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THIS IS A PREPRINT - SUBJECT TO CORRECTION

A STUDY OF FACTORS AFFECTING THE STABILITY OF TAILINGS SLUDGE PRODUCED BY THE HOT WATER BITUMEN EXTRACTION PROCESS

BY

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THIS PAPER IS TO BE PRESENTED AT THE CIM/AOSTRA 1991 TECHNICAL CONFERENCE IN BANFF, APRIL 21-24, 1991. DISCUSSION OF THIS PAPER IS INVITED. SUCH DISCUSSION MAY BE PRESENTED AT THE TECHNICAL MEETING AND WILL BE CONSIDERED FOR PUBLICATION IN CIM JOURNALS IF FILED IN WRITING WITH THE TECHNICAL PROGRAM CHAIRMAN PRIOR TO THE CONCLUSION OF THE MEETING.

Introduction

The Hot Water Extraction Process (HWE) is presently used in Alberta to extract bitumen from surface mineable oil sands. Tailings, remaining after bitumen extraction, are disposed of in sedimentation ponds where the coarse sand and some of the fines settle rapidly to form the dykes surrounding the pond. About one half of the silt and clay and almost all of the unextracted bitumen remain in suspension and are carried out towards the centre of the pond. Eventually, these solids form a sludge having a gel-like structure and containing a significant proportion of the process water (1).

In previous work (2) we have used selective separation techniques to identify several solids fractions, which although occurring in relatively minor amounts in sludge, have the capability of contributing significantly to its structure. These same solids fractions have also been found in the parent oil sands ore and the corresponding fresh tailings. Of special interest is a colloidal solid material showing a tendency for gel formation. Other fractions are associated with various amounts of organic matter and may be separated based on their surface active nature; a particularly

useful technique is oil assisted or emulsion flotation in combination with centrifugation. These partly oil wettable solids are also capable of forming structures through interaction with free bitumen and naphtha, also present in the sludge. This type of structure formation would involve the process of immiscible liquid phase wetting (3).

It has been demonstrated (4) that when sludge is treated under certain conditions to remove selectively the more hydrophobic materials, the gel structure can be broken. The bulk of the solids then settle out as a dense sediment leaving a dilute suspension of ultra-fine particles comprising only about 20% of the original solids. These results indicate that the concentration of solids necessary for gel formation may be critical and that separation of only a small fraction of solids is required to disrupt or prevent the formation of sludge.

Solids Fractionation and Distribution

With some minor modification the separation scheme, shown in Figure 1, can be used to fractionate oil sands ore, sludge or tailings. The only change required is that, for sludge or tailings, a certain amount of the connate water must first be removed by centrifugation,

followed by replacement with fresh water. After vigorous agitation and recentrifugation the original sample will then form three layers which are readily separable from each other. On top is a bitumen layer containing hydrophobic solids (BS) closely associated with significant amounts of strongly bound organic matter. These solids probably do not participate in sludge structure but can reduce bitumen separation by increasing its density. A second, aqueous layer, containing a stable dispersion of ultrafine particles (AS) forms between the bitumen and a compacted sediment, containing the bulk of the residual solids (RS). The colloidal, or ultrafines suspension only forms during this particular sequence of treatments.

Further size separation of the ultrafines can be achieved by centrifugation at different speeds. Also, organic rich solids (ORS) having intermediate wettability towards hydrocarbons may be removed from both the bulk solids layer and each of the different sized fractions from the colloid suspension. The treatment involves addition of controlled amounts of toluene followed by vigorous agitation and centrifugation. The surface-active solids and toluene form an emulsion phase which floats on the surface of the water and which can be skimmed off for separation. This procedure can be repeated until all of the emulsion stabilising solids are removed from the various fractions. Similar treatment of the AS solids results in separation of another surface active material defined as "skin" solids.

Table I lists the various fractions and the amounts in which they occur in oil sands and sludge samples. The numbers in parentheses represent the amount of each of the fractions, calculated as a percentage of the fines (<38 μm) content of the oil sands ore rather than of the total solids. When this calculation is done the amounts of each of the fractions are of the same order of magnitude as those found in sludge samples. This indicates that even high grade ore contains precursors in about the right proportions to form sludge. Lower grade ores simply contain more of these intractable materials.

Composition of Fractions

Although some data is missing the results shown in Tables II, III and IV demonstrate that equivalent fractions, separated from a number of different samples of oil sands ore, fresh tailings and sludge, have quite similar compositions. This indicates that the potential for sludge production exists within the ore body itself and is not necessarily a consequence of changes resulting from process conditions. One

property that is significantly different in the sludge compared to tailings or ore is the insoluble organic carbon content (IOCC). This is particularly noticeable for the ORS fraction where the samples separated from sludge always have a lower IOCC. Examination of subfractions of the AS and 'skin' samples, produced by centrifugation at different speeds, shows that IOCC increases with decrease in particle size, see Table V. This observation may simply be a reflection of increased surface area and the amorphous character of the finer particles. Again, comparison between equivalent fractions from oil sands and sludge shows a drastic decrease in IOCC for the latter cases. This may reflect the result of bacterial action in the pond; whether this has an adverse or beneficial effect on sludge behaviour is not known at present.

