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Dewatering of Athabasca Oil Sands Fine Tails using Agglomeration Techniques*

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ABSTRACT

Liquid phase agglomeration techniques developed at the National Research Council of Canada have been applied for the recovery of organic material from the fine tails produced by surface mining, bitumen extraction plants. The fractions separated by these techniques include residual bitumen and naphtha plus solids associated with strongly adsorbed largely insoluble organic matter. After separation of these hydrophobic components, the cleaned sludge has been found to show altered settling behaviour.

The present study is a continuation of our previous investigation on the settling behaviour of cleaned sludge after the removal of organic components using oil phase agglomeration techniques. In general the sludge separated into three layers: relatively clean water on top followed consecutively by a suspension of colloidal solids and a clean compacted solids layer. Dewatering of the suspension fraction, containing colloidal solids, was also investigated by using conventional coagulation/flocculation techniques. The results of this investigation suggest that up to 60 w/w% of the total water in the sludge can be recovered for recycling. Overall water recovery depends upon the selected levels for the variables in the oil phase agglomeration technique.

INTRODUCTION

All bitumen separation processes, both in situ and surface mine, currently in operation in Alberta have the disadvantage of producing large volumes of aqueous tailings, partly in the form of stable sludge suspensions [1-3]. Considerable quantities of dispersed bitumen and solids associated with organic matter, insoluble in common solvents (IOM), are found with the fines contained in these tailings [4-5]. This organic matter is believed to be partly responsible for the stability of the sludge.

For the past several years, we have been developing liquid phase agglomeration techniques for the recovery of valuable hydrocarbon components from the fine tails produced by surface mining, bitumen extraction plants [6-10]. The fractions separated by these techniques include residual bitumen and naphtha plus a heavy metal minerals fraction associated with strongly adsorbed largely insoluble organic matter [11-13]. After separation of these hydrophobic components, the cleaned sludge was found to show altered settling behaviour [6, 7, 9, 14-18].

In our previous work, we reported settling data for clean sludge after selectively removing hydrophobic components by a liquid-liquid transfer technique, using a mixer with contra-rotating blades and with vacuum still bottoms as the collector phase [9, 15-18]. The present study is a continuation of our previous work on the settling behaviour of clean sludge after the removal of organic components using oil phase agglomeration techniques. In this investigation, we have used refinery coke as the collector phase to remove hydrocarbon components from fine tails mixed in a Waring Blendor. The settling behaviour of clean sludge after various levels of treatment was studied. In addition, dewatering of the suspension fraction containing colloidal solids was also investigated by using conventional coagulation/flocculation techniques.

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EXPERIMENTAL PROCEDURES

Sample description. Aqueous sludge from the 17m level of the Syncrude tailings pond was pumped into 200 L plastic drums [19]. Each drum was inverted five times (with a barrel tipper) before being divided among a number of 5 L plastic jugs, which were then stored in a cooler at ≈ 10°C. The results presented in this report were obtained on these sub-samples, provided courtesy of Syncrude Canada Ltd. Before further sub-sampling, each jug was shaken vigorously by hand to ensure thorough mixing. The physicochemical properties of sludge, provided by Syncrude are presented in Table 1 [20].

Recovery of Bitumen

About 500 g of homogenized sludge was placed in a Waring Blendor jar and the desired amount of sodium silicate or lime added as a conditioning agent. The contents were agitated for 1 minute at 120 rps. Varying amounts of Syncrude refinery coke (100-200μm diameter) were then added and blending continued for another 15 minutes. The contents were allowed to stand until a clear demarcation in the top froth layer was observed (about 30 minutes). The froth was carefully skimmed off so as not to disturb the tails slurry underneath. The clean tails were used for sedimentation/dewatering studies.

Sedimentation/Dewatering

a). <u>Gravity Sedimentation Measurements.</u> Treated aqueous tails, or the colloidal suspension fraction separated from whole tails, were transferred to 500 ml graduated glass cylinders. The cylinders were sealed with parafilm in order to prevent losses due to evaporation during the prolonged settling tests. Gravity settling was followed for periods up to 90 days. The treated samples were observed to separate into layers; the height of the 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 ± 10°C in order to determine their solid contents.

b). <u>Centrifugation.</u> A quick sedimentation/dewatering test was developed using a Sorvall RT6000B centrifuge. This method involved centrifuging treated 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 was noted and then decanted off. The suspension layer (if any) was then carefully separated. Both sediment and suspension fractions were dried at 100 ± 10°C in order to determine their solids content.

