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Majid, A.; Sparks, B. D.

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A Rapid Method for Evaluating Particle Sedimentation in Aqueous Suspensions

A. Majid and B.D. Sparks

Submitted By / Soumis par: [signature] — Project Leader / Chef de projet

Approved By / Approuvé par: Edward Capes Mar 4/94 — Program Head / Dirigeant de programme

Approved By / Approuvé par: [signature] 6/3/94 — Director General / Directeur général

Ottawa, Canada
K1A 0R6

Docufax: (613) 941-2529

Canada

EXECUTIVE SUMMARY

OBJECTIVE

Use of centrifugation to develop a rapid dewatering and consolidation evaluation test for particulate suspensions.

ACHIEVEMENTS

- A significant reduction in time to evaluate the dewatering and consolidation behaviour of treated as well as untreated fine tailings samples.

TECHNIQUE DEVELOPMENT

- A faster test to study the dewatering and consolidation of particulate wastes, such as oil sands fine tailings, was developed using centrifugation.

HIGHLIGHTS

- A fast test to evaluate the dewatering and consolidation behaviour of Athabasca fine tailings was developed using centrifugation to increase settling and consolidation rates.

- At a fixed centrifugal G force, the rate of dewatering was proportional to the time for centrifugation.

- Dewatering data for primary fine tailings samples, obtained using centrifugation at 1500 x G, produced a first order correlation with the corresponding data from 200 days of gravity settling. The dewatering data beyond 200 days of gravity settling may be represented by a different first order constant.

- Comparable amounts of water were obtained for both Syncrude and Suncor fine tailings samples in a matter of minutes using centrifugation compared to months for gravity settling.

- Comparison of the gravity settling results for several treated and untreated samples of fine tailings, from both Syncrude and Suncor, with the corresponding data from centrifugation tests, suggests that, each type of sample needs to be calibrated in terms of its settling conditions, using the two test procedures, in order to produce valid results.

- Compared to the differences in the amounts of water released by the two test procedures, the solids content of the sediments obtained by the two methods were very similar for most of the samples. This suggests that the centrifugation test, gives results similar to those from gravity settling tests for sedimentation/consolidation studies, but may not give comparable results for clear water release.

- Filtration tests correlated poorly with gravity settling results. However, this approach may be useful in evaluating the dewaterability of different samples by filtration techniques, such as filter presses.

RECOMMENDATIONS AND FUTURE WORK

Future work will concentrate on the application of this new test to particulate wastes from other industries.

ABSTRACT

Gravity sedimentation tests to study the dewatering and sedimentation behaviour of fine tailings, can take months to complete. We have developed a faster test to evaluate the dewatering and consolidation of particulate wastes, such as oil sands fine tailings, using centrifugation to increase settling and consolidation rates. The rate of dewatering of oil sands fine tailings by centrifugation was much faster than by gravity settling. Comparable amounts of water were obtained for both Syncrude and Suncor fine tailings samples in a matter of minutes using centrifugation compared to months for gravity settling. Good linear correlations were found between the results of centrifugation tests and gravity settling data for similar samples. However, each type of sample needs to be calibrated in terms of its settling conditions using both test procedures (gravity vs. centrifugation).

Vacuum filtration was also explored as a dewatering test for oil sands fine tailings. The results from these tests correlated poorly with the gravity settling data. However, these results may be useful in the evaluation of dewatering by filtration techniques, such as filter presses.

INTRODUCTION

The extraction of bitumen from the Athabasca oil sands, using the Hot Water Extraction Process, generates large volumes of tailings, composed mostly of sand, silt, clay, water and some residual bitumen. Presently tailings are pipelined to a settling basin where most of the coarse fraction (>22μm solids), readily settle out to form sand dykes and beaches. The finer fraction (<22μm solids), not captured by the dykes and beaches, flows as a thin slurry into the tailings pond. In the pond, the fines fraction slowly thickens to about 30 w/w% solids during 2 to 3 years. However, further consolidation and densification is extremely slow and satisfactory dewatering will require many decades [1-3]. Herein lies one of the major reclamation problems facing the oil sands surface mining industry. Natural consolidation of the fine tailings to a trafficable surface, capable of supporting a productive soil layer, would require hundreds of years. If a significant percentage of the water in the fine tailings can be recovered, the environmental hazard is proportionately reduced, and fines disposal areas may become suitable for reclamation.

For the past several years, we have been investigating the consolidation behaviour of fine tailings after the removal of hydrophobic components by liquid phase agglomeration [4-12]. After separation of hydrophobic components, the cleaned fine tailings were found to show improved consolidation behaviour. In previous studies, we have used gravity settling to study the dewatering and sedimentation behaviour of treated tailings samples; this is a very slow procedure. A more rapid evaluation test could result in considerable savings of time and manpower. In this investigation, we have attempted to develop such a test using centrifugation to increase settling and consolidation rates.

EXPERIMENTAL PROCEDURES

Sample description. Aqueous sludge from the 17m level of the Syncrude tailings pond was supplied in 5L plastic containers [13]. Prior to use the samples were stored in a cooler at 10^{o}C. The composition data, presented in this report, was supplied courtesy of Syncrude Canada Ltd. Before further sub-sampling, each container was shaken vigorously by hand to ensure thorough mixing. A 200L sample of aqueous tailings from Suncor pond 2, provided courtesy of the Fine Tailings Fundamentals Consortium, was also obtained in a plastic drum. After thorough mixing with a long iron rod 5L subsamples were taken and stored in plastic jugs. Before further sub-sampling, each jug was shaken vigorously by hand to ensure thorough mixing. Analysis of the Suncor sample was carried out using standard methods [14]. The physicochemical properties of both fine tailings samples are presented in Table 1.