Characterisation of the IOCC associated with some solids fractions shows it to be a very complex mixture (5); Table VI summarises the available information. Both oil sands and sludge contain about the same amount of alkali soluble humic matter. The predominance of aromatic carbon in this material indicates that it originates from terrestrial sources. The humic matter also shows a high degree of maturation which indicates that it was present in the reservoir prior to migration of the oil. Supercritical fluid extraction with methanol removes a non-humic organic fraction containing a large proportion of long chain fatty acids. These compounds may be the residue from the degradation of biopolymers which have been protected from further attack by being adsorbed onto mineral matter; long chain acids are virtually absent in bitumen. About fifty percent of the IOCC is totally insoluble and has been classified as humin, a fraction that has not yet been well characterised.

The nature and conformation of adsorbed organic species affects the surface wettability of the mineral solids. An adhesion technique (6) has been used for surface characterisation of several of the organic rich mineral fractions. The small, positive values for the spreading coefficient with toluene, S_{toluene} , in Table VII show that for all these fractions, wetting by low surface tension liquids (eg. hydrocarbons) would be favoured. Sample BS, having the highest organic content and contact angle, occurs entirely within the bitumen phase, whereas the other two samples tested, preferentially collect at water/organic interfaces. Normally, a contact angle of 90° or more would be required to permit complete engulfment of a solid by a non-interactive organic phase. The fact that such an occurrence is noted for the BS sample indicates a strong interaction between the adsorbed matter on this fraction and bitumen; this does not seem to be the

case for the other samples tested.

Mineralogy and Amorphous Material

X-ray diffraction has been used for the identification and quantification of minerals present in sludge and its sub-fractions. Kaolinite is the major component with smaller amounts of mica, microcline, quartz and other minerals also being present. This technique can also be used to determine non-crystalline or amorphous mineral content. A summary of results for some colloidal sub-fractions separated from both oil sands and sludge are shown in Table VIII. These fine fractions all exhibit a high and increasing amorphous content with decreasing particle size.

Treatment with alkaline Tiron (1,2-dihydroxybenzene-3,5-disulphonic acid disodium salt) preferentially extracts noncrystalline silica and aluminum and can also be used to determine amorphous material (7). Although the results obtained by the Tiron treatment showed the same trends as the x-ray diffraction data, the absolute amounts of noncrystalline material determined was almost an order of magnitude less in the former case. There are several possible explanations for this discrepancy. It is known that fine particles cause peak broadening in x-ray diffraction and that this results in errors in determining crystalline and hence also non-crystalline content. Also, x-rays do not penetrate deeply and hence, a thin surface layer of amorphous material, encapsulating a crystalline mineral, will distort the results; Tiron will easily remove such a surface layer, which could represent only a small proportion of the mineral mass. So far, this inconsistency in results has not been resolved.

Solid state, magic angle spinning (MAS), nuclear magnetic resonance (NMR) is a useful tool in the identification of minerals and for providing information on their molecular environment. The ^{27}Al MAS-NMR spectra for some oil sands derived, solids fractions, with different particle sizes, are shown on Figure 2. Three progressively finer fractions of the residual solids (RS) and two centrifugally sized colloidal fractions are shown (AS-500 g, settled at 500 g and AS-U/S, unsettled at 8000 g). For the coarser RS fractions the only Al bearing mineral is microcline, having a well defined peak at 52 ppm. With the finest RS fraction, a second peak at 4 ppm characteristic of octahedral Al in kaolinite, appears.

The colloidal fractions also distinctly show the main octahedral Al peak for kaolinite; microcline is now absent but a broad resonance at around -56 ppm has appeared. The latter peak results from Al in

tetrahedral coordination and is characteristic of disordered kaolinites (7). The intensity of this signal has been related to crystal structure breakdown; the increase in peak size observed for AS-U/S compared to AS-500 g could reflect a greater contribution of poorly crystalline material in the former case.

^{29}Si MAS NMR is a particularly useful tool for studying poorly crystalline solids. Figure 3 shows the ^{29}Si NMR patterns for some centrifugally sized colloidal fractions (AS), separated from sludge. For comparison purposes the spectrum for the whole residual solids fraction is also shown; this latter spectrum exhibits resonance at -92 ppm characteristic of kaolinite, a shoulder at 85 ppm indicative of mica and resonance at 108 ppm due to quartz. The AS solids show no resonance for quartz but exhibit broad peaks at the chemical shift (-92 ppm) representative of kaolinite. Such a broad resonance, in the range -80 to -110 ppm is typical for synthetic aluminosilicate gels and allophane (8) and indicates a disordered environment. The increase in broadness with decrease in particle size may reflect an increasingly smaller contribution of crystalline components as particle size decreases. Another factor which can also influence the peak width is the presence of ferro- and para- magnetic species, such as iron. However, for these AS fractions the difference in iron content is not enough to cause a significant effect.

