JOINT SYMPOSIUM ON OIL SHALE, TAR SANDS, AND RELATED MATERIAL
PRESENTED BEFORE THE DIVISION OF PETROLEUM CHEMISTRY, INC.
AND THE DIVISION OF WATER, AIR, AND WASTE CHEMISTRY
AMERICAN CHEMICAL SOCIETY
SAN FRANCISCO MEETING, April 2-5, 1968

HOT WATER PROCESSING OF ATHABASCA OIL SANDS: II. MICROSCOPE STUDIES OF THE CONDITIONING PROCESS

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INTRODUCTION

This paper will consider in detail conditioning, the initial step in the hot water extraction process for the Athabasca oil sands. This term, carried over from extractive metallurgy, refers to the step in which the raw ore is converted to a form from which the desired fraction can be separated from tailings. Seitzer (1) has described experiments which showed the importance of vigorous mechanical working in producing excellent yields of froth even from grades of oil sand as low as 6 percent oil. In this paper we will be concerned with the reason why this mechanical energy input procedure is so effective and if this information can be of help in understanding procedures which are more amenable to scale-up, (for example, procedures using large-scale equipment more immediately available). From this view we had the following objectives:

(a) We needed to understand the chemistry and physics of conditioning so that scale-up could be made with confidence.
(b) A lower primary froth yield from conventional laboratory methods (2) as compared to the pilot plant must be improved or explained.
(c) We needed procedures that were comparable in all respects to large scale methods so that valid laboratory studies could be made on later steps in the process and on variations in the properties of oil sand samples.

Most workers on Athabasca oil sands have, of course, made use of the microscope (3). Early studies in our laboratory were useful, for example, in demonstrating the powerful surface forces that are operating in freeing the oil from sand particles. The need for some minimum amount of shear is easily shown. However, it was only with new techniques that we began to obtain fundamentally useful leads. The first observations were made through the glass wall of the stirred reactor. We then developed a more generally applicable method of quick-freezing samples in the pulp matrix and examining cleavage surfaces.

Observations on Bitumen Morphology in the Stirred Reactor Under High-Energy Conditions

The stirred reactor described by Seitzer (1) has the broad U-shaped blade passing within 4 mm. of the glass wall of the reactor. As the stirring rate is stopped or slowed down, a wealth of interesting observations can be made at the glass interface on the size and shape of the bitumen particle and size and distribution of air bubbles. The controlling variables are the quality of the oil sand and the amount of alkali.

EXPERIMENTAL

A Unitron low-power binocular microscope (range of magnification 5X to 90X) with inclined eye-pieces was removed from its normal stand and mounted horizontally on a heavy adjustable frame such that it could be focused through the walls of the heating bath and reactor and onto the contents of the flask. Sharp focus was only possible when walls of bath and reactor were quite close together. Lighting was provided by two 100 watt illuminators.

A Nikon-F 35 mm reflex camera fitted with a 1/2 X microscope adaptor was used to record visual observations of pulp morphology. With full illumination from two 100 watt illuminators, good photographs could be obtained at a magnification of 4 X with 1/4 or 1/8 sec. exposure using Ektachrome B film (ASA 125). In rapidly changing situations (i. e. stirring or bubble coalescence, etc.), the Nikon-F was fitted with an extension bellows and a reversed f-2 lens to give total

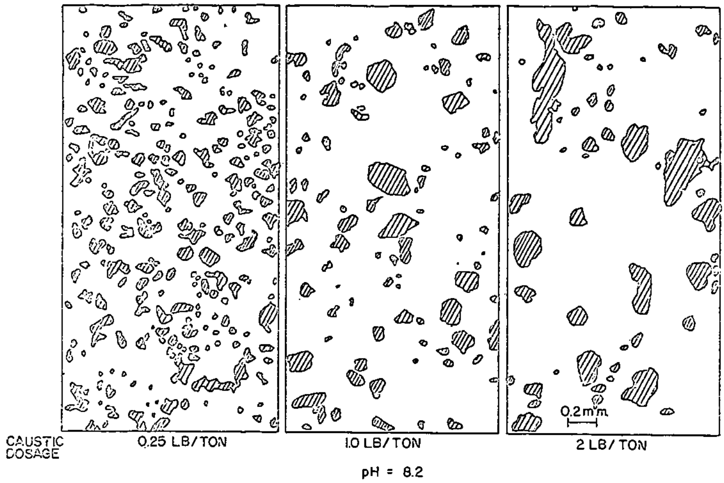

Figure 1. Tracing of oil globules (cross hatched) against clean mineral backgrounds in a photomicrograph of conditioned oil sand. Stirred reactor, 1.75 in. diameter, 1000 r.p.m. Lean oil sand, 8%

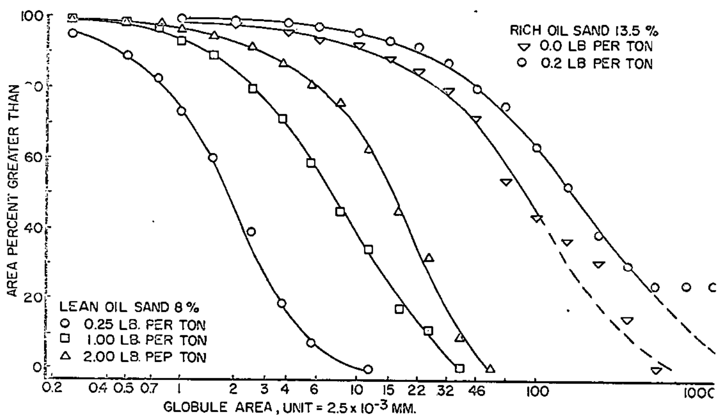

Figure 2. Particle size distribution of oil globules for lean and rich oil sand samples with variation in caustic dosage. Stirred reactor 1.75 in., 1000 r.p.m.

