# The Effect of Feed Source in the Hot Water Processing of Utah Tar Sand

**M. Misra and J. D. Miller**

**Abstract**—*The processing strategy for the effective separation of bitumen from low grade ( <10 weight percent bitumen) Utah tar sands by a hot water process differs significantly from that used for the processing of high grade (> 10 weight percent bitumen) Utah tar sands. Excellent separations (coefficient of separation = 0.9) can be achieved for high grade Utah tar sands by intense hot water digestion followed by a modified froth flotation step. Such a technique is ineffective for low grade tar sands.*

*In order to change the characteristics of the low grade tar sand an attempt was made to enrich the bitumen content of the feed to greater than 10 weight percent by recycle of bitumen concentrate to the digestion stage. However, the quality of the separation was not improved under these circumstances. Similarly feed enrichment by heat treating the low grade feed with recycled concentrate prior to digestion did not improve the separation.*

*More successful separations of low grade Utah tar sands were finally achieved by modification of the viscosity of the low grade bitumen with controlled addition of diluent. These results of the hot water separation tests and variations in the quality of separation are interpreted in terms of the physical properties of the bitumen and the surface bonding forces operative at the bitumen-sand interface.*

## Introduction

The dramatic projection for energy demand in the future has accelerated the renewed interest in energy sources other than petroleum; such as coal, oil shale, and tar sands. Although much attention in the US is being directed toward the exploitation of coal and oil shale resources, unfortunately, only a modest research effort has been initiated for the development of tar sand resources. At the University of Utah fundamental and processing studies for the recovery, upgrading and characterization of bitumens from Utah tar sands are in progress. The studies (Oblad, et al., 1976; Sepulveda, Miller, and Oblad, 1977; Sepulveda and Miller, 1978) have identified some of the unique properties of Utah tar sands and in this paper particular attention is focused on the effect of feed source in the hot water processing of Utah tar sands.

---

**M. Misra,** Member SME, is a graduate research assistant and **J. D. Miller,** Member SME is professor of metallurgy, University of Utah, Salt Lake City, UT 84112. SME preprint 79-68, AIME Annual Meeting, New Orleans, La., Feb. 1979. Manuscript Dec. 27, 1978. Discussion of this paper must be submitted, in duplicate, prior to May 30, 1980.

Table 2—Extent of Utah Tar Sand Deposits and Their Average Bitumen Content

| Deposit | Location | In-Place Bitumen (billion bbls) | Average Bitumen Content, wt% |
|---|---|---|---|
| Tar Sand Triangle | SE, Utah | 12.5 – 16.0 | 5.0 |
| P.R. Spring | NE, Utah | 4.0 – 4.5 | 12.2 |
| Sunnyside | NE, Utah | 3.5 – 4.0 | 9.0 |
| Circle Cliffs | SE, Utah | 1.3 | – |
| Hill Creek | NE, Utah | 1.2 | – |
| Asphalt Ridge | NE, Utah | 1.0 | 13.1 |

Table 1—World Reserves of In-Place Bitumen

| Deposit | In-Place Bitumen (billion bbls) |
|---|---|
| Canadian tar sands (Athabasca) | 900 |
| Utah tar sands | 25 |
| Other US deposits (principally California, Kentucky, and New Mexico) | 3 |
| Venezuela | 700 |
| Africa | 2 |
| Europe | 3 |

*Source: Walters (1974)*

Tar sand is typically dark brown to black in appearance, a consolidated mixture of bitumen and sand. The existence of tar sand and it's utilization by mankind goes back to the beginning of recorded history. In ancient time it was called "pitch" and used extensively for the construction of boats and as a cement for building material. In Europe during the Napoleonic era, rock asphalt was used as a paving material for road construction. Late in the 18th century the Cree Indians, from the northeastern region of the province of Alberta, recognized the utility of this sticky material and used the bitumen to waterproof their canoes (Camp, 1974). In 1890 the Canadian government began to take an interest in these Athabasca tar sand deposits and supported the study of these reserves. Since then, the untiring perseverance and ingenuity of great entrepreneurs finally resulted in the development of a full scale plant by Great Canadian Oil Sand Ltd. (GCOS) in 1967, which currently produces 9500 $m^3/d$ (60,000 bbls per continuous day-bpcd) of synthetic crude (McConville, 1975).