In summary the results of ^{29}Si and ^{27}Al NMR characterisation indicate that aluminosilicates, having the same chemical shifts as kaolinite are the main inorganic components of the isolated colloidal fractions. Also, the degree of molecular disorder (amorphous character) increases with decrease in particle size.

Gel Forming Characteristics of Colloidal Particles

Ultrafine particles are believed to be a major factor in the poor settling and compaction of tailings pond sludge. Some of the colloidal fractions separated in this work have been noted to form gels on standing. There is considerable interest in relating this phenomena to sludge behaviour; in order to do this it is important to understand how the gel microstructure is formed.

Recently (9), it has been shown that the structure and dynamic behaviour of water molecules near surfaces of 2:1 phyllosilicates can be probed with a quadrupole nucleus such as deuterium. This approach is based on the fast exchange of D_2O molecules between bulk water and a charged solid surface. Typically, the

formation of an ordered structure in the applied magnetic field results in a splitting of the deuterium resonance for D_2O . Such behaviour has been reported for a montmorillonite gel in water (9). This observation points to the existence of a local order among the colloidal particles which in turn orders the water molecules closest to the clay interface.

Figure 4 shows the concentration dependence of the deuterium resonance splitting; the relationship between this residual splitting and concentration of colloidal solids is linear. Providing that aggregation of colloidal particles occurs even in dilute suspension, then these results indicate an increase in the number of aggregates rather than in their size as concentration of suspended solids is raised. The observed residual splitting is proportional to the mole fraction of water at the periphery of the aggregates and to the quadrupole coupling constant.

Another important observation is that the residual quadrupole splitting for the gel forming solids is time dependent. This is illustrated on Figure 5, which shows the residual splittings for a suspension, measured immediately after preparation and then after one week and one month of standing undisturbed. Gel formation occurs when a network of particles is created. Such an arrangement is aided by particles contacting each other at selected spots while still repelling each other over the rest of their surfaces. The so-called "card house" structure of clay platelets with oppositely charged faces and edges is one such network. Network formation limits the ability of particles to orientate in a magnetic field; also, it may result in a decrease in the total periphery of the particles. Both of these factors affect the local order of the colloidal solids and, in turn, the degree of ordering of water molecules closest to the solid-water interface. Further work in this area is continuing to determine critical gel formation for different colloidal fractions in contact with pond water. Model systems will also be used to determine the range of mineralogical characteristics of solids exhibiting this type of behaviour.

Settling Characteristics of Treated Sludge

Techniques such as liquid-phase agglomeration (3) (oil phase agglomeration) can be used to remove selectively a hydrophobic component from a generally water wet system. It has been found that when sludge is treated in this way to separate free hydrocarbons and hydrophobic solids, its settling characteristics are markedly altered (4). Figure 6 is a photograph of settled sludge samples in which each has been

pretreated in some way. The sludge in the centre cylinder was mixed under the same conditions as the other two but was otherwise unmodified. The sample in the cylinder on the right was treated with sodium silicate and mixed, while the remaining sample on the left was additionally subjected to oil phase agglomeration. The two cylinders on the right therefore represent reference or blank samples. Comparison of settling after ninety days showed that the two reference samples remained largely undifferentiated; in the sample treated with sodium silicate a narrow band of brownish, clear water separated but otherwise it resembled the original sludge. However, the sample treated to remove free organics and hydrophobic solids separated into three distinct layers none of which showed any of the unusual gel-like properties of the original sludge.

Figure 7 summarises the change in interface heights as a function of settling time for the three samples. Each of the layers was separated and analysed. The bottom sediment layer comprised light coloured and relatively clean kaolinite and quartz; it most closely resembled the residual solids (RS) in the fractionation scheme described earlier. A narrow, dark brown, interface layer separated the clean sediment from a dilute suspension of grey, colloidal solids. The interface layer corresponded to the organic rich solids (ORS) while the suspension showed the same characteristics as the colloid fraction (AS). On average the three layers contained the following percentage of total sludge solids: RS (75 w/w %), ORS (5 w/w %) and AS (20 w/w %). The corresponding solids concentrations were 60 w/w %, 40 w/w % and 8 w/w % in the three layers respectively.

The oil phase agglomeration treatment removed 98.5 w/w % of the free organics and 4.2 w/w % of the solids present in the sludge. Removal of lesser amounts of organics and hydrophobic solids also resulted in disruption of the sludge structure, although separation into layers did not occur so cleanly or quickly in these cases. The fact that removal of only a small fraction of solids results in sludge destabilisation indicates that the concentration of certain solids fractions is critical to sludge formation. At present the nature of the solids separated has not been established. Removal of free organics also eliminates the possibility of structure formation by solids flocculation through liquid phase bonding between partly hydrophobic solids (10).

Conclusion

A number of solids fractions, separated from sludge, have been identified as having the capability of forming

have been identified as having the capability of forming the type of structures observed in sludge. The total amount of these intractable components amounts to about 20 w/w % of the total sludge solids. The bulk of the solids, consisting mostly of coarser quartz and kaolinite particles, appear to be trapped in the sludge without contributing much to its structure.