Treatment Procedures. Table 2 lists a summary of various procedures, used for the treatment of fine tailings. Details of these methods can be found in several previous publications [5-12].

Dewatering and Sedimentation Test Procedures

a). **Gravity Settling Tests.** Treated aqueous tailings, or the colloidal suspension fraction separated from whole tailings, were transferred to graduated glass cylinders (250 mL). The cylinders were sealed with parafilm in order to prevent evaporation losses during the prolonged settling tests. Gravity settling was followed for periods of up to 90 days. The treated samples were observed to separate into layers; the height of each interface (in mL) was recorded along with the elapsed time. At the end of each test the layers were carefully separated, weighed and then dried at 100 $\pm$ 10^{o}C in order to determine their solid contents.

b). **Sedimentation Evaluation by Centrifugation.** A method for rapid evaluation of sedimentation and dewatering was developed using a Sorvall RT6000B centrifuge. This method involved centrifuging samples in a 100 mL preweighed glass jar for 15 minutes at 1500 x G. At the end of the centrifugation period, the volume of clear water and sediment layers was noted and then the clear water decanted off. The suspension layer, if any, was then carefully separated. Both sediment and suspension fractions were dried at 100 $\pm$ 10^{o}C to determine their solids content.

Filtration Tests. A sample of fine tailings (100g) was accurately weighed into a tared glass filtering funnel, fitted with a coarse porosity frit. The suspension was filtered for 30 minutes using a Nalgene Polypropylene Aspirator Filter Vacuum Pump (Fisher Scientific Catalogue number 09-690-2), at a vacuum of up to 96 kPa (28.5"Hg). The clear water was collected in a tared filtering flask. At the end of the 30 minute period, the filtering flask was weighed and the amount of clear water collected determined by difference. The filter funnel was also weighed to determine the weight of wet cake. Sample weight change was always more than the weight of water collected, suggesting some evaporation losses. The wet cake was dried at 110^{o}C to determine its solids content.

Calculations:

Wt. of filter funnel, (FF)	= F1
Wt. of FF plus fine tailings	= F2
Wt. of FF plus wet cake (residue after 30 minutes filtration)	= F3
Wt. of FF plus dry cake	= F4
Wt. of filtering flask	= W1
Wt of filtering flask plus water collected	= W2
Wt of water collected	= W2-W1 = W3g
Total amount of water in the sample	= 100 - (F4-F1) = W4
Amount of water collected as % of total	= (W3 ÷ W4) x 100
W/W % solids in cake	= [(F4-F1) ÷ (F3-F1)] x 100

RESULTS AND DISCUSSION

Gravity settling to study the dewatering and sedimentation behaviour of treated tailings samples is a very slow procedure. After removal of residual bitumen and oil wettable solids, most of the coarse solids settle within 24 hours, but consolidation takes at least 10 days, [8-12]. The settling of finer solids takes much longer and the differentiation between clear water and the colloidal suspension layers takes about 90 days. The gravity settling of mature, primary tailings, for both Syncrude and Suncor samples is even slower as evidenced from the data plotted in Figure 1. A more rapid evaluation test is desirable. An attempt has been made to develop such a test using centrifugation to increase settling and consolidation rates.

Evaluation of Centrifugation Test Results

Primary Tailings. The results of these tests for primary tailings samples from both Syncrude and Suncor have been plotted in Figures 2-4. The data in Figure 2 demonstrates the effect of centrifugation time on the dewatering of Syncrude primary tailings for three G force values. Linear first order correlations, for both Syncrude and Suncor samples was only obtained for the data at 1500 x G.

Figure 3 is a plot of the amount of water released by centrifugation at 1500 x G as a function of time. A good first order correlation was obtained between the amount of water released and the time of centrifugation at 1500 x G; for Syncrude and Suncor samples the correlation coefficients were 0.98 and 0.99 respectively. Comparable rates of dewatering were obtained in a matter of minutes using centrifugation compared to months for gravity settling. Similar solids consolidation was achieved by 150 minutes of centrifugation at 1500 x G compared to 400 days of gravity settling for Suncor samples and 1200 days for Syncrude samples.

The data plotted in Figures 4 and 5 represent the time required for a given amount of water release under gravity settling compared to the time required to release the same amount of water by centrifugation at 1500 x G. A single correlation cannot be used to represent the data over the entire range. It is obvious from Figure 4 that a linear correlation may exist for the data corresponding to about 75 minutes of centrifugation at 1500 x G which is equivalent to about 200 days of gravity settling. A second linear correlation with different slopes may also exist for the data beyond 200 days of gravity settling.

Treated Samples of Syncrude Fine Tailings. The data in Table 3 compares the volume of clear water obtained from a number of Syncrude fine tailings samples using both gravity settling and centrifugation tests. These samples were obtained from several treatments. Significant differences in the volume of clear water released in the two tests were noted. The results were different for each treatment. The data suggests that in order for the centrifugation test to be valid, each type of sample needs to be calibrated in terms of its settling conditions. This point will become more obvious if we consider the results from tests 1, 3 and 7. For equivalent amounts of water released, centrifugation required only 15 minutes at 1500 x G but corresponding gravity settling times for the three samples were, 215, 90 and 50 days respectively. In all other cases there was a considerable variation in the amount of water released from the sedimentation evaluation tests. For treatments 3 and 6, considerably more water was released by centrifugation than by gravity settling for up to 200 days. However, the reverse was noted for treatments 2, and 7.