Tar sand deposits are found throughout the world with the exception of Australia and Antarctica. The location and size of the larger deposits are summarized in Table 1 (Walters, 1974). About 95% of the mapped tar sand resources of the US are located in Utah amounting to 4 billion $m^3$ (25 billion bbls) of in-place bitumen (Ritzma, 1978). Of the 51 deposits along the eastern side of the state, six deposits are of sufficient size to be of commercial significance. The amount of in-place bitumen for each of these six deposits is given in Table 2 and their location can be seen from the map presented in Fig. 1. Also, the estimated average bitumen content of some of these deposits is presented in Table 2. These estimations emphasize the fact that the bitumen content varies from deposit to deposit and significant variation is found even within a given deposit. Although many occurrences of bitumen saturation, up to 17 weight percent, can be found in the Utah tar sand deposits (e.g., Asphalt Ridge and P.R. Spring), current information included in Table 2 indicates that the overall average bitumen content for Utah reserves may be from 5% to 10% by weight.

## Low Temperature Separation Technology

There are several low temperature processing strategies that can be used for the recovery of bitumen from mined tar sand. These separation methods are:

- Solvent — only
- Solvent — assisted water
- Water — only

Solvent-only processes were probably the first methods used to remove bitumen from sand and the principle is fairly straightforward. Virtually any hydrocarbon solvent will remove bitumen from oil-impregnated rock (Cottrell, 1974). Although simple in principle, solvent techniques present several disadvantages. First, a large amount of solvent is required to completely dissolve the bitumen and the large volume of recycled solvent necessitates the construction of larger reactors. Secondly, the significant amount of solvent loss due to evaporation and adsorption on the sand has an adverse effect on the cost of the operation. These features make the solvent process strategy unattractive. As a result, commercial utilization of this process has not been successful.

Solvent-assisted water processes along with solvent processes have received most of the attention by investigators as recently reviewed in the literature (Lowe, 1975). These processes usually have features similar to the hot water process. Again the economics of this process depend on the amount of solvent used and anticipated solvent loss. Most processes of this type have only been applied on a laboratory scale. One exception is the process developed by Mines Branch of the Canadian Department of Mines and Technical Surveys which uses a combination of cold water and solvent (Djingheuzian, 1951).

Numerous water-only processes have been proposed. The "Sand Reduction Process" developed by Imperial Oil Enterprises Ltd. uses cold water only (Bichard, et al., 1974). A novel oleophilic sieve process developed by Kruyer (1978) is claimed to be more efficient in terms of bitumen recovery and conserves on water and energy. The original hot water process was described for the Athabasca tar sands by Clark (1923) and modified thereafter by Clark and Pasternack, 1932; Clark, 1944; and In-