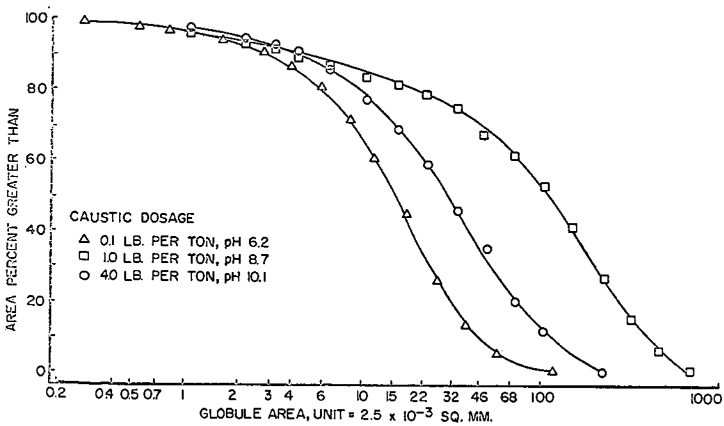

Figure 3. Particle size distribution of oil globules for an average oil sand, 10.7%, (Pit A Lot 25) as a function of pH. Stirred reactor, 3-inch, 350 r.p.m.

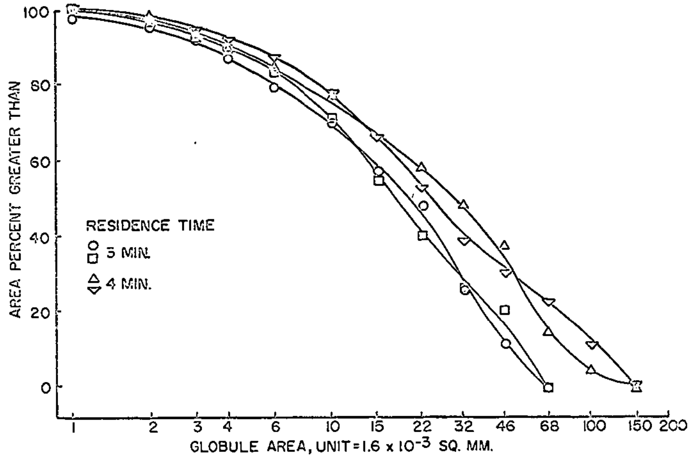

Figure 4. Particle size distribution of oil globules for average oil sand, 10.7%, (Lot 25 Pit A) with variation in residence time. Samples from FTU conditioning drum October 29, 0.55 lb/ton;pond water

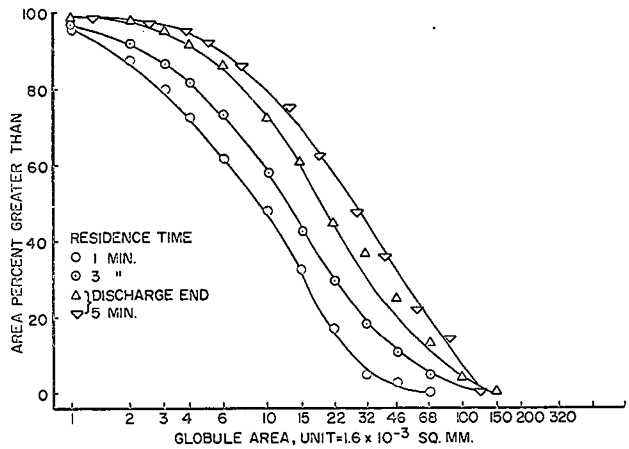

Figure 5. Particle size distribution of oil globules for average oil sand, 10.7%, with variation in residence time. Samples from FTU conditioning drum October 31, 0.45 lb/ton, fresh water.

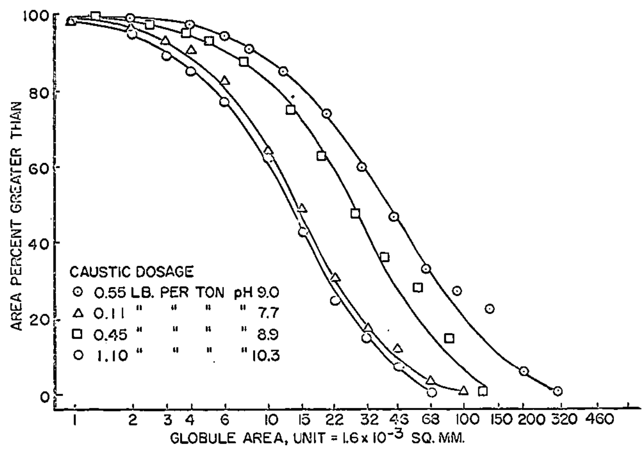

Figure 6. Particle size distribution of oil globules for average oil sand, 10.7%, (Lot 25 Pit A) with variations in caustic dosage. Samples from FTU conditioning drum discharge October 31.