RESULTS AND DISCUSSION

Gravity settling to study the sedimentation/dewatering behaviour of treated tails samples is a very slow procedure. Although, most of the coarse solids settle within 24 hours, consolidation takes about 10 days. The settling of finer solids takes much longer and the differentiation between clear water and the colloidal suspension layers takes about 90 days. A quicker test would be more desirable; attempts were made to develop such a test using centrifugation to increase settling and consolidation rates. The data in Table 3 compares the volume of clear water obtained from a number of samples using the two test procedures. Significant differences in the volume of clear water released in the two tests were noted. Consequently, caution should be exercised when comparing this type of data from the two test procedures.

Table 4 lists the data for solids content of compact sediments and colloidal suspension fractions obtained using the two test procedures. The solids content of the sediments obtained by the two methods were similar. However, there was still some variation in the results for the solids content of the colloidal suspension fractions. This is consistent with the differences found in data for the volume of clear water. This suggests that the centrifugation test, developed in this investigation, 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 sludge 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.

Treatment-1, Gravity Settling Results

Gravity settling of sludge samples after conditioning with sodium silicate and treatment with coke, was carried out in graduated glass cylinders for 90 days. The sludge separated into four layers, namely: coke-oil froth on top, followed consecutively by relatively clean water, a suspension containing 7 ± 1% (w/w) of colloidal solids, and a well compacted layer with a solids content of over 60 % (w/w), (sediment) which also contained some of the coke/oil fraction.

Figure 1 illustrates the results of gravity settling tests for both Syncrude and Suncor Sludge samples, including blanks. The data include: volume of clear water released (% of total water), % solids in the sediment and a Separation Index [18], which is a measure of the overall consolidation behaviour of the sludge. The amount of clear water released was insignificant because of the dispersion of aggregated clay particles by sodium silicate. However, the consolidation behaviour of the sludge improved considerably after treatment with coke, which removed most of the free bitumen by adsorption. The improvement in the consolidation behaviour was more pronounced for samples conditioned with sodium silicate.

The role of sodium silicate appears to be complex because conditioning with sodium silicate alone does not result in significant consolidation of sludge solids. Also, its role as a pH modifier does not appear to be important as pH modification by other alkalies [9] does not produce similar results. It is known to act as a viscosity modifier by dispersing aggregated clay particles. This would be important in the breakup of sludge structure and could result in the rapid settling of coarser solids while the finer colloidal, clay particles would remain dispersed in sol form rather than as a gel type structure. A third role could be as a surface modifier, rendering the hydrophobic solids hydrophilic either by direct adsorption or by displacement of organic coatings. This latter action would result in bitumen displacement and subsequent removal by coke.

The results of settling tests obtained in the present investigation are plotted in Figure 1. It is obvious that the removal of organic components improved the settling behaviour of sludge as indicated by the higher sediment solids content and SI values for the treated samples, as compared to the blanks.

The test results for sodium silicate conditioned sludge samples suggests a considerable improvement in the settling performance over the unconditioned samples. Increasing sodium silicate concentration further improved the settling behaviour as indicated by the best settling performance obtained with sodium silicate conditioning at pH 10, compared to lower pHs.

The sludge samples from Suncor showed essentially the same consolidation behaviour as those from Syncrude tailings ponds. However, considerably more clear water was released in the blank tests for Suncor sludge compared to those for Syncrude sludge. Contrary to this behaviour, no clear water was released from sodium silicate treated Suncor samples while a small amount of clear water was obtained from Syncrude samples. This suggests that the nature of ultra-fines in the two types of samples could be different either in composition or in surface characteristics.