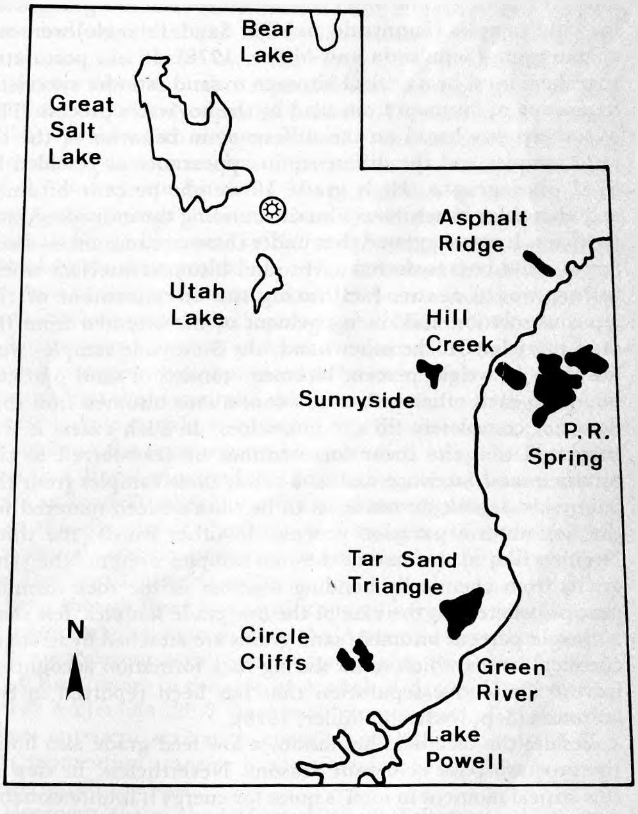

Fig. 1—Major tar sand depositis in the state of Utah.

Table 3—Existing and Proposed Commercial Operations

| Project | Capacity bpcd | Start-up Date |
|---|---|---|
| GCOS | 60,000 | 1967 |
| Syncrude | 129,000 | 1978 |
| Shell | 100,000 | 1980 |
| Petorfina | 122,000 | 1982 |

*Source: McConville (1975); Porteous (1978)*

nes and Fear, 1967). Currently, this is being used on a commercial scale by GCOS. The basic concept of the process is described by Camp (1974). The mined tar sand is placed in a conventional tumbling mill, into which steam, water and a caustic wetting agent are added. The resulting strong hydration forces acting at the surface of the sand particles give rise to the displacement of the bitumen into the aqueous phase. Once the bitumen has been displaced, it is recovered in a settler for phase separation.

Research and development on tar sand processing has been limited largely to the Canadian tar sand deposits and a number of companies plan to build processing plants. The existing and proposed commercial ventures along with their plant size and start-up date are presented in Table 3.

## Hot Water Processing of Utah Tar Sand

Although similar in principle, the separation strategies in the processing of Athabasca and Utah tar sands are distinctly different (Sepulveda and Miller, 1978; US Patent, 1978). Because of the higher viscosity of Utah tar sand samples a high shear, stirred tank reactor is used for digestion of Utah tar sand samples. In addition a wetting agent is used to assist the phase disengagement process. While excellent separations were obtained for two high grade Utah tar sand samples (Asphalt Ridge and P.R. Spring), hot water separation tests of low grade Utah tar sand samples (Sunnyside and Tar Sand Triangle) were not encouraging (Sepulveda and Miller, 1978). It was postulated that there must be a critical bitumen to sand ratio for successful separation of bitumen from sand by the hot water process. This hypothesis was based on the difference in behavior of the tar sand samples and the difference in appearance as revealed by SEM photographs. High grade 10 weight percent bitumen exhibit a thick bituminous film surrounding the individual sand particles. It was suggested that under these circumstances, shear forces could be transferred to the sand-bitumen interface where failure would occur, facilitating the advancement of the aqueous solution and disengagement of the bitumen from the sand particle. On the other hand, the Sunnyside sample, with less than 10 weight percent bitumen, consists of sand particles bound to each other with a non-continuous bitumen film that does not completely fill the interstices. In such cases, it was suggested that the shear force cannot be transferred to the bitumen-sand interface and as a result these samples from the Sunnyside deposit do not seem to be suitable feed material for the hot water separation process. In other words, the thick bitumen film in the case of the rich samples prevents the sand grains from chemically bonding together in the rock forming process, whereas in the case of the low grade samples, less than 10 weight percent bitumen, sand grains are attached by stronger chemical bonds which result during rock formation accounting for the ineffective separation that has been reported in the literature (Sepulveda and Miller, 1978).