Table 4 lists the data for solids content of compact sediments and colloidal suspension fractions obtained using the two test procedures. Compared to the differences in the amounts of water released by the two test procedures, the solids content of the sediments obtained by the two methods were very similar for most of the samples. This suggests that the centrifugation test, gives results similar to those from gravity settling tests for sedimentation/consolidation studies, but may not give comparable results for clear water release.

In our previous work [9], we had demonstrated that sediment compaction (as measured by the percent solids in the sediment) data alone is not a good indicator of the overall consolidation behaviour of fine tailings, because it does not take into account the amount of solids remaining in suspension. Therefore, a Separation Index (SI), designed to take into account the total amount and degree of compaction of solids separated into the sediment layer, was developed. This parameter allowed better discrimination between the results of settling tests when compared to the compaction data alone.

Filtration Tests

Filtration was also used for comparison of dewatering rates. Tables 5 and 6 list the results of filtration tests, for both Syncrude and Suncor samples, after various treatments. The results from gravity settling and centrifugation are also listed for comparison purposes. Although these results are not comparable to the results obtained from the other test procedures, they confirm the trends observed from the centrifugation data. Thus, these results together with centrifugation data can be used to compare the relative dewaterability of the two types of tailings samples. This is demonstrated in Figure 6, which is a plot of the amount of water released from both types of samples after various treatments. It is apparent from these plots that Suncor samples released considerably more clear water under similar conditions than did the Syncrude samples. This is further supported by the significantly higher solids content of the dewatered cake obtained from Suncor samples (52-66%) compared to Syncrude samples (39-61%).

CONCLUSIONS

1. A fast test to evaluate the dewatering and consolidation of Athabasca fine tailings was developed using centrifugation to increase settling and consolidation rates. In order for the test to be valid, each type of sample needs to be calibrated by comparison with the equivalent gravity settling data.

2. The results of gravity settling of untreated fine tailings samples (blanks) suggest that the rate of release of water from Suncor tailings is much faster than that for the Syncrude fine tailings. This is further supported by data obtained on treated samples using centrifugation and filtration tests. The major cause for this difference in settling behaviour for the two types of tailings, could be a result of the differences in the physical and chemical characteristics of certain components of fine tailings such as ultra-fines, humic matter and oil wettable solids.

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Table 1. Physicochemical properties of Fine Tailings Samples

Property	Value	
	Syncrude	Suncor
pH at 25 ± 0.5°C	7.6	8.1
Conductivity (mS/cm)	1.8	1.7
Total Solids, wt%	35.1	29.1
Bitumen, wt%	1.2 ± 0.1	5.7 ± 0.3
Density, g/mL	1.22	1.20
Solids, <44μm (%)	99 ± 1	84 ± 2

Table 2. Summary of various treatment procedures

ID	Collector	Conditioner	Description
Blanks	None	None	-
LLT	Heavy Oil	Na_2SiO_3	Ref. 9
Agg.1	Coke	Na_2SiO_3	Ref. 7, 8, 10
Agg.2	Coke	Lime	" " " "
Agg.3	Fly ash	None	" " " "
Agg.4	Hydrophobic Solids except carbonaceous materials	None	Mild agitation on a roller mill
SS-1	Fine tailings Solids	None	Agitated in a Waring blender, 20 minutes, 10,000 rpm
SS-2	Fine tailings Solids	Na_2SiO_3	As above
SS-3	Silica Sand	None	As above

LLT = Liquid-Liquid transfer; Agg. = Agglomeration; SS = Sand Surcharging

Table 3. Comparison of Gravity and Centrifugation Test Procedures. The Volume of Clear Water released (Syncrude samples).

TEST #	Sample/Treatment	Clear Water (% of total Volume)	
		Gravity	Centrifugation
1	Blank (Whole fine tailings)	15 (215 days)	15 ± 5
2	Blank (Colloidal suspension)	6 ± 1 (90 days)	10 ± 1
3	Treatment-1	10 ± 5 (90 days)	10 ± 2
4	Treatment-2	28 (200 days)	0
5	Treatment-3	23 (200 days)	34
6	Treatment-4	7 ± 1 (90 days)	10 ± 2
7	Treatment-5	10 (50 days)	10
8	Treatment-6	21 (200 days)	41
9	Treatment-7	21 (200 days)	0

Treatment-1, Liquid-liquid transfer with heavy oil as collector and Na_2SiO_3 as conditioner.
Treatment-2, Agitation with 0.75 Kg/m^3 of lime.
Treatment-3, Treated with kg/m^3 of H_2SO_4
Treatment-4, Agglomeration with coke as collector and Na_2SiO_3 as conditioner.
Treatment-5, Agglomeration with coke as collector and lime as conditioner.
Treatment-6, As above followed by treatment with kg/m^3 of H_2SO_4.
Treatment-7, Sand surcharging.