The colloidal solids found in sludge will form gels only above a critical concentration. The nature of these ultrafine solids has not yet been well established, but preliminary studies indicate that they are highly disordered, kaolinite type clays. There is some indirect evidence that montmorillonite also may be present. Other solids fractions are associated with strongly bound organic matter which imparts intermediate wettability characteristics to their surfaces. Such partly hydrophobic solids are also capable of forming structures through an immiscible phase wetting mechanism.

Acknowledgement

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Fraction	DISTRIBUTION OF SOLIDS (Percent (%w/w) of Total Solids on Bitumen and Water Free Basis)			
	Oil Sands		Sludge	
	High Grade	Low Grade	Syncrude	Suncor
Total Fines ($\leq 38 \mu\text{m}$)	10.4	25.7	98.0	95.0
BS	0.1 (0.8) ^a	tr.	tr.	0.8
A	1.2 (10.0)	5.5 (17.4)	18.4	32.4
ORS	0.3 (2.5)	2.0 (6.3)	5.2	7.9
RS	98.4	92.5	76.4	58.9

Table I: Comparison of Solids Distribution by Fraction

(a) Values in parenthesis recalculated as percent of fines content.

Fraction ID	Description	Percent of Total Solids (%w/w)	Composition (%w/w)			Particle Size Range (μm)	Contact Angle (water wettability)
			IOCC	Fe	Al		
Total Fines	Nominally $\sim 38 \mu\text{m}$	10.4 - 25.7	0.1 - 0.5	1.0 - 1.4	5.0 - 5.3	≤ 38	NA
A	Colloidal Solids	1.2 - 5.5	NA	NA	NA	0.01 - 1	NA
AS	Colloidal Solids with 'skin' removed	0.8 - 4.4	3.4 - 4.4	1.1 - 2.0	9.6 - 11.4	NA	69.5
'Skin'	Surface active - Interface Solids	0.4 - 1.1	10.0 - 16.3	2.5 - 3.8	5.5 - 8.5	NA	68.2
ORS	Emulsion Stabilising Solids	0.4 - 2.0	20.0 - 41.0	6.4 - 6.5	1.3 - 5.3	≤ 100	NA
RS	Residual Solids	92.5 - 98.4	NA	NA	NA	< 250	NA

Table II: Description and Composition of Solids Fractions from Oil Sands

Note: NA - result not yet available

Fraction ID	Description	Percent of Total Solids (%w/w)	Composition (%w/w)			Particle Size Range (μm)
			IOCC	Fe	Al	
Total Fines	Nominally $\sim 38 \mu\text{m}$	10.0 - 23.3	0.3 - 0.5	NA	NA	≤ 38
A	Colloidal Solids	1.1 - 3.8	NA	NA	NA	0.01 - 1
AS	Colloidal Solids with 'skin' removed	0.8 - 3.2	3.2 - 4.1	NA	NA	NA
'Skin'	Surface active - Interface Solids	0.3 - 0.6	12.3 - 13.1	NA	NA	NA
ORS	Emulsion Stabilising Solids	0.2 - 1.9	18.3 - 36.1	NA	NA	≤ 100
RS	Residual Solids	94.3 - 98.7	NA	NA	NA	< 250

Table III: Description and Composition of Solids Fractions from Fresh Whole Tailings

Fraction ID	Description	Percent of Total Solids (%w/w)	Composition (%w/w)			Particle Size Range (µm)
			IOCC	Fe	Al	
Total Fines	Nominally <38 µm	95 - 98	NA	NA	NA	≤38
A	Colloidal Solids	18 - 33	NA	NA	NA	NA
AS	Colloidal Solids with 'skin' removed	14 - 30	0.8 - 1.3	1.4 - 2.1	11.9 - 14.0	NA
'Skin'	Surface active - Interface Solids	3 - 4	12.2 - 13.9	2.6 - 3.7	9.0 - 10.2	NA
ORS	Emulsion Stabilising Solids	5 - 8	14.0 - 14.9	11.2 - 13.8	4.0 - 5.8	≤100
RS	Residual Solids	59 - 77	0.3 - 0.8	0.7 - 1.1	9.7 - 10.0	<250

Table IV: Description and Composition of Solids Fractions from Sludge

Fraction ID	INSOLUBLE ORGANIC CARBON CONTENT (%w/w)			
	Oil Sands		Sludge	
	High Grade	Low Grade	Syn crude	Suncor
AS-500 g	2.0	1.3	0.6	0.6
AS-1500 g	2.4	2.3	0.7	1.2
AS-8000 g	4.1	7.3	0.9	1.3
AS-u/s	7.5	11.0	1.7	2.3
Skin-500 g	7.9 (35.0)	12.6 (29.9)	14.4 (21.8)	18.3 (4.5)
Skin-1500 g	8.0 (26.9)	20.6 (14.9)	13.7 (18.4)	10.5 (16.2)
Skin-8000 g	12.9 (35.6)	20.0 (17.0)	14.1 (20.5)	11.1 (12.4)
Skin-u/s	14.5 (35.8)	19.9 (13.5)	13.6 (22.8)	8.0 (11.3)