Besides the technical limitation, a low feed grade also limits the processing for economic reasons. Nevertheless, in view of this critical moment in man's quest for energy it is unreasonable to limit the cutoff feed grade for hot water processing at 10 weight percent bitumen. Moreover, as can be seen from Table 2, it is estimated that more than 76% of the total tar sand deposits in Utah contain less than 10 weight percent bitumen. Therefore, these large deposits are certainly a significant resource for several generations to come. In order to fully develop the potential of these low grade deposits a systematic study of the physical and chemical properties of a low grade Sunnyside tar sand sample was initiated with the explicit objective to establish an effective processing strategy for low grade feed material and delineate the differences between the nature of low grade deposits and the nature of previously studied (Sepulveda and Miller, 1978) samples from high grade deposits.

## Experimental Procedure

Tar sand samples from four different Utah deposits: Sunnyside, Asphalt Ridge, P.R. Spring, and Tar Sand Triangle were used in this investigation. With the exception of the P.R. Spring sample, all others were surface samples. The majority of the experiments focused on the behavior of the Sunnyside sample. Unlike the preparation of the Asphalt Ridge sample, size reduction of the Sunnyside sample was accomplished by conventional crushing and grinding techniques after freezing in liquid nitrogen. The Tar Sand Triangle sample, with less than 6 weight percent bitumen was easily ground to -4 mesh in conventional size reduction equipment without cryogenic treatment. High grade samples with greater than 10 weight percent bitumen (Asphalt Ridge and P.R. Spring) can only be reduced in size to a limited extent, -9.5 mm (-3/8 in.) by extrusion with a modified meat grinder.

After size reduction all tar sand samples were kept in airtight polyethylene bags until used in order to eliminate possible oxidation effects on the subsequent separation experiments.

**Hot Water Separation Test**—As discussed earlier, due to the high viscosity of Utah tar sands, a high shear, stirred tank reactor was selected for digestion of the tar sand sample and wetting agents were added to assist the phase displacement-disengagement process. Unless otherwise mentioned, sodium carbonate

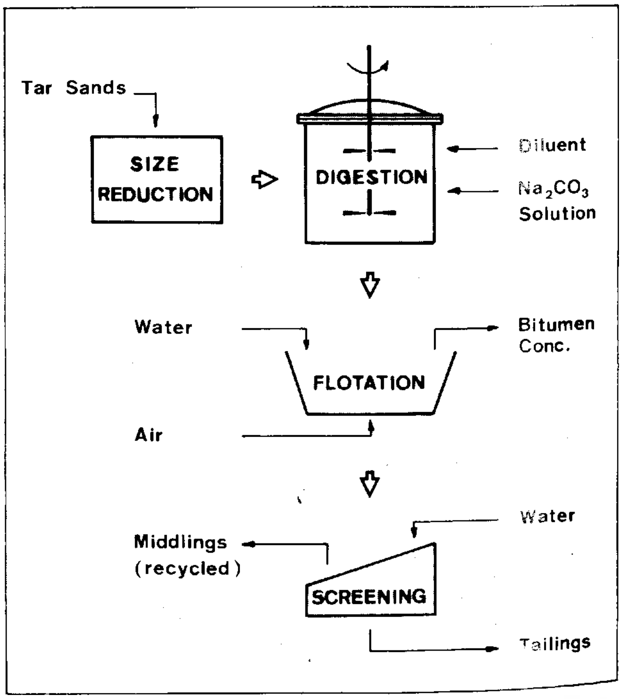

Fig. 2—Modified hot water process for the separation of bitumen from low grade Utah tar sands.

was used as the wetting agent. The one gallon stirred tank reactor was obtained from Bench Scale Equipment Co. Essential parts of the reactor are an impeller, (two pitched blade turbines, 102 mm or 4 in.-diam), torquemeter, reflux condenser, temperature controller, heating system, SCR speed controller and tachometer. Inside the reactor the feed material was contacted with a mixture of the hot aqueous solution of soda ash and diluent and stirred at constant temperature (95°C) and speed (750 rpm) for a specified digestion time. Diluents were added at a specified volume ratio based on the feed bitumen content.