Table 4. Comparison of Gravity and Centrifugation Test Procedures. Solids content of separated fractions (Syncrude samples)*

Sample	Solids Content (w/w%)			
	Gravity		Centrifugation	
	Suspension	Sediment	Suspension	Sediment
Blank (Whole fine tailings)	29 ± 1	-	30 ± 1	-
Blank (Colloidal Suspension)	7.9 ± 1	-	8.2 ± 1	-
Treatment-1	7.4 ± 1	58 ± 3	7.5 ± 1	60 ± 2
Treatment-2	-	43	-	31
Treatment-3	-	41	-	42
Treatment-4	6.4 ± 1	63 ± 1	3.7	63
Treatment-5	-	45	3.3	54 (45)**
Treatment-6	-	44	-	51
Treatment-7	-	42	6.7	63

* All conditions as listed in Table 3 above.

** Average of combined sediments.

Table 5. Comparison of Dewatering Data obtained using various Techniques (Syncrude Samples)

Test #	Treatment	Amount of clear water released (% of total)		
		Gravity	Centrifugation	Filtration
1	Blank	8 ± 2 (29)	4 (30)	4 (37)
2	H_2SO_4 (1 kg/m^3)	23 (34)	34 (42)	10 (39)
3	Acid plus lime	ND	31 (45)	7 (39)
4	Lime plus acid	ND	8 (39)	29 (45)
5	Agitation with 5w/w% fly ash	ND	39 (57)	2 (48)
6	Agglomeration-1	7 ± 1	0 (61)	0 (36)
7	Above plus acid	ND	0 (59)	0 (36)
8	Agglomeration-2 plus acid	21 (44)	42 (53)	21 (47)
9	Agglomeration-3	ND	4 (52)	8 (36)
10	Above plus acid	ND	33 (43)	11 (37)

* Values in parenthesis represent w/w% solids in the cake. Fly ash sample used in this study was from Suncor. ND, not determined.
Agglomeration-1, 2 and 3 have been defined in Table 2.

Table 6. Comparison of Dewatering Data obtained using various Techniques (Suncor Samples)

Test #	Treatment	Amount of clear water released (% of total) Centrifugation	Filtration
1	Blank	6 (59)	11(39)
2	H_2SO_4 (1 kg/m^3)	60 (54)	26 (41)
3	Acid plus lime	61 (56)	43 (51)
4	Lime plus acid	64 (57)	27 (42)
5	Agitation with 5w/w% fly ash	58 (56)	32 (46)
6	Acid plus 1% fly ash	62 (57)	32 (45)
7	Acid plus 5w/w% fly ash	62 (59)	52 (56)
8	5w/w% fly ash plus acid	64 (61)	48 (55)
9	Acid plus 5w/w% Syn. coke	61 (58)	18 (42)
10	Agglomeration-1	0 (66)	0 (35)
11	Above plus acid	11 (56)	5 (35)
12	Agglomeration-2	0 (64)	0 (35)
13	above plus acid	60 (58)	20 (44)
14	Agglomeration-3	15 (54)	11 (32)
15	Above plus acid	65 (52)	24 (35)

* Values in parenthesis represent w/w% solids in the cake. Fly ash sample used in this study was from Suncor.