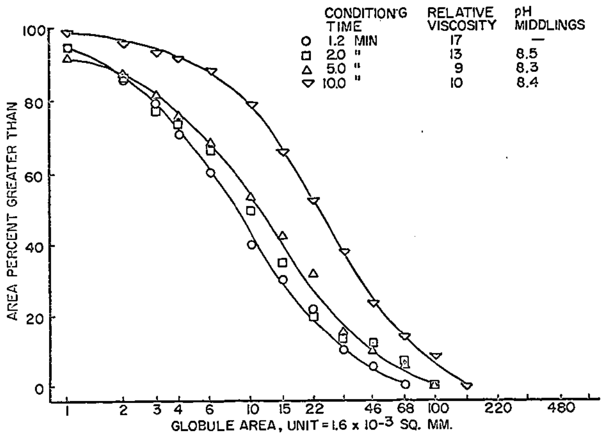

Figure 7. Particle size distribution of oil globules for average oil sand, 10.7% (Lot 25 Pit A) with variation in conditioning time. Samples from 3-inch stirred reactor, 50 r.p.m., 0.5 lb/ton.

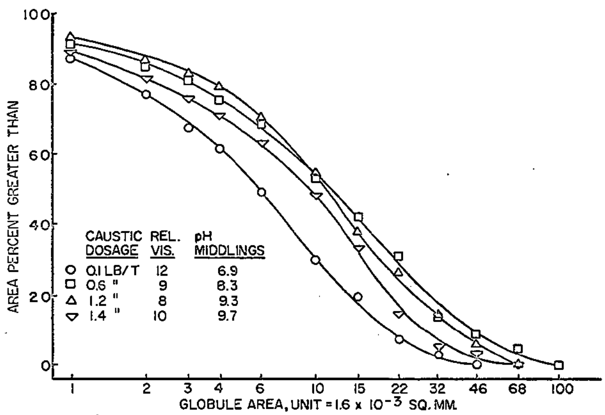

Figure 8. Particle size distribution of oil globules for average oil sand, 10.7% (Lot 25 Pit A) with variation in caustic dosage. Samples from 3-inch stirred reactor, 50 r.p.m., 5 minutes conditioning time.

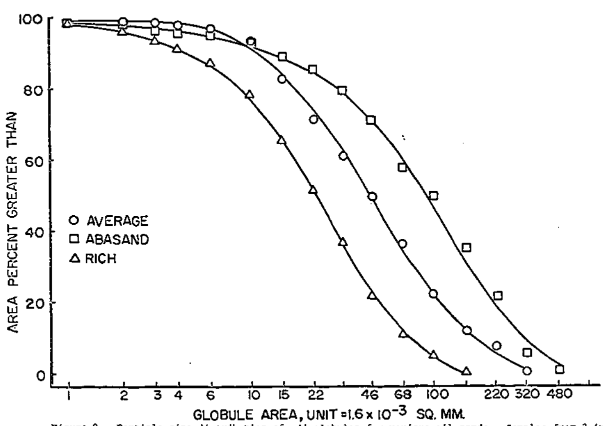

Figure 9. Particle size distribution of oil globules for various oil sands Samples from 3 inch stirred reactor, 100 r.p.m., 10 minute conditioning time, caustic dosage for optimum pH.

Sample	% Oil in Feed	Primary Froth Yield	Relative Viscosity
Average	10.0	60	11
Abasand	14.6	95	24
Rich	13.5	92	20

magnification on the film of 4.5 X. For lighting, an electronic flash was used. This arrangement was quite effective in "freezing" the action.

Visual observations and color transparency photographs provide a wealth of information that is impossible to communicate in black and white prints. Line tracings of bitumen globules of Pit C oil sand are shown in Figure 1. The progressive increase in size of the globules as caustic is added is obvious to the eye, but a description capable of finer discrimination is required if this method is to be applied widely. One solution to this problem is shown in Figure 2, in which the globule sizes from Figure 1 are plotted as a conventional size distribution. The shape of this curve and the size of the median globule provide a characterization adequate for present purposes. This method of representation will be used throughout this paper.

RESULTS

For the studies in the stirred reactor at high speed, all the observations are at steady-state conditions -- no change with time. Poor quality oil sand (Pit C, Figure 2) have in general much smaller oil globules, but the size increases as added caustic soda brings the pH up to the operating level, 8.4. The Pit A sample in Figures 2 and 3 show the characteristic larger size of an average oil sand, but with the same pH response. In addition, Figure 3 shows the decrease in oil-globule size as the operating pH is exceeded; globule size forms a rather wide flat maximum from pH 8.4 to around 10. At this high pH many average and low grade oil sands form emulsions rather than froth. This behavior is associated with a pronounced drop in the interfacial tension at this same high pH.