At the end of digestion, ideally, the bitumen has been displaced from the sand and can be separated from the sand by a modified flotation technique. Constant air flow rate and moderate stirring speed were maintained during flotation. Neither frother nor collector was added to the flotation cell. During flotation relatively large lumps of nonflotable sand-bitumen aggregates (middlings) were found with the tailing. This middling was recovered from the tailing by screening at 20 mesh. The grade of the middling was sufficiently high to be recycled. A schematic representation of the processing strategy used in the hot water separation technique is presented in Fig. 2.

**Analytical Technique**—Representative samples of feed, concentrate, middling and tailing obtained during experimentation were analyzed to establish their composition with respect to bitumen, sand and water, using a set of Dean and Stark tube assemblies. The assemblies were set up in accordance with the procedure described by the USBM. The specially designed flask and operational details have been described by previous investigators (Sepulveda and Miller, 1978).

**Bitumen Viscosity Measurements**—The bitumen viscosity of the tar sand samples is of great importance from a processing standpoint. Samples of pure bitumen were prepared from feed sources by using a carefully controlled procedure, because the method of preparation has a significant effect on viscosity measurements. In this regard, bitumen from Asphalt Ridge, P.R. Spring, Sunnyside, and Tar Sand Triangle samples were prepared under identical conditions. Preparation of bitumen for viscosity experiments was effected by Soxhlet extraction using benzene as solvent and a glass extraction thimble of porosity "A" to hold the tar sand sample. After complete extraction, the benzene extract was filtered slowly using a 4.0-5.4 μm fritted glass filter. Removal of the solvent from the bitumen was effected by flash distillation using a rotary evaporator at 90°C and 533.3 Pa (4 mm Hg) pressure.

The viscosities of the four different bitumens were determined with a rotational viscometer, Rotovisco. The sample was introduced in the gap between a stationary cylindrical cup and a coaxial rotating cylinder which was turned at a specified angular velocity. The instrument is equipped with an electrical torsion dynamometer to measure the torque required to maintain the specified angular velocity. From these measurements the "flow curve" for the fluid can be established. The viscosity is then calculated from the slope of the flow curve at a given rate of shear.

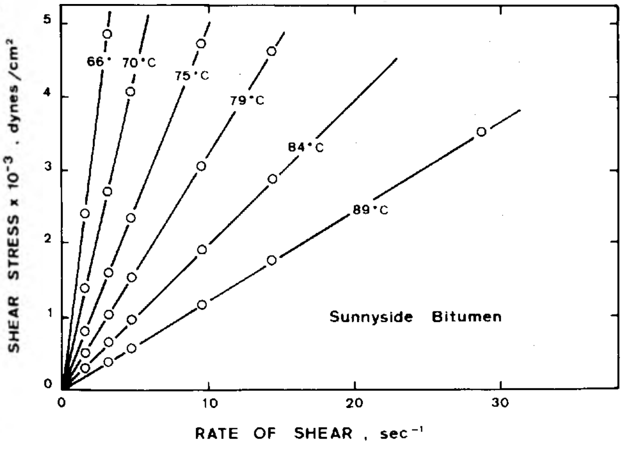

Fig. 3—Flow curves for Sunnyside bitumen, at various temperatures.

**Molecular Weight Determination**—Average molecular weights of the same bitumen sample that had been used for viscosity measurements were determined by Vapor Pressure Osmometry in benzene using the Model 117 Molecular Weight Apparatus manufactured by Corona Electric Co., Japan.

**Particle Size Analysis**—From a processing standpoint, particle size distribution is an important property of the tar sand feed. The sand size distributions for Utah tar sand samples were determined by conventional wet/dry sieving techniques in the size range of 590 μm to 38 μm.

## Results and Discussion

**Tar Sand Properties**—Some of the more important tar sand properties, with respect to the development and characterization of hot water processing technology, are the bitumen viscosity, bitumen molecular weight and the particle size distribution of the sand.