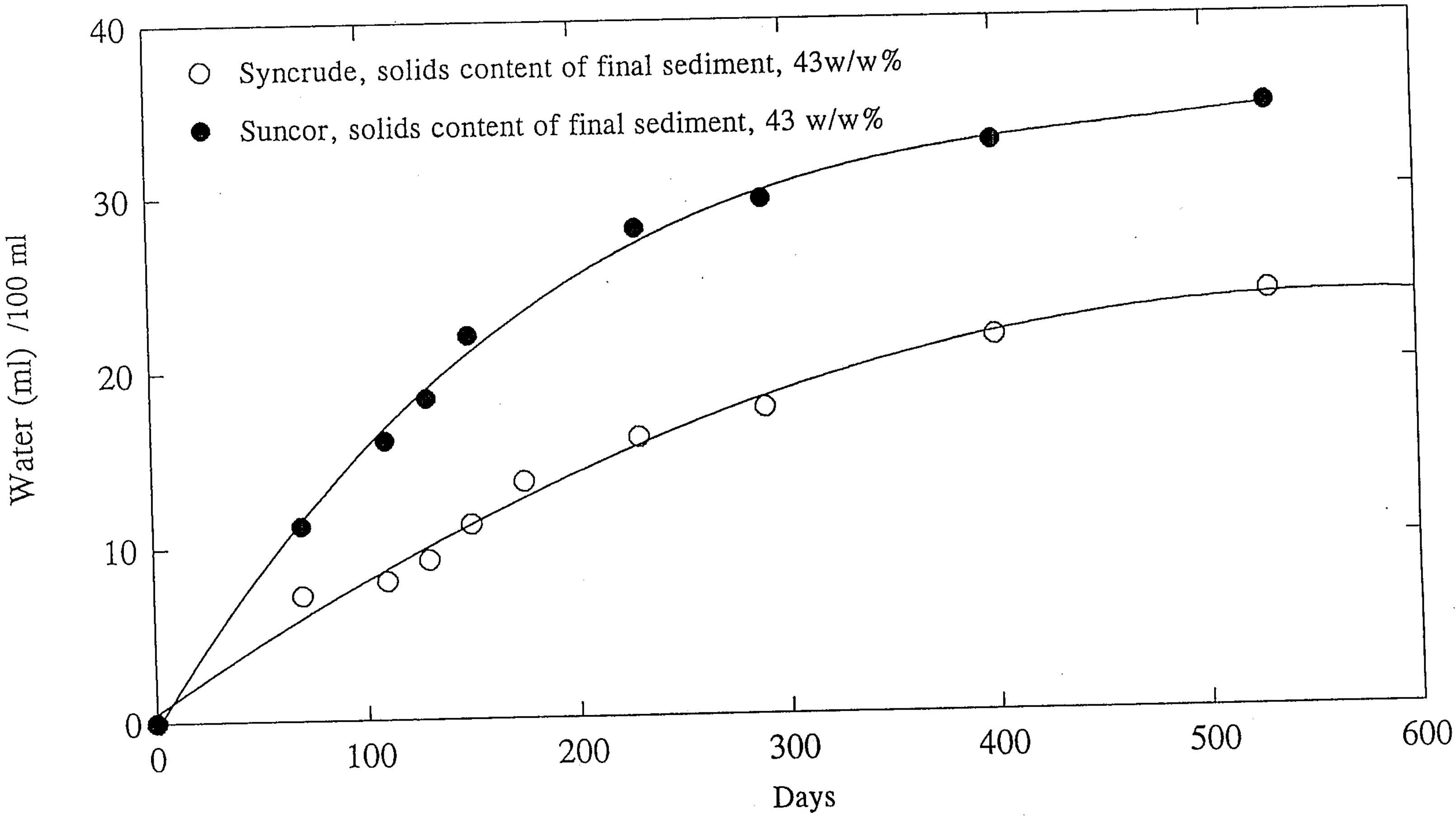

Figure 1. Gravity Settling Curves for Primary Tailings

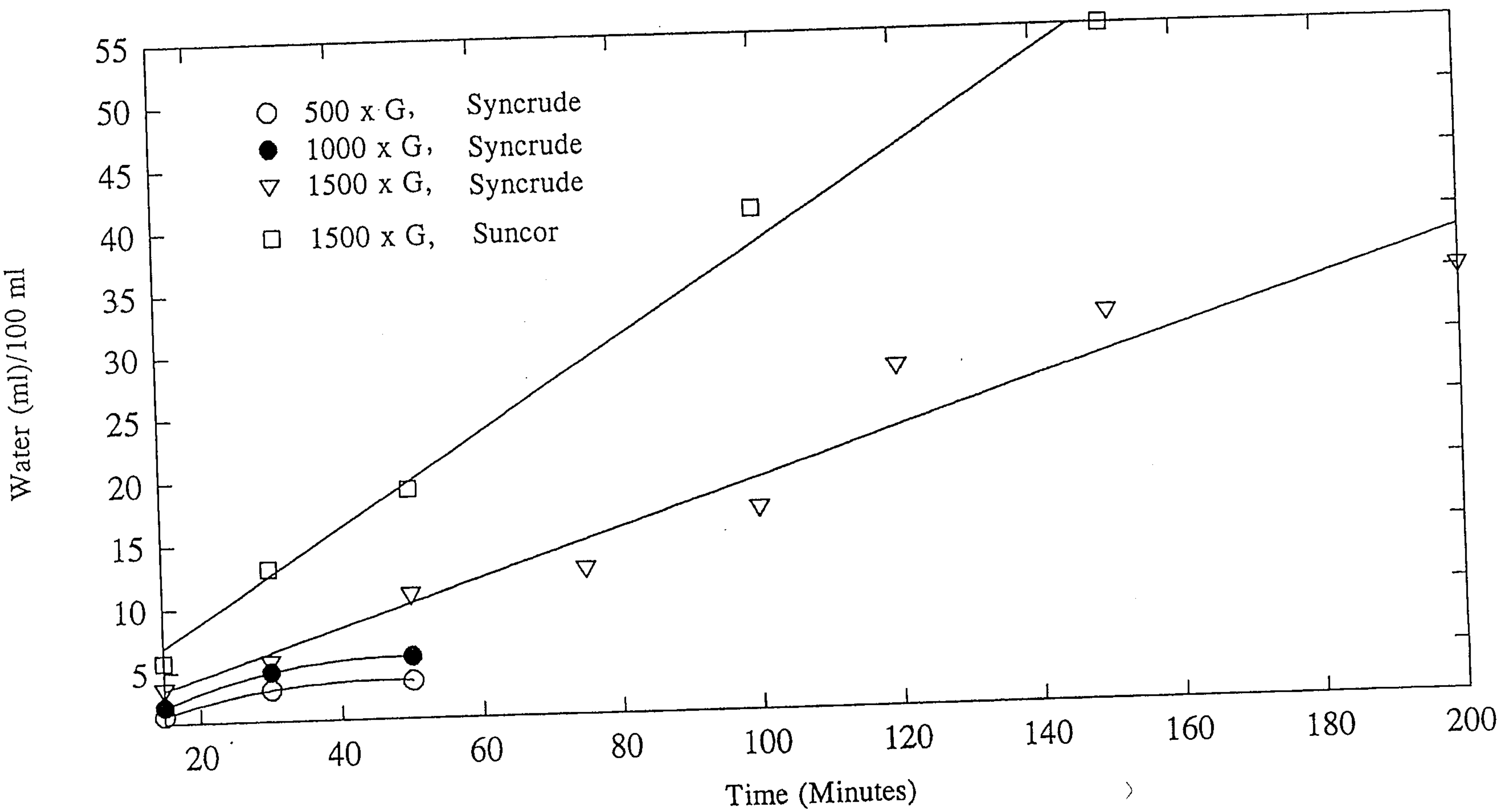

Figure 2. The Effect of Centrifugation Time on the Dewatering of Primary Tailings at Various G forces

