OW	72	-	30	0.124	0.248	2.41	
1OW	85	4.5	30	0.129	0.258	2.50	
22W	97	6.5	30	0.132	0.264	2.56	
3OW	102	9.2	30	0.132	0.264	2.56	

Ten minutes air blown.

10.5 pounds of gangue collected.

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Experiment No. XVI
Date: 4-6-45

c.

Filtration
Rate
Gal/hr/ft.²
~~R~~

Time Min. T	Volume Gallons V	Pressure lbs./in ² P	Time Minutes ΔT	Volume Gallons ΔV	Flow Gal./hr F	Filtration Rate Gal/hr/ft. ² R	Remarks
6	-						Gravity until 6 minutes - wash side turbid - closed off
9.75	-	5	3.0	0.03	1.80		
15	1.2	10	1.0	0.198	8.88	3.64	Pressure to 10
20	2.5	10					" " 20
25	3.5	20	1.0	0.142	11.50	4.66	
30	4.5	20	1.0	0.140	8.40	3.40	Filtrate 54
36	4.75	20	1.0	0.119	7.15	2.89	
41	5.75	20	1.0	0.107	6.42	2.60	4th Plate shows
46	6.75	20	1.0	0.113	6.78	2.74	slurry leakage
55	7.0	20					
60	7.5	20					
61	7.6	20	1.0	0.100	6.00	2.43	
66	7.8	20	1.0	0.084	5.04	2.10	
71	8.5	30	1.0	0.125	7.44	3.01	Changed to 30 lbs.
78		30	1.0	0.106	6.36	2.57	
88	17.1	30	1.0	0.095	5.70	2.31	
100	18.0	30	1.0	0.087	5.22	2.11	
105	18.5	30	1.0	0.079	4.74	1.92	Changed to 40 lbs. at 106
109	18.8	40	1.0	0.110	6.60	2.67	
117	19.5	40	1.0	0.091	5.46	2.21	
123	20	40	1.0	0.079	4.74	1.92	Filtration stopped
W 135		55					
1OW 145	2.5	55	1	0.178	10.7	4.34	Sampled each wash - T analysis
2OW 155	4.0	55	1	0.174	10.4	4.21	
3OW 165	5.75	55	1	0.162	9.72	3.93	
4OW 175	6.75	51	1	0.161	9.65	3.90	

Cake air blown
for ten minutes

Density of gangue 98 ± 2 lbs./ft³

Percent H₂O 56%

Specific gravity of slurry 1.354

Viscosity of filtrate 0.890 centipoise

Filtration area 4.94 ft.² 4 plates

Volume 0.0515 ft³ 4 frames

Filtration temperature 72°C $\rightarrow$ 54°C

The cake formed in the frames at the end of the filtrate was fine in texture, completely filled the frame, and buff colored. Excellent cake, firm and easily removed.

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PROBLEM: Filtration of Gangue -
1.5X digestion
Sperry Filter Press

Experiment No. XIX
Date: 4-14-45

Time Minutes T	Volume Gallons V	Pressure lbs./in ² P	Time min. Δ T	Volume Gallons Δ V	Flow Gals/hr F	Filtration Rate Gals/hr 1 ft. ²
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S.S. cloths -
Temp. 82°C. 9 frames
recirculated
23 minutes at 20
p.s.i.g. - turbid -
up to 50 p.s.i.g.
5 min. - turbid 28
back to 20 p.s.i.g.
no clarity

31.0	-					
32.0	-	20	0.5	0.0460	5.52	2.23
35.0	-	20	0.5	0.045	5.40	2.18
53.5						

no brilliance
filtration stopped-
frames full

0	0					
5.0	0.5	32	1	0.102	6.12	3.30
16.0	1	35	1	0.0988	2.93	1.58
30	1.5	35	1	0.0304	1.83	0.988
45	2.7					

Recirculated 4
min. - cotton duck
3 areas-temp. 60°C

Filtration stopped-
frames about 1/2 full

6		25	0.5	0.129	15.50	
12.5	2.6	24	0.5	0.0805	9.65	2.23
18	3.4	25	0.5	0.0661	7.92	1.83
25	4.2	26	0.5	0.0554	6.65	1.54
31.5	5.0	25	0.5	0.0462	5.53	1.28
38	5.6	26	0.5	0.0423	5.07	1.17
44.5	6.0	26	0.5	0.383	4.59	1.06
51	6.4	26	0.5	0.0370	4.43	1.02
63		60				

Recirculated 3 min.
7 area temp. 38°C
cotton duck

Notes: Specific gravity of slurry 1.435
Density of wet cake 100 lbs./ft³

No washing was attempted since great emphasis was placed on completing
the filtration.