*Bitumen Viscosity.* The rheological properties of a fluid are well described by the relationship between the shear stress applied to a fluid element and the rate at which the fluid element is deformed under the applied stress. The above relationship referred to as a flow curve, or rheology diagram, is a distinct characteristic of a fluid and is useful to describe the behavior of a fluid. This is particularly true in the case of bitumens because their complex structure may result in unpredictable rheological properties.

Flow curves for the Sunnyside bitumen sample are presented in Fig. 3 for various temperatures. The linear response in each case indicates that the Sunnyside bitumen sample exhibits Newtonian behavior over the range of applied shear rates. Bitumens from Asphalt Ridge and P.R. Spring also have been shown to exhibit Newtonian behavior (Sepulveda and Miller, 1978), results which were confirmed in this present study. From a practical standpoint, it is important to note that the viscosity of the Sunnyside bitumen is approximately one order of magnitude greater than the viscosities of the P.R. Spring and Asphalt Ridge bitumens in the temperature range studied, as shown by the data presented in Fig. 4. Indeed this difference in viscosity undoubtedly accounts for the poor separation that had been reported previously (Sepulveda and Miller, 1978).

Furthermore, analysis of bitumen from a Tar Sand Triangle outcrop sample which appeared to be extensively weathered indicated that the viscosity of this bitumen was even higher than the Sunnyside sample, so much so that measurements could not be made in the temperature range studied using the Rotovisco apparatus. It was estimated that the viscosity of the bitumen from the Tar Sand Triangle outcrop sample was significantly greater than the viscosity of the Sunnyside sample. This observation may explain why no separation was achieved in the processing of the Tar Sand Triangle sample.

Even though there is a significant difference in viscosity, all bitumen samples seem to obey the Arrhenius-type relationship with an apparent activation energy ranging from 98.8 kJ/mole (23.6 Kcal/mole) in the case of Asphalt Ridge and P.R. Spring to 102.6 kJ/mole (25.7 Kcal/mole) in the case of Sunnyside. These apparent activation energies are indicative of the fact that momentum transfer is accompanied by significant structural transformations.

Moreover, the method of preparation of the bitumen sample has a tremendous effect on its apparent viscosity. The viscosity

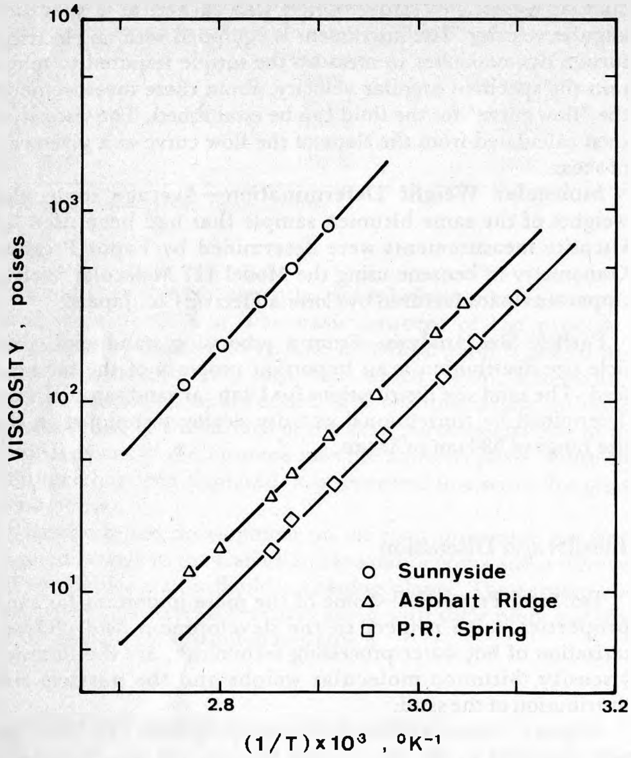

Fig. 4—Arrhenius-type plot illustrating the effect of temperature on viscosity for bitumen from various Utah tar sand deposits.