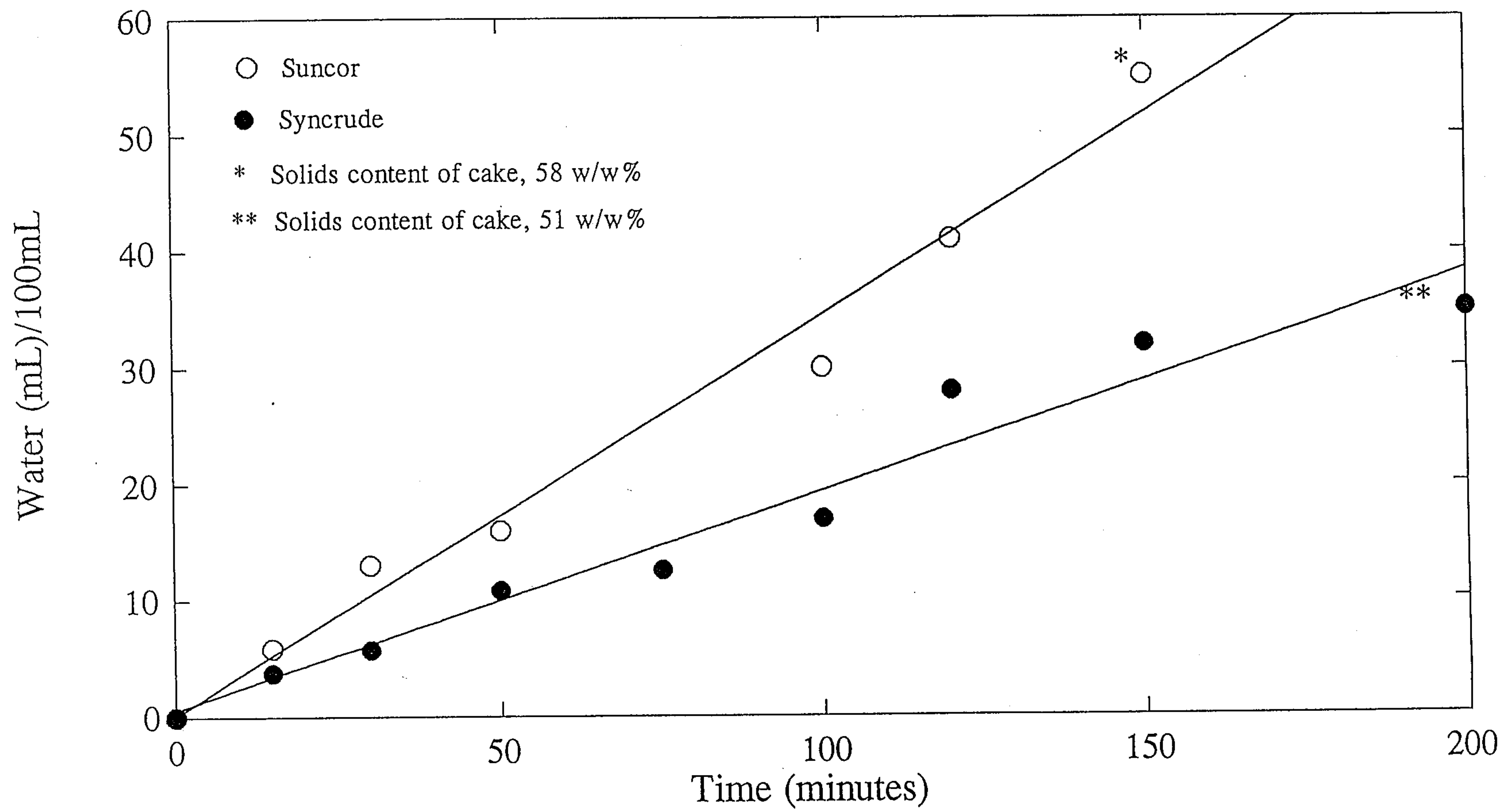

Figure 3. Dewatering of Fine Tailings by Centrifugation

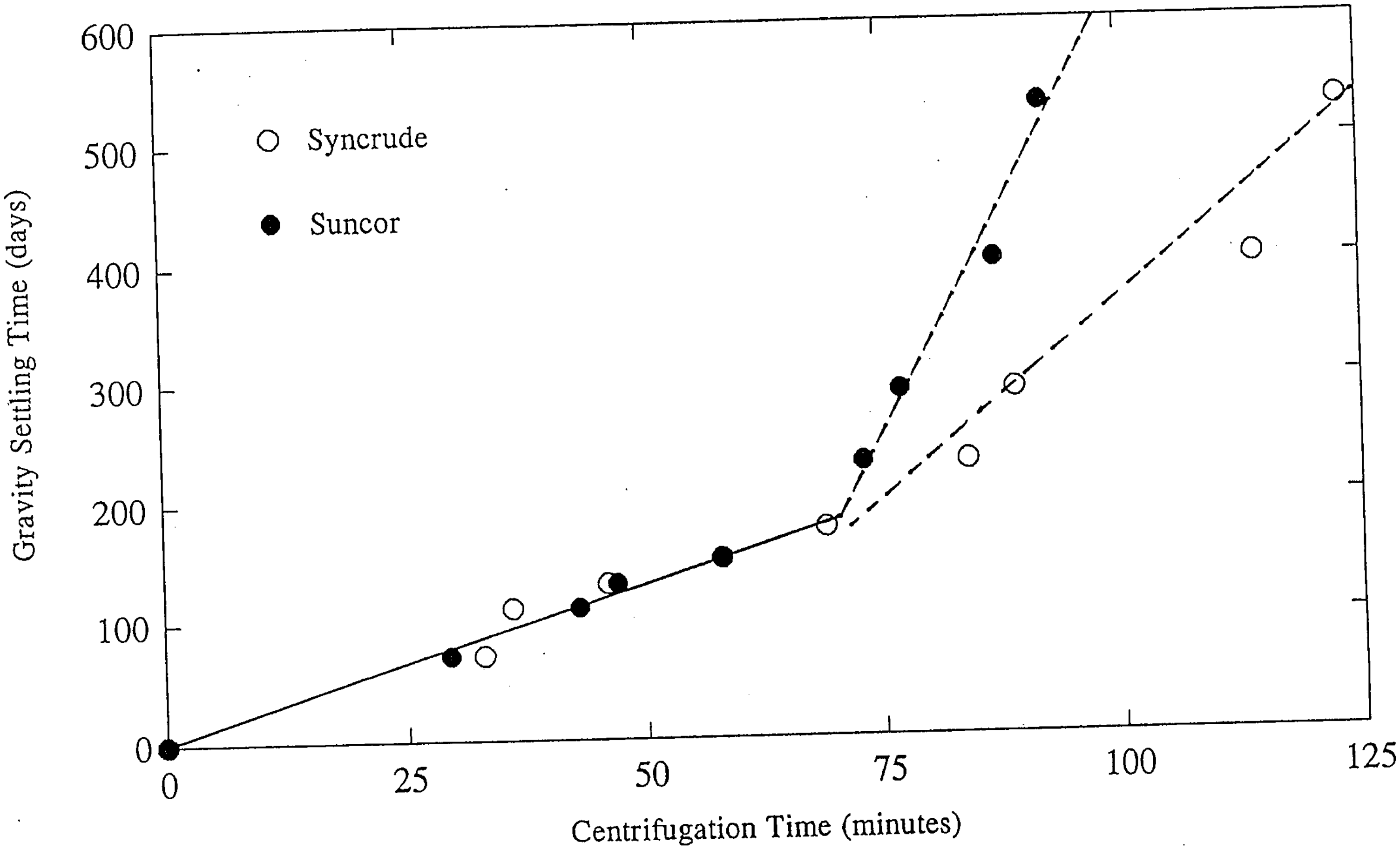