Dewatering of Colloidal Suspension from treatment-1

Dewatering of the suspension fraction, containing dispersed, fine colloidal clay particles was attempted by using a number of coagulants/flocculants as well as sulphuric acid [21-22]. During the initial stages of this investigation it was found that similar levels of dewatering could be achieved with most of the reagents tested, including sulphuric acid. However, because of its low cost sulphuric acid has an economic advantage over other materials and subsequent studies were limited to this reagent only. The test results for the dewatering of the colloidal suspension fraction from treatment 1 are shown in Figure 2. Over 30 % of the total water from the original sludge (45 % of suspension water) can be recovered by the use of 2 kg/m^3 of sulphuric acid (specific gravity 1.84g/ml). The separated cake had a solids content of 15 % compared to 8 % for the suspension. At this acid loading the pH of the recovered water was very low (2.5), however, the pH slowly increased if the treated sample was allowed to stand for several weeks. In one case the final pH was found to be around 5; this increase in final pH is consistent with Schutte's work [22].

Table 5 lists the summary of combined results from treatment-1. It is obvious that about 40 % of the total water present in sludge can be recovered by removing free and adsorbed bitumen by oil phase agglomeration using coke and subsequent treatment of the free colloidal suspension with sulphuric acid. The sediment from the initial settling stage is very compact and can be mixed with the cake obtained from the treatment of the colloidal suspension. The combined sediments had a solids content of about 48 % and would be suitable for further mechanical treatment and subsequent dry disposal.

Treatment 2

Recently, we have developed a technique whereby sulphur dioxide adsorbents can be incorporated into coal or coke agglomerates by liquid phase agglomeration using bitumen or heavy oil as the binder [23-27]. This technique allows the advantageous use of very small and more active sulphur dioxide adsorbent particles in fluid bed combustion by binding them tightly within larger coal agglomerates, thereby reducing the possibility of their elutriation from the bed. As a result, higher adsorbent utilization efficiencies were obtained for the co-agglomerated fuel, compared to conventional systems in which a coarser adsorbent is added separately to the fluid bed. In this investigation we have studied the effect of sulphur dioxide sorbents on the settling behaviour of coke treated sludge. The sludge was first conditioned with lime before treatment with coke. After treatment, the froth was skimmed off and the remaining slurry was centrifuged at 1500 x G for 15 minutes. The sedimentation results for a number of tests using a Syncrude sludge pond sample are listed in Table 6. No significant amount of clear water was released in these tests. However, the amount of fine colloidal solids in the suspension fraction was much lower than in the cases where sodium silicate was used (3.5 ± 0.3 % vs 7 ± 1 %). With increasing amounts of lime, the amount of solids in both the froth and sediment increased. Lime concentrations greater than 0.5 kg/m^3, gave compact sediments with solids contents higher than 50 %.

Dewatering of treated sludge with sulphuric acid

Froth was skimmed off after treatment of lime conditioned sludge with coke. The remaining slurry was then treated with various amounts of sulphuric acid, and centrifuged at 1500 x G for 15 minutes. The results are listed in Table 7. Although significant amounts of clear water were recovered from whole sludge by the use of sulphuric acid, without any prior treatment (blank), the sediment was not sufficiently compact. The coke treated samples gave almost the same amount of clear water as obtained in the blank tests but the sediment contained more than 50 % solids.

Dewatering of Colloidal Suspension from Treatment-2

The colloidal suspension obtained from treatment-2, using lime as a conditioner, was also dewatered by pH modification with sulphuric acid. The sulphuric acid treated samples were centrifuged at 1500 x G for 15 minutes to assess the degree of dewaterability for the various acid doses. The results are listed in Table 8. It is obvious from these results that about half of the total water (70-80 % of the suspension water) can be recovered from the treatment of this colloidal suspension with sulphuric acid. Also, the pH of the recovered water was near to that of natural, surface water. The solids content of the sediment was about 20 % and could be mixed with the coarse solids obtained from the treatment of sludge with coke to give an overall solids content of 45 %.

Table 9 lists the summary of treatment-2 results. For this treatment the dewatering tests were carried out on the whole sludge as well as on the suspension fraction. An additional 20 % more water was released if treatment was carried out in two stages. However, the sediment obtained from the one stage treatment was more compact than that obtained from the two stage treatment (51 % solids compared to 45 % solids). The pH of the water recovered from the two treatments was similar.