We had originally hoped that this globule size characterization would provide some measure of the cohesiveness of the oil globules which, in turn, might be related to froth yield. As we have seen, the high fine mineral content of low-grade oil sands is accompanied by a smaller oil globule size, and these samples in general give lower froth yields. However, when the change in globule size with pH is used as a variable, no correlation with froth yield is seen. Representative experimental results are shown in Table 1; in general, this behavior has been observed consistently over a wide range of samples. In the final analysis, working hypotheses which do not involve particle size or cohesiveness have been found to be much more useful.

TABLE 1

TYPICAL RESULTS SHOWING THE ABSENCE OF CORRELATION BETWEEN PRIMARY FROTH YIELD AND OIL GLOBULE SIZE

Caustic Soda lb. per ton	pH	Size of Area Median Globule mm^2	Primary Froth Yield
0	6.1	0.075	30
0.1	6.2	.035	49
0.25	6.7	.038	63
0.5	7.6	.150	70
1.0	8.7	.275	69
2.0	9.6	.338	75
4.0	10.1	.071	76

Further examples of this behavior are presented in Figures 9 and 10.

There were two factors observed which can account for the higher froth yields obtained from the high-speed stirred reactor. In average and high-grade oil sands, informative observations were made on the behavior of the gas bubbles in the globules of bitumen. By the squeezing action in rapid stirring of the pulp, the bubbles were made very small, perhaps less than one-hundredth of the size of the oil drop, and there were very many of them. When the stirrer was stopped, coalescence of the bubbles began immediately and within 10 to 20 seconds most of the gas was in a very few bubbles of the order of one-fourth the size of the globule. With the dynamic interchange of oil among globules which was also observed (continual coalescence and disruption of globules) a very even distribution of air was assured when flooding was performed with no time delay after the rapid stirring. Decreased froth yield is experienced when bubble coalescence is allowed to occur, and globules broken up during flooding may then yield fragments with no bubbles plus fragments containing most of the original flotation gas.

The second factor is perhaps the most important when all grades of oil sand are

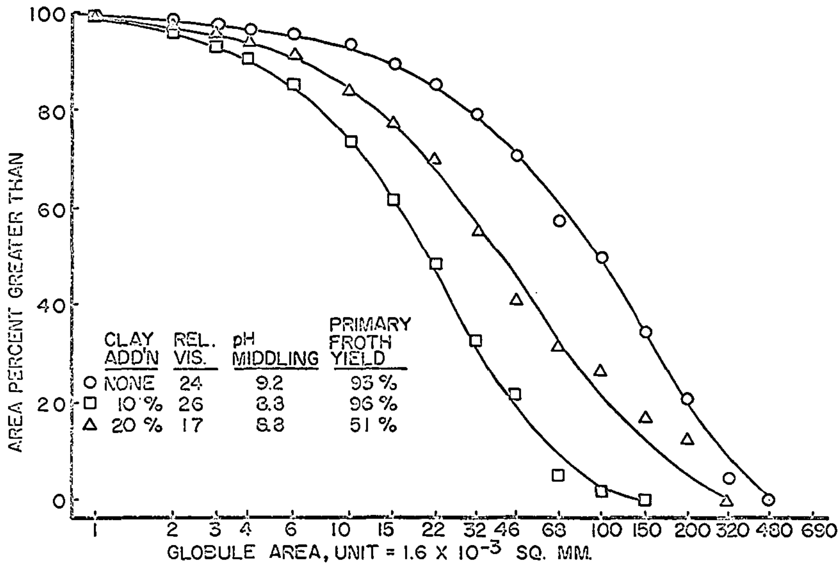

Figure 10. Particle size distribution of oil rich globules for Abasand oil sand with various amounts of seam clay added. Samples from 3 inch stirred reactor, 100 r.p.m., 1.0 lb/ton.

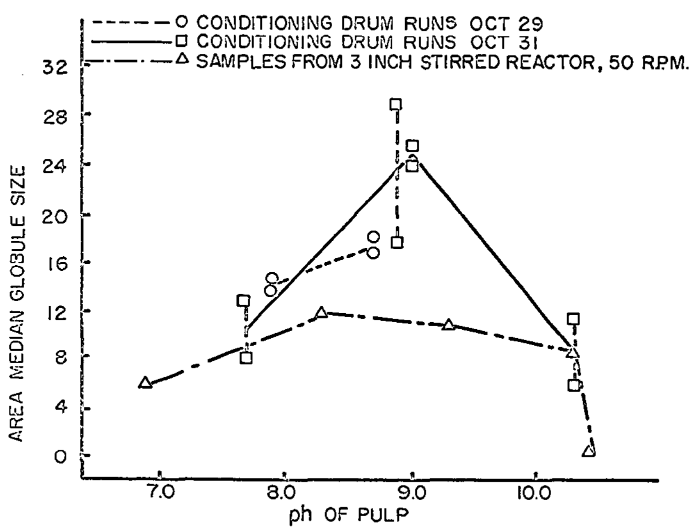

Figure 11. Summary of the change in the oil globule size distribution with pH of conditioning. Area is shown by the area median globule size.

considered. Measurement of the amount of flotation gas showed that rapidly stirred pulp contained about three times as much as pulp produced by several other procedures. Mineral contents of these pulps were all approximately the same. All other things, therefore, being equal, a higher froth yield will result from a higher content of flotation gas.