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Experiment XVI
Date: 4-7-45

PROBLEM: Filtration of Lead Sulphate - 1.

One frame was used in the Sperry plate and frame press. This gave a total filtering area of 1.236 sq. ft., volume, 0.0515 ft³. The filter medium was FMC style 0184 glass cloth sealed with Permatex No. 1.

Time Min. T	Total Volume Gallons V	Pressure PSIG P	Time differential Sec. Δt	Volume differential gal. Δv	Gal. per min. $\left(\frac{\Delta v}{\Delta t}\right) 60$	Gals. per hour per sq.ft.	Remarks
6.7	0						Recirculated liquor by gravity until clear
9	10	grav.*	30	0.85	1.7	82.8	Brilliant filtrate
14.5	19	grav.					receivers changed
17.0		5					Pump started at 5 psig
18.5		10					filtrate turbid
24.0	0	10					started recirculating
24.5	0	10					Recirculation stopped.
							Filtrate brilliant,
							collection started.
26	6.0	10	15	.808	3.23	157	
30	16.0	10	20	.961	2.88	140	
33	23.0	10					Stopped to change receivers.
36.3	4.5	10					
37.5		10	10	0.314	1.88	91.3	
38.5	9.2	10					End of filtration
56.5		20					Wash started
57.3		20					
57.5	5.75	20					Wash water -water white

Cake air blown for 11 minutes.

Cake weight 7 pounds, density 170 lbs./ft³, 18.2% water
Frame filled to a depth of 8-1/8".

Total filtrate 51.2 gal., Avg. rate 166 gal./ft²/hr.

*Note: Gravity head equal to 3 ft. of soln.

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Experiment XVI
Date: 4-9-45

PROBLEM: Barium Sulphate - filtration

Initial filtration area equal to six plates - reduced to 3 during run.
Filter medium cotton duck.

	Time Min.	Volume Gal.	Pressure lbs/in ²	Time Sec.	Volume Gallons	Rate gals/ft ² /hr.	
First filtration	6.5	0	5				Recirculated liquor until clear 6.5 min.-wash side wouldn't come clear. Filtration temp. 60°C
	10	5.7	21	15	0.264	34.1	
	14.5	9.7	21	15	0.211	27.3	
	18.5						Filtration stopped to test wash side -30 sec. delay
	20.0	13.0	21	15	0.153	19.8	
	25.0	14.7	21	15	0.121	15.7	
	30.0	18.0	21	15	0.116	15.0	
	36.0	19.5	21	15	0.106	13.7	Changed receivers at 37.0
	42.0	2.75	21	30	0.174	11.2	
	47.0	4.00	21	60	0.327	10.6	
	50.5	5.25					Filtration ended - frames full
W	4		54	30	0.189	12.2	Used water turbid - clear of yellow color in seven min. but remained turbid.
W	7	2.75					
Ten minute air blow							
Second Filtration	7.5		21				Recirculated 7.5 min.
	9.0	3.0	21	15	0.375	72.8	Filtrate slightly turbid throughout filtration
2 frames	15.0	9.7	21	15	0.211	41.0	
used area	20.0	13.5	21	15	0.161	31.3	
equal to 2 plates	27	17.2	21	15	0.135	26.1	
W	11	4.75	53				Wash water again turbid.

First cake weighed 18.5 pounds. Cake density 120 pounds per cubic foot.
Second cake weighed 9.5 pounds - frames only about 1/3 full - cake was
thick at bottom narrow at the top.

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Experiment No. XVI
Date: 4-10-45

PROBLEM: Molybdenum Sulphide Filtration

Cotton duck filter medium. Precipitation and filtration carried out at room temperature.