Table 10 contains a summary of key results from various treatments including blanks. The two important parameters are the amount of total clear water released and the average solids content of sediments. A comparison of this data for various treatments leads to a number of conclusions:

1. About 45 % of total sludge water was recovered by agitating whole, untreated sludge with 1 kg/m^3 of concentrated sulphuric acid . However, the sediment obtained from these tests contained only 42 % of solids.

2. A combination of agglomeration treatments followed by dewatering using 0.75-2 kg/m^3 of concentrated sulphuric acid, released 10-15 % additional water. This treatment also resulted in the settling of over 80 w/w % of total solids into a compact sediment containing up to 63 w/w % of solids. The combined sediments from the two treatments had a solids content of 45- 48 % (w/w).

3. The pH of the recovered water ranged from 3-6.8.

CONCLUSIONS

Conditioning of fine tails with sodium silicate or lime, followed by oil phase agglomeration treatment, using refinery coke as a collector for hydrophobic components, resulted in sludge destabilization. Over 80 w/w % of the solids settled rapidly to a compact layer containing up to 63 w/w % solids. After oil phase agglomeration treatment the remaining suspended solids could be dewatered using sulphuric acid.

About 40 % of clear water could be recovered by treating the whole sludge with sulphuric acid without any prior treatment. The sediment from this treatment had a solids content of 42 w/w %. The coke treated whole sludge gave almost the same amount of clear water as obtained in the blank tests, but the sediment from these tests had a solids content of about 50 w/w %. The amount of clear water recovered could be increased to 50-60 % by carrying out the sedimentation and dewatering steps separately. Oil phase agglomeration treatment resulted in sedimentation of over 80 % of the solids and produced a dilute suspension of colloidal solids that could be further dewatered with sulphuric acid to give an additional water recovery of 40-50 %. The cake obtained from the dewatering of this colloidal suspension had a solids content of only 20%. Blending of this material with the coarser sediment produced a material suitable for further mechanical dewatering. The recovered water was free of suspended solids and had a pH of about 6.6 to 6.8.

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Table 1.Physicochemical properties of Syncrude Sludge

Property	Value
pH at 17.8±0.5°C	**7.83±0.03**
Conductivity (ms/cm)	**1.6±0.01**
Total Solids [Gravity], wt. %	**26.6±0.1**
Bitumen [OWS], wt. %	**0.9±0.05 (1.1±0.2)**
Solids [OWS], wt. %	**25.6±0.1 (26.5±0.9)**
Density (calculated), g/ml	**1.19**
Solids, < 44μ (%)	**99.2±1.0**
Solids, < 22μ (%)	**94.9±1.9**
Solids, < 11μ (%)	**83.7±2.1**

Values in parenthesis were determined at NRC.

Table 2. Summary of various tests

Sample	Treatment ID	Collector	Conditioner	Coagulant/ flocculant
Whole Sludge	Blank-1	-	-	-
Whole Sludge	Blank-2	-	Na_2SiO_3	-
Whole Sludge	Blank-3	-	-	H_2SO_4
Whole Sludge	Agg-1	Coke	Na_2SiO_3	-
Colloidal Suspension	Agg-1	Coke	Na_2SiO_3	H_2SO_4
Colloidal Suspension	Blank-4	Coke	Na_2SiO_3	-
Whole Sludge	Agg-2	Coke	Lime	-
Whole Sludge	Agg-2	Coke	Lime	H_2SO_4
Colloidal Suspension	Agg-2	Coke	Lime	H_2SO_4

Table 3. Comparison of Gravity and Centrifugation Test Procedures. The Volume of Water released from Syncrude Sludge samples.

Sample	Clear Water (% of total Volume)	
	Gravity (90 days)	Centrifugation
Blank (Whole Sludge)	8 ± 2	15 ± 5
Blank (Colloidal Solids)	6 ± 1	10 ± 1
Treated Sludge-1*	10 ± 5	10 ± 2
Treated Sludge-2**	7 ± 1	10 ± 2

* Liquid-liquid transfer with heavy oil as collector and Na_2SiO_3 as conditioner.