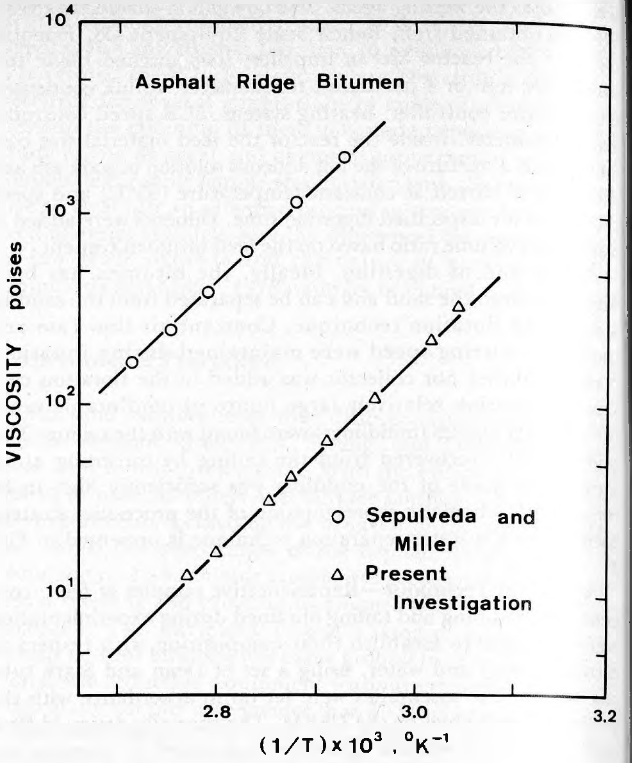

Fig. 5—Arrhenius-type plot illustrating the effect of the bitumen preparation technique on the measured viscosity of Asphalt Ridge bitumen.

of Asphalt Ridge bitumen previously reported (Sepulveda and Miller, 1978) is contrasted with the viscosity of the bitumen from the Asphalt Ridge sample as determined during this investigation in Fig. 5. The significant difference between these viscosity measurements is due to the difference in the procedure used to prepare the bitumen sample. The previous Asphalt Ridge bitumen sample was not prepared under the controlled conditions that were used for bitumen sample preparation in this study. In fact, the high viscosity of the Asphalt Ridge sample reported previously reflects the more severe thermal conditions and atmospheric exposure experienced during preparation.

Although these viscosities may not be the true viscosities of the virgin bitumen because of possible entrainment of benzene and/or loss of light ends, they are at the least representative of the bitumen character on a relative scale and are of significant value for the analysis of the results from hot water separation experiments.

*Molecular Weight.* With the exception of the P.R. Spring bitumen, the bitumen viscosities presented in Fig. 4 can be correlated with the number average molecular weights of those samples presented in Table 4. The highly viscous Tar Sand Triangle sample exhibits the highest molecular weight. The difficulty in achieving effective hot water separations for the Tar Sand Triangle and Sunnyside samples together with the higher molecular weights and higher viscosities of the respective bitumens suggest a different state of molecular aggregation for these samples with stronger intermolecular bonds than would be found for the Asphalt Ridge sample which can be easily separated and whose bitumen has a viscosity which is significantly less than viscosities of the Tar Sand Triangle and Sunnyside bitumens.

It is premature at this point to speculate whether bitumens from different deposits exhibit differences with respect to a specific adsorption potential at the sand surface. Bonding characteristics at the sand-bitumen interface are currently under investigation for the Sunnyside and Asphalt Ridge systems. It is anticipated that the results from this phase of the research will produce a better description of the nature of the bond and interfacial activity of the respective bitumens. Nevertheless, other bulk phase properties such as tar sand composition, bitumen viscosity, bitumen molecular weight and SEM photographs (Sepulveda and Miller, 1978) of the tar sand samples already demonstrate significant differences between the various Utah tar sand deposits.