Table V: Insoluble Organic Carbon Associated with Colloidal Solids Fraction

(Values in parentheses reflect percentage of total fraction represented by each subsample. Numeric suffixes in fraction IDs represent separation g force; u/s is sub-fraction unsettled at 8000 g)

Fraction	Organic Content (Percent of Total SOM) ^c	Comments
Humic Matter^a (alkali solubles)		<ul style="list-style-type: none"> Aromatic carbon is predominant in both cases Humic matter is largely terrestrial in origin ¹³CNMR suggests high degree of maturation
Oil Sands	29.0	
Sludge	26.0	
Non-Humic Matter^b		<ul style="list-style-type: none"> Long chain fatty acids are main contributor to SFE extracts These acids are virtually absent in bitumen Low MW asphaltic fractions could also report to SFE extract
Oil Sands	23.8	
Sludge	27.4	
Humins^d		<ul style="list-style-type: none"> Totally insoluble residue Not well characterised
Oil Sands	47.2	
Sludge	53.4	

Table VI: Description and Origin of Strongly Bound Organic Matter from Sludge and Oil Sands

- a. Removed from organic rich solids (ORS) by extraction with 2% NaOH solution
b. Separated by SFE with Methanol
c. Calculated from initial carbon content assuming organic material contains 70% carbon
d. By difference

Sample	γ_{sv}^\dagger dyne/cm.	S _{Water}	S _{Toluene}	θ^* (°)	IOCC (^{w/w} %)
		(25°C)	(25°C)		
Bitumen Solids (BS)	42.7	-53.5	11.2	74.8	42.1
'Skin' - solids	48.6	-45.6	14.1	68.2	12.8
Colloidal Solids (AS)	47.4	-47.1	13.6	69.5	12.8

Table VII: Surface Properties of Solids Fractions from Oil Sands

† Solid-vapour interfacial tension.
* Contact angle with respect to water.

Sample Fraction ID	Amorphous Content (^{w/w} %)	
	X-ray Diffraction	Tiron Treatment
High Grade Oil Sands		
AS-500 g	59	7.4
AS-1500 g	69	9.9
'Skin'-500 g	68	5.6
'Skin'-1500 g	80	9.5
Syncrude Sludge		
AS-500 g	50	2.9
AS-1500 g	60	10.2
AS-u/s	80	29.0
'Skin'-500 g	50	5.3
'Skin'-1500 g	80	8.1
'Skin'-u/s	90	12.8

Table VIII: Poorly Crystalline or Amorphous Content of Colloidal Solids Fractions

(Numeric suffixes in fraction IDs represent g force used for size separation; u/s is unsettled material at 8000 g)