** Agglomeration with coke as collector and Na_2SiO_3 as conditioner.

Table 4. Comparison of Gravity and Centrifugation Test Procedures. Solids content of separated fractions

Sample	Solids Content (w/w %)			
	Gravity (90 days)		Centrifugation	
	Suspension	Sediment	Suspension	Sediment
Blank (Whole Sludge)	29 ± 1	-	30 ± 1	-
Blank (Colloidal Solids)	6 ± 1	-	10 ± 1	-
Treated Sludge-1*	7.4 ± 1	58 ± 3	7.5 ± 1	60 ± 2
Treated Sludge-2**	6.4 ± 1	63 ± 1	3.7	63

* LLT, using heavy oil as collector, and Na_2SiO_3 as a conditioner.

** Agg, Using coke as collector and Na_2SiO_3 as a conditioner.

Table 5. Combined results of sludge treatment by Agglomeration-1 followed by Dewatering of colloidal suspension

Clear water released from gravity settling of whole sludge	6 %
Water released from dewatering of colloidal suspension, (centrifugation)	34 %
Total clear water released	40 % of total
Amount of H_2SO_4 required for dewatering	2kg/m^3 of suspension
Final pH of the recovered water	5
Solids content of the sediment from gravity settling of whole sludge	63 %
Solids content of the cake from suspension	15 %
Average solids content of the combined sediments	48 %
Total solids recovered in combined sediments	100 %

Table 6. Sedimentation data for treatment-Agg-2 (Whole Sludge)

Test #	Lime (kg/m^3)	Clear water (% of total)	pH	Froth	Suspension	Sediment
				Solids Content (W/W %)		
Blank	0.75	0	10.5	31	8.2	60
1	0.5	0	9.7	33	3.2	45
2	0.75	10	10.3	52	3.3	55
3	1.0	0	10.7	56	3.9	57

Table 7. Dewatering of Syncrude sludge from treatment-Agg-2 (whole sludge)

Test #	Lime (kg/m^3)	H_2SO_4 (kg/m^3)	Clear water (% of total)	pH	% solids in Sediment
Blank 1	-	0.5	0	6.5	28
Blank 2	-	1.0	45	6.0	42
1	1	0.5	17	6.7	43
2	1	1.0	41	6.6	51

Table 8. Dewatering of colloidal suspension from treatment-2 (lime conditioning)

Test #	Lime (kg/m^3)	H_2SO_4 (kg/m^3)	Clear water (% of total)	pH	Solids in sediment (W/W %)
Blank*	0.75	0.5	18	6.6	10.9
1	0.75	0.5	50	6.8	21
2	0.5	0.5	40	5.6	21
3	1.0	1.0	60	4.1	20

* No coke

Table 9. Combined Results from Agg-2 Treatment.

Single Stage treatment (treatment of whole sludge from Agg-2 with sulphuric acid)

Clear water released	40 %
Amount of sulphuric acid used	$1kg/m^3$ of sludge
Final pH of the recovered water	6.6
Solids content of the sediment	51 %

Two stage treatment (Agglomeration with coke followed by dewatering of colloidal suspension)

Clear water released from settling of whole sludge	10 %
Water released from the treatment of colloidal suspension	50 %
Total clear water released for two stage treatment	60 % of total
Amount of H_2SO_4 required for dewatering	$0.75kg/m^3$ of suspension
Final pH of the recovered water	6.8
Solids content of the sediment from sludge settling	55 %
Solids content of the cake from suspension	21 %
Average solids content of the combined sediments	45 %
Total solids recovered in compact sediments	100 %

Table 10. Summary of Various Treatments

	Blank-1	Blank-2	Blank-3	Agg-1	Agg-2a	Agg-2b	LLT
Total Clear water released	8 ± 2	7	45	40	40	60	50
Final pH of recovered water	7.8	10	6	5	6.6	6.8	3
Average solids content, w/w %*							
Sediment-1	29 ± 1 (100)	27 (100)	42 (100)	63 (90)	51 (100)	55 (83)	60±2 (83±7)
Sediment-2	-	-	-	15 (10)	-	21 (17)	20 (17±7)

Blank-1, Untreated without agitation.