Time Min.	Volume Gallons	Pressure lbs/in ²	Time t min.	Volume v gal.	Flow gal/min	Filtration rate gals/ ft ² /hr.	Remarks
0	0						3 minutes gravity feed 3 minutes recirculated with pump
8	0	25					Filtrate clear on one side -
9.5		25	1	0.119	0.119	11.6	filtration started - one
15.0		24	1	0.109	0.109	10.6	plate used.
21.5		24	1	0.120	0.120	11.6	
25.5		24	1	0.128	0.128	12.4	
28	2.5	23					
30	2.7	23	1	0.127	0.127	12.3	
32		23	1	0.119	0.119	11.6	
38.5	3.7	23	1	0.119	0.119	11.6	
45	4.3	23	1	0.111	0.111	10.8	
52	5.2	23	1	0.103	0.103	10.0	
61	6.5	23	1	0.094	0.094	9.1	
67	6.7	23	1	0.089	0.089	8.6	
75	7.5	23	1	0.082	0.082	8.0	
80	7.7	23	1	0.082	0.082	8.0	
90	8.75	23	1	0.077	0.077	7.5	Filtration stopped - (cake very slimy and rubberlike 1/4"-1/16" thick) wash started - turbid
OW		55					
5W	5	55	1	0.177	0.177	17.2	
8W	8	55					Wash clear and water white.
0	0	0					New cloths liquor recircu- lated 10 min. - 2 plates used
11	-	11	0.333	0.198	0.597	29.0	
19	5.2	12	0.333	0.116	0.348	18.6	
25	6.7	12	0.333	0.101	0.303	14.7	
	10.7	12					Clock stopped filtration time not known.
OW	0	-	35	0.216	0.648	31.5	Wash started
9W	9	13					Wash clear and water white

In both filtrations air was blown thru press for about ten minutes. Resulting cake was thin, black, tar-like material.

Initial volume 23.7 gals.

PROBLEM: Filtration of Gangue

Experiment No. XIX

Date: 4-14-45

Oliver Continuous Rotary Filter equipped with SS. 20 x 350 cloth. No washing attempted.

Time Min.	Drum speed MPR	Vacuum In Hg	Gallons filtrate	Pounds wet cake	Temp. slurry	Rate ² gal. fil- trate /ft ² /hr.	Rate ¹ lbs.dry cake/ft ² /hr.	
0	4	10			82°C			Cake very thin 1/32" broke fairly clean. Air blow seems to leak into vacuum and blow fil- trate back making cake very wet. Filtrate turbid
16	4	2						
18	4	4			80°C			
27	4	12						
31	4	3						
36	4							
	4	6.2 avg	0.408			1.36		
37	4	19						Air blow and wash vacuum off
38.5	4	15						
42	4	18			38°C			
47	4	15						
56	4	20			35°C			Cake barely covers wires Filtrate turbid
60	4	17.4 avg	0.158	0.132		0.77	0.28	
62	4				32°C			Cake uniform dry - no cracks
69	4	18			33°C			
70	4	10						
72	4	16						
74	4	16			31°C			
76.5	4	18						
80.5	4	8						Filtrate turbid
		14.3	0.075	0.077		0.45	0.20	
90	4	12						Filtrate turbid
95	4	14			30°C			
96	4	18						
100	4	18	0.098	0.066				
		15.5	0.098	0.066		1.10	0.32	
104	4	15						
108	4	17						
112	4	18						
120	4	18						
123	4	18						
130	4	18						
143	4	18						
			0.592	0.177		1.62	0.20	

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Experiment No. XIX
Date: 4-14-45

-continued-

Time Min.	Drum speed MPR	Vacuum in. Hg	Gallons filtrate	Pounds wet cake	Temp. slurry	Rate ² gal. fil- trate /ft ² /hr	Rate ¹ lbs. dry cake/ft ² /hr.
149	4	8			55		
155	4	13					
158	4	18			47		
160	4	14					
165	4	16			42		
167	4	15					
168	4						
	4	14 avg.	1.01	0.0925		5.97	0.236

Cake moisture 56.9 70

Oliver - PbSO₄

Too little slurry - insufficient agitation.

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