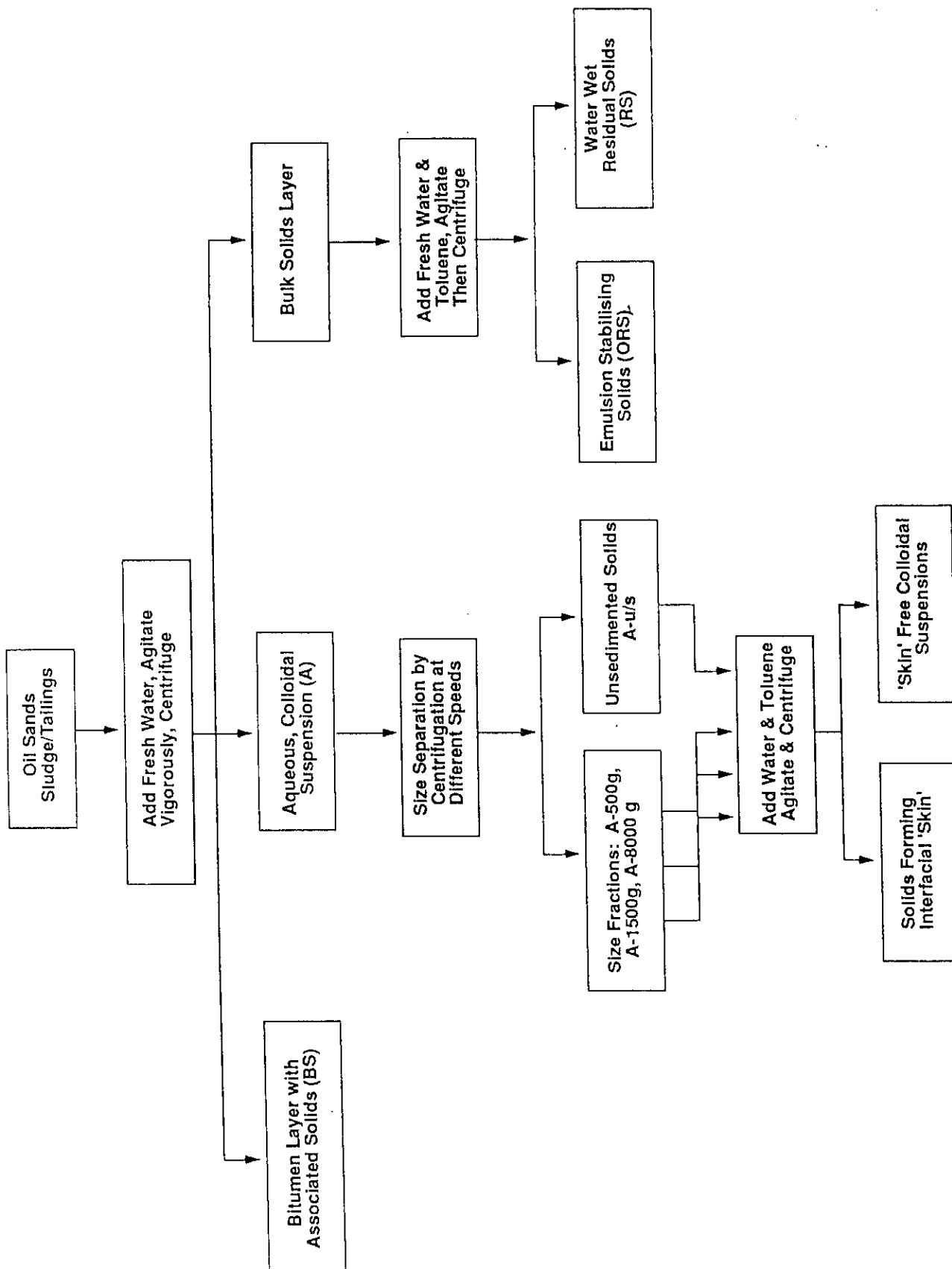


Figure 1: Treatment Scheme for Component Separation

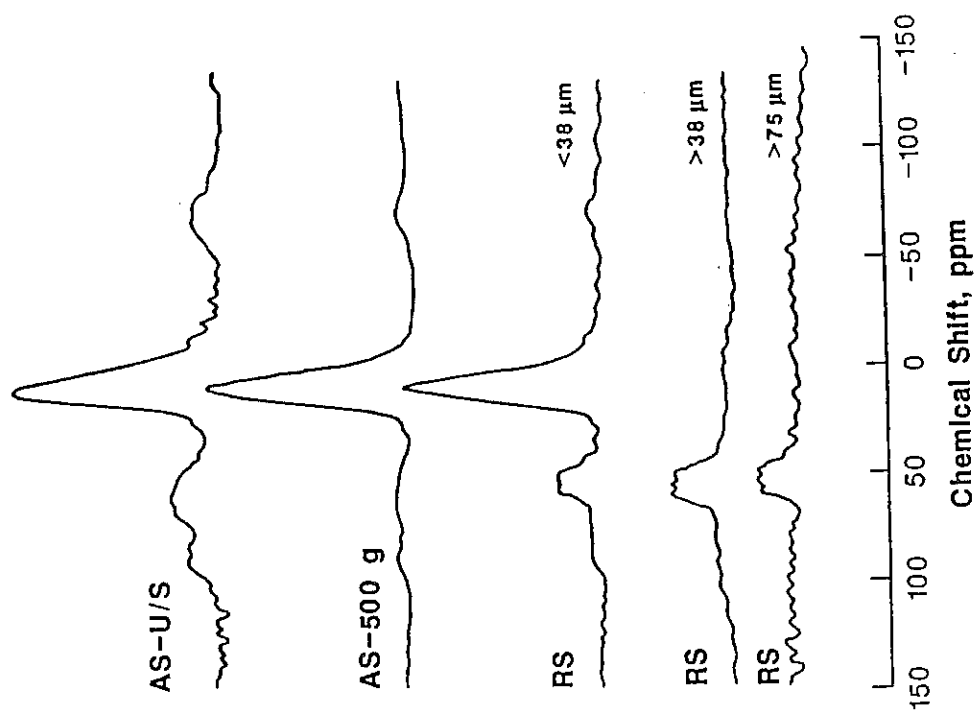


Figure 2: Solids Fractions - ^{27}Al MAS NMR Spectra

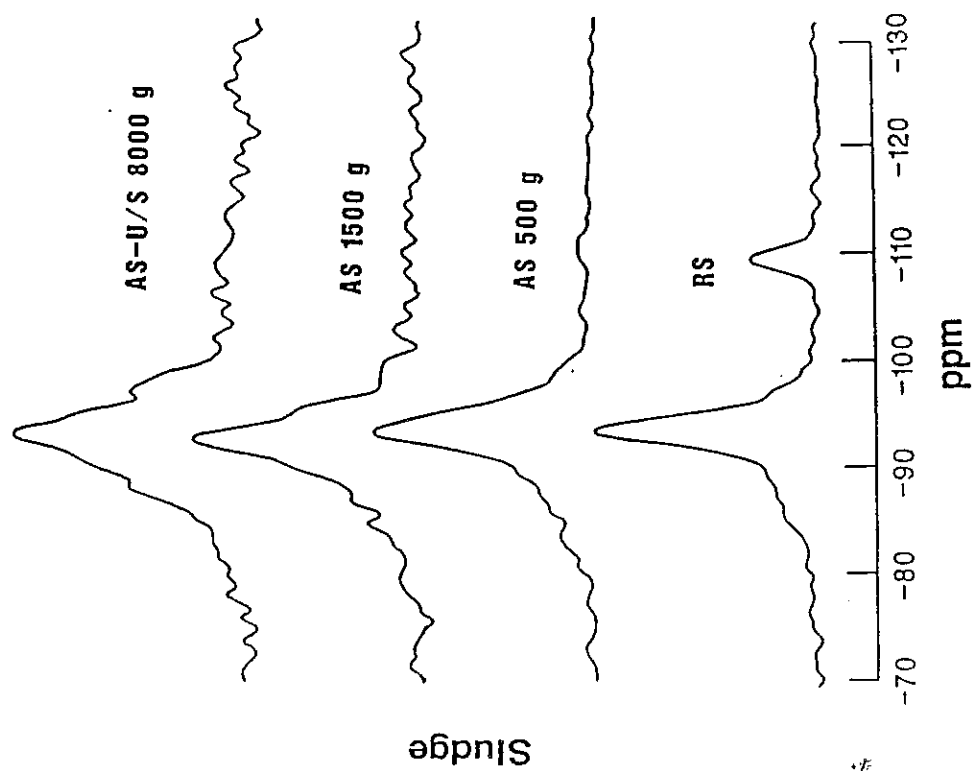