Observations on the Conditioning Drum in the GCOS Field Test Unit

Early studies on conditioning showed that the rotating drum had a rather large minimum diameter for operability, and laboratory scale work was not feasible. In order to make direct comparisons between drum and laboratory methods, a method of quick-freezing samples of pulp was developed and applied in a microscope study of conditioning in a rotating drum.

EXPERIMENTAL

Sampling Procedure

The procedure consists of quick-freezing samples of pulp, cleaving the frozen lump to produce cross-sectional surfaces for visual and photographic microscope studies, and measuring the particle size distribution of the oil globules. The sample is frozen on a flat surface cut from a solid copper bar, three quarters of an inch in diameter and six inches long; the bar is insulated except for this flat surface and is cooled in liquid nitrogen before immersion in the pulp. Efforts to detect orientation effects or effects from the sampling procedure were made in two ways. Two and three flat surfaces at right angles to each other were machined on the bars and samples frozen at each surface were examined. They were examined with cleavage surfaces parallel and perpendicular to the plane of the metal. In some cases, a partial "growth ring" effect was observed close in to the metal and these samples were eliminated. Samples were, in general, layers about one centimeter thick, and all observations corroborated the premise that the quick chill immobilized the sample the way it existed in the process of conditioning. Trial of a number of methods of sampling showed that the most satisfactory way was to stop rotation of the drum and immediately insert the sampling device at the selected point in the drum.

The cleavage planes were studied on the cold stage microscope. At magnification of 5X and 8X the depth of focus is usually adequate to give a sharp picture of the entire sample. The remainder of the magnification for measurement was done by projection of the 35 mm. color transparency. The light source was a powerful electronic flash. A number of photomicrographs were taken as the sample was allowed to warm up.

We expected that no significant observations could be made on the flotation gas bubble morphology in the frozen samples, and in general, no bubbles were seen. The rapid cooling would be expected to collapse the bubbles very rapidly since at 85°C they are predominantly steam. Photomicrographs of both surfaces of a cleavage (i.e., the mirror images) agreed quite well, showing that cleavage, in general, went through solidified bitumen particles as well as around them. In neither case could any useful observations on flotation gas bubbles be made. It should be remembered that measurements at other times showed only about one-third as much flotation gas in a conditioning drum sample as in the pulp produced in a stirred reactor at high speed.

Conditions for preparation of the pulp in the drum

These experiments were performed at a time the Field Test Unit was using a feed known as Pit A Lot 25. This sample is a mixture of lean silty oil sand plus some rich oil sand, averaging 9-10 percent oil and 17 percent fines in the mineral ($<$ 325 mesh). For optimum froth yield, it required a dosage of 0.5 - 0.6 pounds of caustic soda per ton. From extensive experience with a wide variety of oil sands, we regard this sample as an excellent representative of feed of somewhat less than average quality. These experiments were all run at 80 percent solids (80 percent mineral plus oil, the remainder water); in general, oil or water does not separate at this composition. Although much of the Field Test Unit operation was at 70 percent solids, no differences in operation have been observed that are pertinent to this study.

Conditioning was done in a drum 2.5 ft. in diameter as already described (4) at 4 tons per hour feed rate. Alkali dosage was varied so that results would parallel the effects observed in the rapidly stirred reactor if possible. Samples were taken at the discharge end, 1, 2, and 4 feet into the drum.

Experiments were made with :

(a) fresh water used in the drum

(b) with water from a settling pond which contained 6 - 7 percent of fine suspended mineral ("pond water").

RESULTS

As already mentioned, no time rate of conditioning was detected in the rapidly stirred reactor; only steady state effects were observed. The microscope observations on frozen samples from the drum served to define a rate process of physical changes in conditioning. Samples of short residence time showed a dirty background of oil-mineral still combined with oil present as a film. There is a relatively small amount of agglomerated oil (as measured by the "percent oil agglomerated", which is simply the percent of the total area occupied by oil globules). With appreciable dirty mineral background, the accurate tracing of the droplets becomes very difficult and the results correspondingly less reliable.

Some representative results are shown in Figures 4-6. The conclusions may be summarized as follows:

1. The following aspects of conditioning as a rate process were observed.
   a. Sand becomes clean and oil agglomerates into globules of increasing size as the rolling motion of the drum applies shear and mixing.
   b. The required residence time for this process under the given conditions is about two minutes. Aside from lump ablation (4), this rate is the slowest of any of the physical or chemical processes we have observed.
2. The effect of caustic dosage, at, below, and above the amount which results in optimum pH, is the same as observed in the rapidly stirred reactor. A broad maximum in globule size is observed at and somewhat above the optimum operating pH of 8.4 (Figure 6).
3. For this particular oil sand, no differences were observed between conditioning with fresh water as compared to pond water containing 6 - 7 percent fine mineral (Figures 4 and 5).
4. The oil sand, Pit A Lot 25, is probably most similar to the Pit C sample of Figure 2. Hence the oil globule size is somewhat larger in the conditioning drum as might be expected under milder mechanical treatment.