Figure 4. Gravity Settling vs Centrifugation at 1500 x G

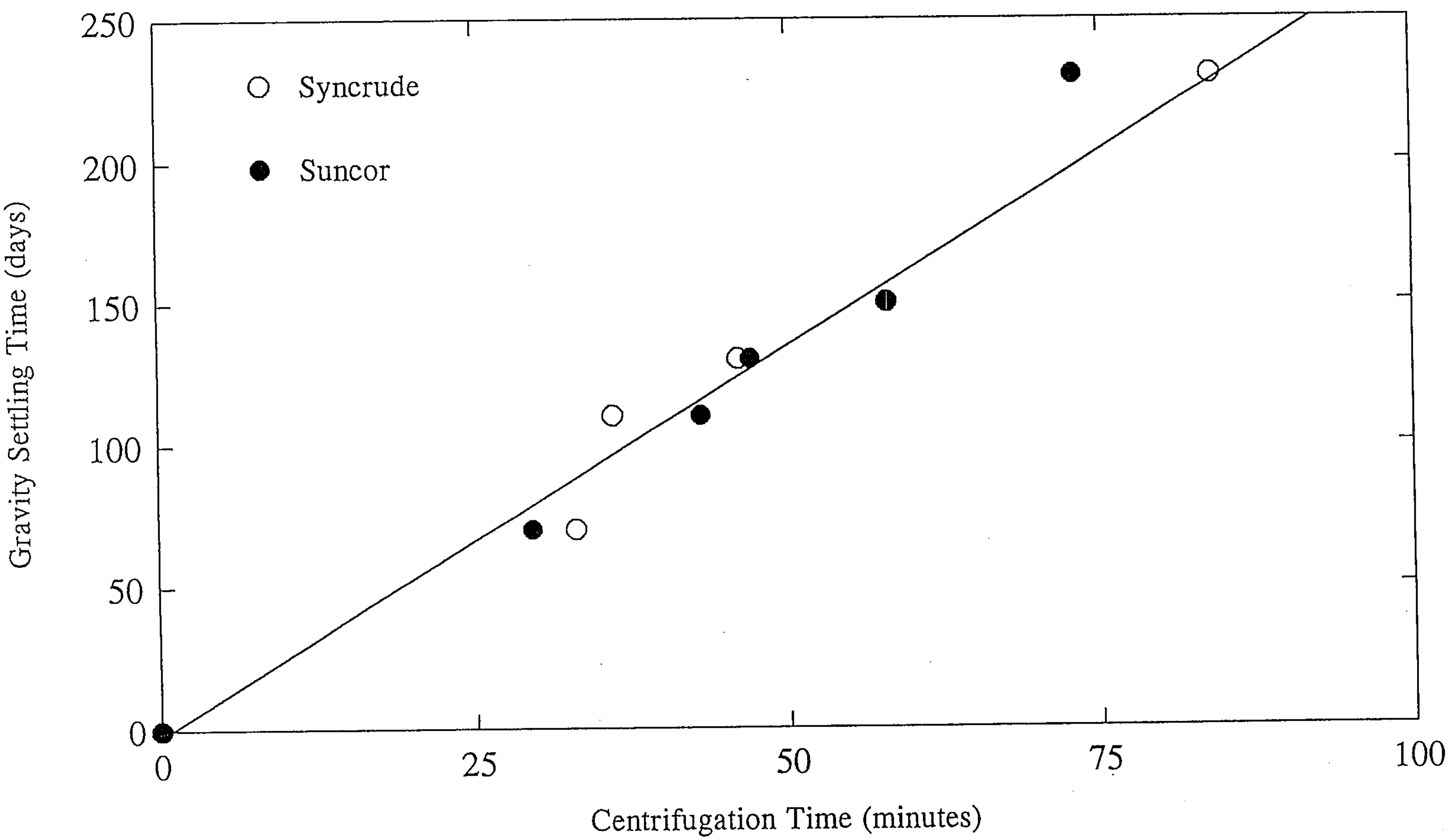

Figure 5. Gravity Settling vs