Figure 3: Solids Fractions - ^{29}Si MAS NMR Spectra

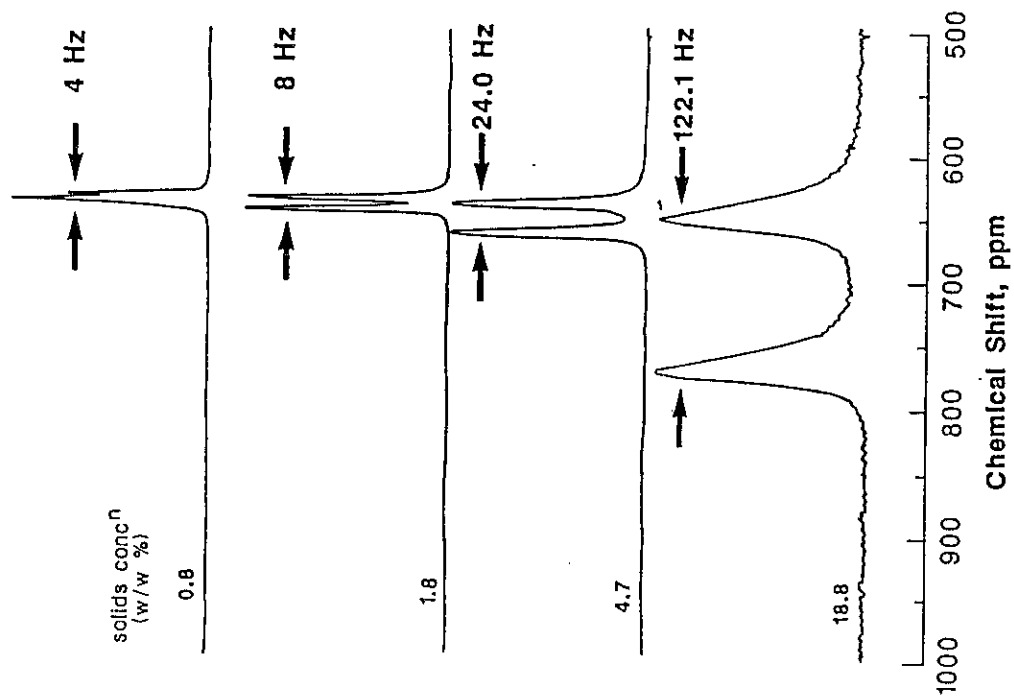


Figure 4: Deuterium Resonance Splitting for Colloidal Suspension

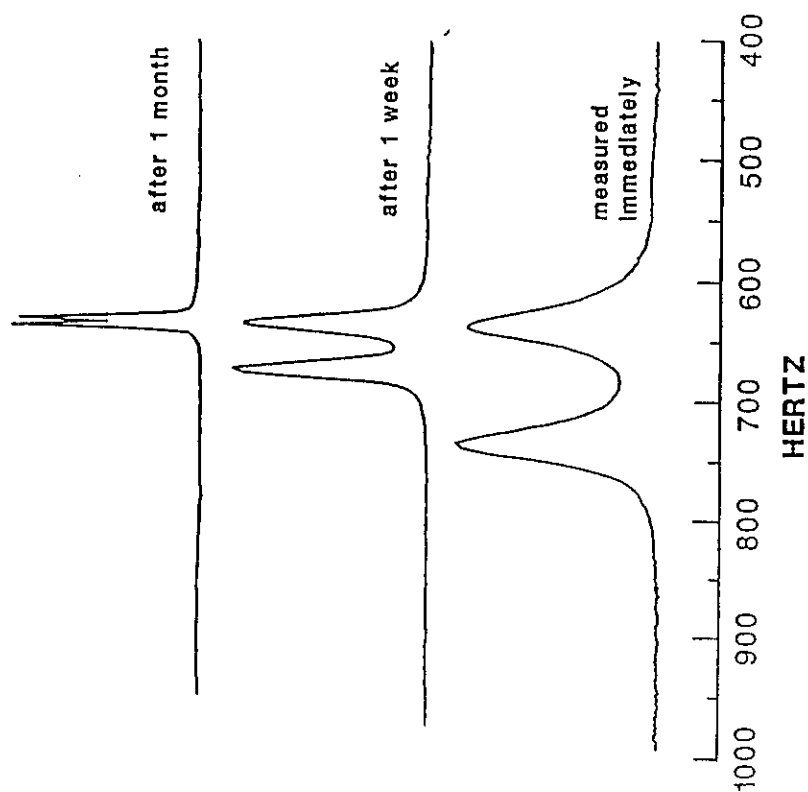


Figure 5: Time Dependence of Deuterium Resonance Splitting for Colloidal Suspension