Application to Laboratory Conditioning: The Stirred Reactor at Slow Speed

The observations on pulp from the conditioning drum led us to reconsider laboratory procedures for the conditioning step. Of continuing concern was the problem of lower froth yields from small scale laboratory conditioning as compared to the conditioning drum in the Field Test Unit. The fast stirred reactor clearly did better than the Field Test Unit in froth yield, but just as clearly was greatly different in the incorporation of flotation gas. The crux appeared to be how much energy was needed in the process and how it was applied as shear. This approach led to studies with the stirred reactor at slower and slower speeds until a sequence of photomicrographs of frozen samples was obtained which showed essentially the same rate of conditioning and other features as seen in the drum. Pulp produced by hand stirring in a beaker was also characterized by a microscope study.

EXPERIMENTAL

With a choice between freezing samples in the interior of the stirred mixture or making observations at the wall, all further work was done with the frozen samples for two reasons. The primary reason for this choice was that the progress of conditioning, i.e., the earlier stages, can be observed only in the interior, not at the wall.

The high-speed rate of stirring for the 3-inch reactor is 350 r.p.m. Upon slowing this speed to 100 and then 50 r.p.m., we reached a time scale of conditioning which was comparable to the observations in the conditioning drum. The most important parameters were again time and caustic dosage. Most of the comparison experiments were done with a sample identical with the pilot plant feed, Pit A Lot 25.

A number of additional oil sand samples were studied to confirm the observations made with the representative sample.

RESULTS

Time Effect

The time effect in conditioning that can be expressed quantitatively is the growth of the bitumen particle size during the stirring. This effect is shown, for example, in Figure 7. In general, after about 5-10 minutes of stirring at 50 r.p.m., a steady state is reached and no further changes in the particle size distribution are observed. However, just as in the samples from the conditioning drum, the most important results are qualitative observations on the transformation of oil sand to clean mineral plus bitumen globules. The intermediate stages of small lumps of mineral plus oil and thin, yellow oil films can only be described in words. The significance of this intermediate state will be discussed in a following section.

pH Effect

A series of steady state oil globule size distributions are shown in Figure 8 that demonstrate the effect of caustic dosage. Again, as in the conditioning drum, a broad maximum in globule size is observed at and somewhat above the operating caustic dosage.

Selected oil sand samples

Although Pit A Lot 25 is an excellent representative oil sand for experimental work, a number of selected oil sands were also studied in the stirred reactor. Similar behavior was observed in all cases. We were particularly concerned in obtaining further evidence on

(a) a relation between oil globule size and primary froth yield

(b) the effect of viscosity of the pulp on the globule size

In Figure 9, the characteristic behavior of the rich oil sand from the Abasand Quarry in the Horse River Valley near Fort McMurray is shown. This sample gives very large globules of oil. This behavior may be associated with the generally much higher viscosity of the oil from this source, but any attempt at correlations of this kind immediately finds further difficulties in results such as in Figure 10.

Comparison of all the results in Figures 9 and 10 confirms the conclusion that the primary froth yield, in general, is not related to the bitumen particle size. Similarly, viscosity does not have a pre-eminent role in determining the globule size. Care must be taken to control solids contents of the pulp because decreases in globule size parallel to increases in viscosity have been observed when samples have been stirred over long periods and water lost by evaporation.

In general, although a given oil sand sample shows a consistent behavior with time and pH in these studies, wide variations among different oil sands are observed in the bitumen morphology. Many complex factors are operating to result in these differences between samples.

Application to Hand-stirred Conditioning: The Significance of Partial Conditioning

Seitzer has described some of the small scale laboratory methods of conditioning that were used in the early stages of our oil sand studies (1, 5). In an effort to understand the low froth yields from conditioning by hand stirring in a beaker, examination by microscope was applied to pulps produced by this method. Although agglomerated bitumen could be seen on the walls of the beaker during the stirring, all samples frozen in the interior of the mixture showed very little agglomeration and large amounts of dirty mineral and oil films. There were random variations in samples treated identically; no consistent pattern could be observed. Samples, in general, contained large proportions of oil not agglomerated. The significance of these results will be discussed in a following section.

Study of the Relation Between Conditioning Methods and Primary Froth Yields

The results of the preceeding experiments came to have an important bearing on one pre-eminent problem -- the low froth yield in the initially most important procedure, conditioning by hand stirring. Before this aspect is discussed, the results of a number of related experiments will serve to describe the problem and define some of the factors. Part of the difficulty results from the dichotomy of conditioning and flooding. From the viewpoint that the primary froth yield is the single most important characteristic in studying an extraction process, we are immediately confronted by the fact that the two parts, conditioning and flooding-settling, must always be part of the process for producing froth. Early studies seemed to indicate that higher pilot plant froth yield might come from quite vigorous mechanical treatment (including exposure to air) of the pulp in and after flooding. Several devices were fabricated to test this hypothesis on a laboratory scale. The results are shown in Table 2.

The most important behavior that stands out in Table 2 is -- whatever the flooding procedure -- the drum discharge produces a higher froth yield than the laboratory procedure of hand stirring in a beaker. Experiments of this kind on other types of oil sands have shown the same results: hand conditioning in a beaker, except for very high quality oil sands, gives lower froth yields than drum discharge.