Blank-2, Agitated and conditioned with sodium silicate.

Blank-3, Agitated with 1kg/m^3 of conc. sulphuric acid.

Agg.-1, Sodium silicate conditioned sludge agitated with coke, froth skimmed off and residual slurry allowed to settle, colloidal suspension decanted off and treated with 2 kg/m^3 of concentrated sulphuric acid.

Agg.-2a, Lime conditioned sludge agitated with coke, froth skimmed off and the residual slurry treated with 1 kg/m^3 of conc. sulphuric acid.

Agg.-2b, Lime conditioned sludge agitated with coke, froth skimmed off, residual slurry centrifuged at 1500 x G for 15 minutes, suspension decanted off and treated with 0.75 kg/m^3 of conc. sulphuric acid.

LLT, Residual bitumen and oil wettable solids removed from sodium silicate conditioned sludge, and allowed to gravity settle for 90 days. The colloidal suspension treated with 1.5 kg/m^3 of sulphuric acid.

* Values in parenthesis represent percentage of total solids contained in the sediments.

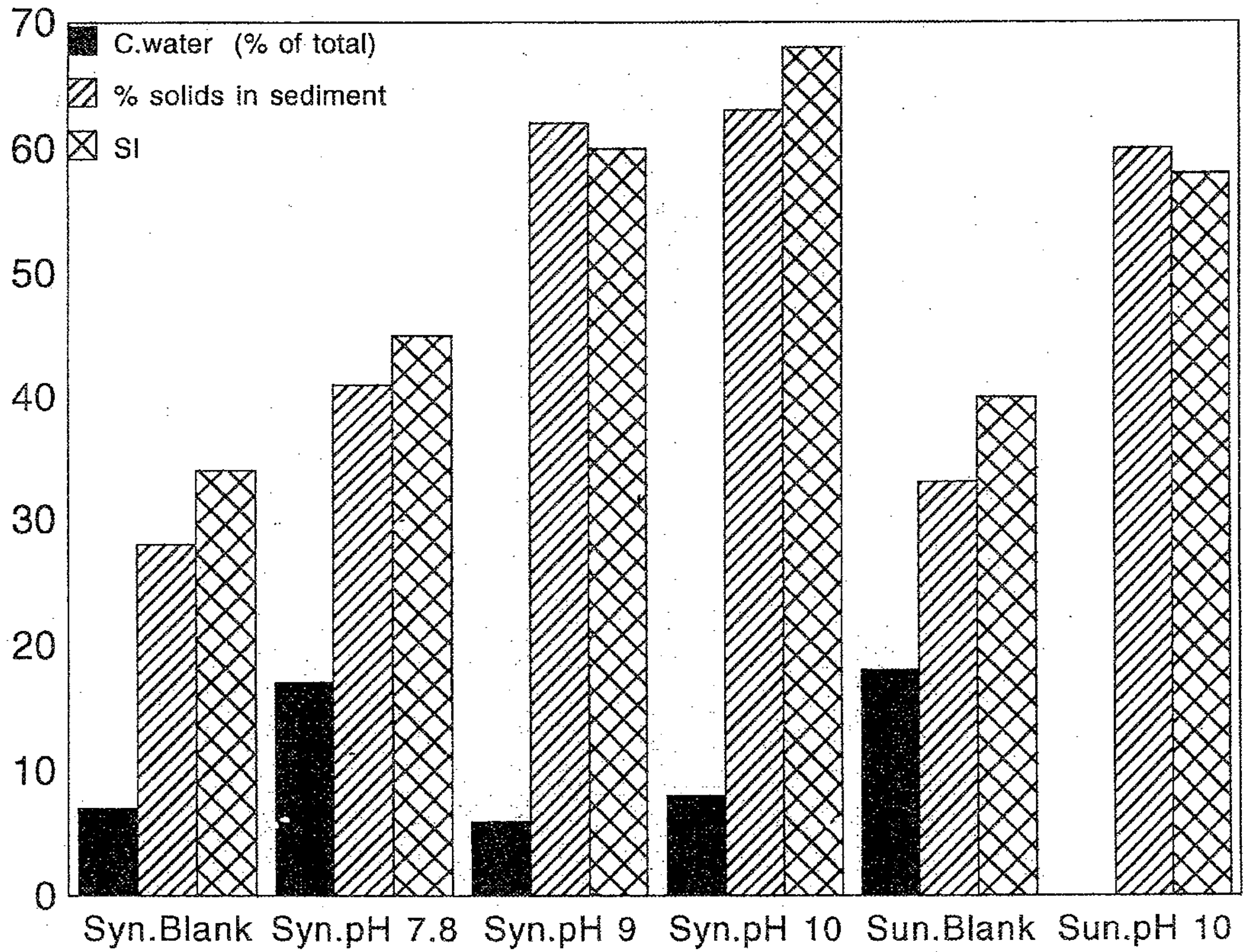

Figure 1. Gravity Settling data for Treatment-1 (Whole Sludge)

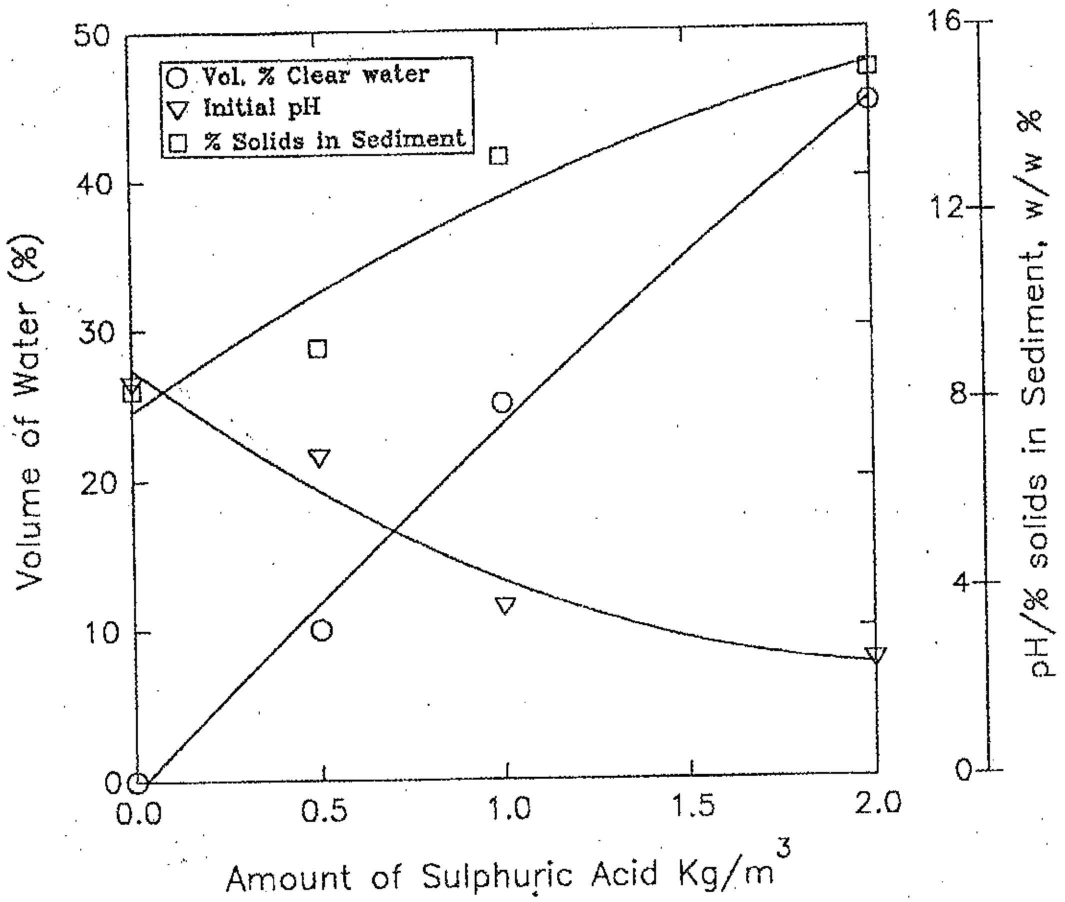

Figure 2. Dewatering of Colloidal Suspension from Treatment-1 with Sulphuric Acid