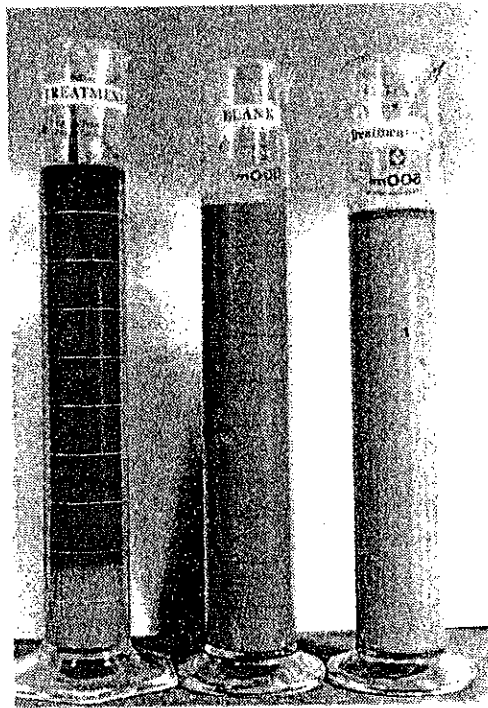


Figure 6: Settling Characteristics of Sludge Samples After Different Treatments

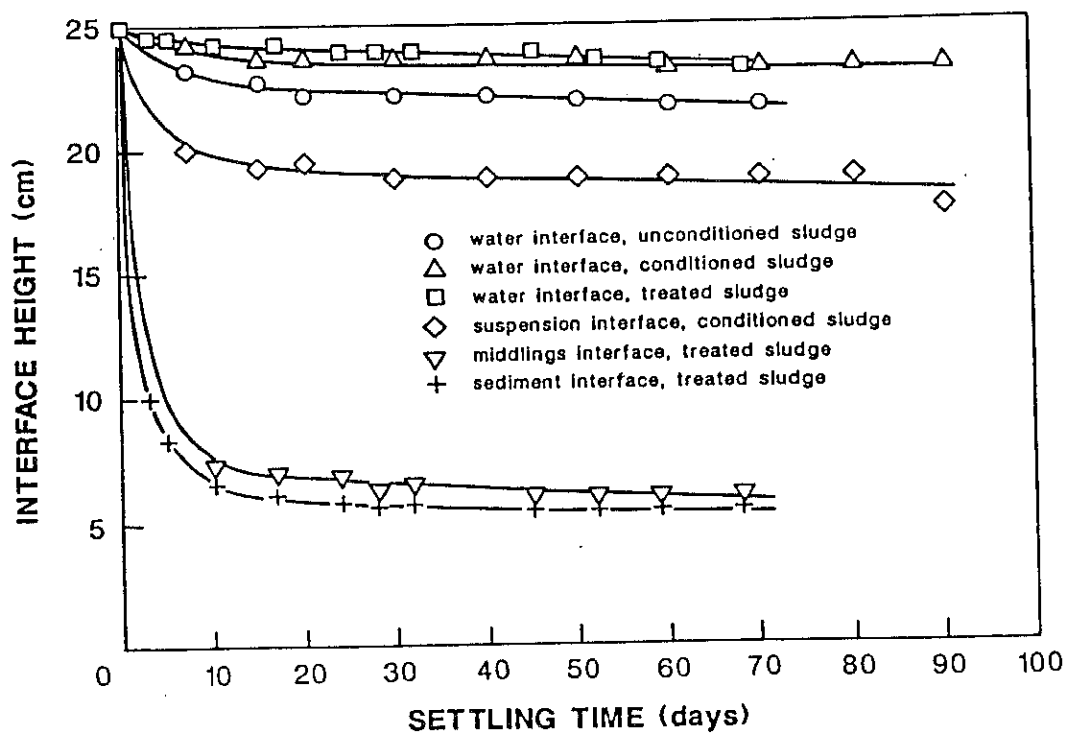


Figure 7: Settling Curves for Treated and Untreated Syncrude Sludge