TABLE 2

PRIMARY FROTH YIELD WITH VARIOUS FLOODING PROCEDURES
APPLIED TO CONDITIONING DRUM DISCHARGE AND TO HAND-STIRRED PULP

Fraction	Conditioning Drum Discharge Duplicate Runs		Laboratory Conditioning Duplicate Runs	
Flooding: Procedure A				
Percent of total bitumen in:				
Primary Froth	52	51	26	24
Middlings	28	32	54	50
Tailings	20	17	20	26
Percent mineral in:				
Primary Froth, dry basis	8	11	7	7
Flooding: Procedure B				
Percent of total bitumen in:				
Primary Froth	84	91	50	47
Middlings	11	6	29	44
Tailings	5	3	21	9
Percent mineral in:				
Primary Froth, dry basis	10	8	11	11
Flooding: Procedure C				
Percent of total bitumen in:				
Primary Froth	72	83	68	59
Middlings	26	14	23	21
Tailings	2	3	9	20
Percent mineral in:				
Primary Froth, dry basis	9	11	12	16

Flooding Procedures:

A. Simplest batch procedure, placing conditioned tar sands into hot water in mechanically stirred receiver.

B. Feeding conditioned tar sand by means of a plunger into a stirrer-water spray-rotating screen assembly, from which the flooding is completed in a stirrer-equipped beaker.

C. Apparatus as in B plus a two-foot cascade of twelve steps prior to the stirred receiver.

Oil Sand Feed

Pit A bucket wheel product, 12.3 percent bitumen; conditioning done with fresh water and caustic soda to pH 8.4. Pilot plant primary froth yield 84-88%.

At this point the correlation between this poor froth yield and the microscope observations suggested a hypothesis and some experiments to test it. If the unresolved oil-mineral or dirty mineral which is seen in frozen samples of hand-stirred pulp is present because conditioning is incomplete, then the froth yield from short-time stirred reactor experiments should be lower also. The results of these suggested experiments are shown in Table 3. The characteristic low froth yield from hand conditioning is observed again. The low froth yields from "partial conditioning" in the stirred reactor are gratifying; for these cases, microscope studies showed the same incomplete separation of oil and mineral and partial agglomeration as from hand stirring. The hypothesis was confirmed; experiments with other oil sands have given the further corroboration of this hypothesis. In addition, the utility of microscope examination of quick-frozen samples of oil sand was demonstrated.

Additional results were included in Table 3 to show the decrease in froth yield with over-treatment by caustic. Oil sands vary in this response, but in this case, emulsification of the oil occurs during conditioning at pH around 10 and froth yield drops to zero. Again, this emulsification can be observed in a microscope examination of frozen samples of pulp. That insufficient alkali will result in dirty mineral throws a valuable side-light on the usefulness of the surface forces in conditioning. At low pH many of these inherent surfactants have not been "activated", which presumably occurs by salt formation at the proper pH. Hence more mechanical work is required. A number of results have indicated that mechanical energy (stirring of the proper kind) can be traded off for alkali.

TABLE 3

CORRELATION OF CONDITIONING, MORPHOLOGY, AND PRIMARY FROTH YIELD

Oil Sand - Pit A Lot 25, 10% Oil. Flooding: Procedure A, Table 2

Conditioning: Alkali dosage lb./ton	pH	Time min.	Oil Globule Size: Area median size globule[1]	Flooding: % of oil as primary froth
Stirred Reactor				
I. Partial Conditioning[2] - insufficient mechanical working or alkali				
0.1	5.7	2.5	9,6	44
0.1	6.8	5.0	6	27
0.5(3)	7.6	1.2	8	27
0.5(3)	8.2	1.2	11	36
II. Adequate Conditioning				
0.5	8.5	2.0	10,10	54
0.6	8.3	5.0	11	54
0.5	8.4	10.0	23,24	54
1.0	8.9	2.5	9,10,8	60
III. Conditioning at high pH - borderline of emulsification				
1.2	9.2	5.0	11	49
1.4	9.7	5.0	9	33
IV. Conditioning at high pH - emulsification predominant or complete				
1.6	9.8	5.0	Almost completely emulsified	14
2.0	10.2	10.0	Completely emulsified	1
Hand Conditioning				
Partial Conditioning - insufficient mechanical working[3]				
0.5	8.2	10.0	Not measurable	18
0.5	8.2	10.0	Not measurable	26

1. Area median globule size is taken from size distributions such as Figure 2 It is the value of the abscissa where curve crosses the ordinate = 50%. Multiple values are from independent duplicate determinations on the same pulp.
2. Degree of conditioning - as shown by microscope examination of frozen samples Inadequate conditioning is evidenced by the presence of large amounts of oil-mineral still in combination and areas covered by visible yellow oil films.
3. Oil sand preheated to processing temperature 85°C; time indicated is total time of stirring

The froth yields for optimum conditions in Table 3 approach those reported for separation cell froth in the pilot plant. The laboratory values are still somewhat low; experiments have established that with middlings recycle in the laboratory as used in plant scale, the primary froth yields are completely comparable. Because this aspect is more closely related to study of settling, these experiments will be reported in detail in another paper.