Centrifugation at 1500 x G

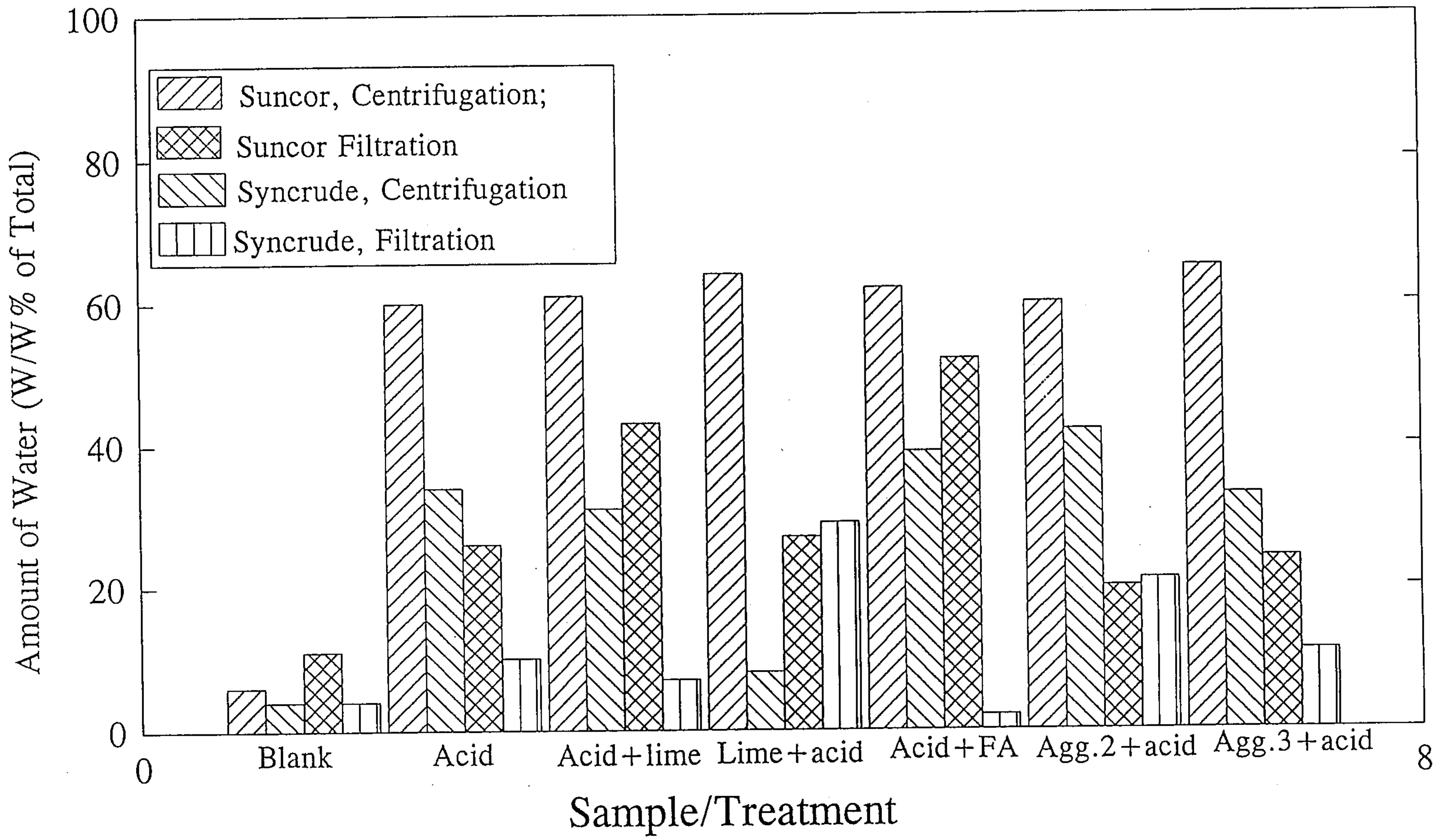

Figure 6. Comparison of Dewaterability of Syncrudevs Suncor Tailings