DISCUSSION

The goal of understanding conditioning as it is judged by the primary froth yield has been achieved over a scale ranging from small laboratory experiments to a pilot plant at 10 tons per hour

(a) We now understand in terms of requirements for uniform shear and mixing, experiments with a scale difference of 100, 000. As might be expected, the physical factors were most important to understand; from our present understanding of the effect of shear and mixing, we would expect full-scale conditioning drums to improve on the performance of the pilot plant. The chemical factors, such as effect of sodium hydroxide in engendering surface active agents, deflocculating clay, etc., are on a molecular and interfacial level and do not vary appreciably with scale on the gross level.

(b) The microscope studies led to an understanding of the poor results of hand conditioning of oil sands. The information has been fitted into a consistent detailed picture of the extraction process.

(c) With this background of understanding of and control over the conditioning stage and returning to simple flooding procedures, we could proceed to study the next stage of the process. Future papers will describe the results of study of the settling process and further understanding of the processability of widely different oil sand samples.

Certain special topics in the present results have interesting implications which are discussed below.

1. The effect of pH: The action of caustic soda is evidenced in deflocculation of clay, forming salts with potential surfactants, and neutralizing acidic species in general. In the effect of pH on oil globule size are seen mostly secondary results. Figure 11 summarizes the results of the oil globule size measurements over the pH range of interest. The curves serve to point out one difference between the stirred reactor and the conditioning drum. The particle size is larger in the drum and the tendency to emulsification is greater in the stirred reactor because the reactor is characterized by a higher shear stress, at least at the end of the conditioning period.

2. Rate-limiting step: Conditioning in the drum and with suitable laboratory methods includes a rate process of freeing and agglomerating the oil. The drum showed a residence time requirement of about two minutes. Innes and Fear show several runs with good froth yields at residence of 0.5 minutes. However, we are describing the time in terms of immediate flooding as can be done in the laboratory. In the pilot plant the drum discharge went through a vibrating screen, a centrifugal pump, and lengthy runs of pipe before separation was allowed. This process has the slowest rate of any of the chemical or physical processes we observed in the laboratory.

3. Importance of shear properly applied: In this work the microscope observations and primary froth yield from conditioning by hand stirring testify to the need for a certain minimum shear applied in a suitable systematic way. One of the useful working hypotheses is that this extraction process is related to the problem of why bituminous pavement fails.

4. Factors directly involved in the primary froth yield: Primary froth yield, as for example in Table 2, is best understood in terms of a simple working hypothesis which we have found will explain a very wide range of empirical facts concerning the hot water extraction process. If we assume a settling medium in which Stokes law can operate, the buoyancy of the froth determines the primary froth yield. This buoyancy is determined by the flotation gas content and its distribution, the mineral held in the froth, and the density of the medium. As is well known, the pure oil has a density very close to that of water.

Dr. K. A. Clark has stated (5) that procedures such as B and C in Table 2 are inadvisable because the mineral in the froth is increased. This conclusion is based primarily on his studies with Bitumont oil sand, which is of quite high grade. Our work has necessarily been concentrated on average and poorer grade oil sand. The following modification of Dr. Clark's view is offered in order to explain our results with lower oil sands. The factors in oil buoyancy which are listed above are used plus our understanding of conditioning.

A standard flooding procedure allows a certain amount of oil to float as froth, with a given oil content. Agitation of middlings or flooded pulp in the presence of air, in general, increases the amount of flotation gas, and hence, oil with a larger mineral content can float. Primary froth yield increases and mineral in the froth increases. This behavior is observed in the column for "Laboratory Conditioning" in Table 2. This occurs because as the microscope has shown, important quantities of oil still attached to gross amounts of mineral (i.e., large sand grains) are still present. The general concept in mineral flotation is that an oil attached to a mineral provides the situation necessary for attaching the bubble of air for levitation. Perhaps the behavior first noted by Dr. Clark (6), that oil globules in the middlings will film out on the water-air surface, is part of the explanation. Whatever the mechanisms for floating, the inhomogeniety in the conditioning accounts for these results. On the other hand, in the "Drum Discharge" column we see increases in froth yield with no detectable increase in mineral. Here it will be remembered the mixture is very homogeneous and the oil is free of major amounts of mineral. Oil globules have mineral in the form of emulsified (w/o) middlings and at best silt or clay. Quiet flooding (Procedure A) gives a much better froth yield, and the oil remaining in the middlings, resulting from an initially very homogeneous pulp, is on the borderline of floating. When the more energetic procedures of flooding are used, this oil will float readily but will not have any large amount of mineral to carry up. Confirmation of this physical picture is found in sedimentation studies. The "black layer" of oil that settles slowly and forms above the settled sand in a sedimentation from a gentle flooding is generally copious in hand-stirred conditionings. However, it is missing or very small in flooded samples from drum discharge. This unrecovered oil in these cases does not have enough mineral to cause it to sink. Further confirmation of this interpretation is described in another paper.

5. Techniques in microscope work: The use of frozen samples for microscopy proved to be a major help in studies of conditioning. Conditioning on a microscope slide does not give information on the fundamental mass-shear problem. Transferring samples of pulp to the microscope for observation invariably causes changes that vitiate the most valuable observations The quick-frozen samples were invaluable in this study of conditioning, and the technique can be applied to problems in other aspects of recovering oil from the Athabasca deposit.

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