*Sand Analysis.* Particle size distribution of the sand from various tar sand samples is an important characteristic in the analysis of the hot water separation process. In general, it would be expected that coarser sand distribution in the feed would be most desirable. Indeed, size classification has been shown to occur in the processing of the Asphalt Ridge sample (Sepulveda and Miller, 1978). The particle size distribution of four Utah tar sands are presented in Fig. 6. As can be seen, the Sunnyside sample contains more fine sand than any of the other tar sand samples. Assuming a critical size of 100 μm it can be seen that 7.7% of the sand in the Asphalt Ridge is finer than 100 μm. Whereas, in the case of the Sunnyside sample, 25% of the sand is finer than 100 μm. The greater amount of fines in the sample feed stock would be expected to have a detrimental influence on the bitumen-sand separation process.

Table 4—Average Molecular Weight of Utah Bitumens

| Bitumen Source | Average Molecular Weight |
|---|---|
| Asphalt Ridge | 763.2 |
| Sunnyside | 891.3 |
| P.R. Spring | 938.9 |
| T.S. Triangle | 1222.1 |

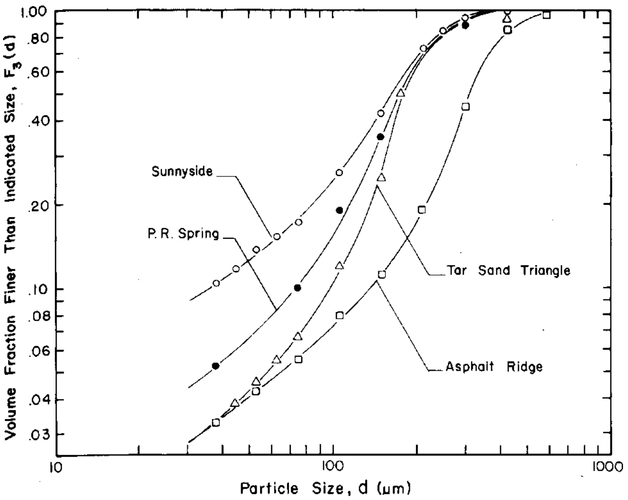

Fig. 6—Particle size distribution of sand from four different Utah tar sand deposits. Sunnyside, P.R. Spring, Tar Sand Triangle and Asphalt Ridge.

The sand in the Sunnyside sample contains α-quartz along with calcite, dolomite and clay as determined by XRD analysis. The amount of clay present has not yet been established quantitatively. The sand in the Asphalt Ridge sample, however, contains only α-quartz and no clay or carbonate mineral could be detected by XRD. The presence of the clay in the Sunnyside sample undoubtedly contributes to the finer size distribution of the sand.

**Hot Water Process**—Because of the significant difference in the physical and chemical properties of the low grade Sunnyside sample, as compared to the high grade Asphalt Ridge sample, effective separations of the Sunnyside sample were not realized (Sepulveda and Miller, 1978). Whereas a coefficient of separation of 0.9 could be achieved for the Asphalt Ridge sample, a coefficient of separation of only 0.55 could be achieved with the lower grade Sunnyside sample. This poor separation was attributed to discontinuity of the bitumen and subsequent failure within the bitumen phase rather than at the bitumen-sand interface (Sepulveda and Miller, 1978).

In this regard an attempt was made to enrich the bitumen content of the feed to greater than 10 weight percent by recycle of the bitumen concentrate to the digestion stage thus promoting bitumen phase continuity. However, the quality of the separation was not improved under these circumstances. Similarly feed enrichment accompanied by heat treatment of the low grade feed with recycled concentrate in a furnace at 196°C prior to digestion did not improve the separation.

Results from this study have shown the viscosity of the Sunnyside bitumen is one order of magnitude greater than the viscosity of the Asphalt Ridge bitumen and suggest the poor separation of the Sunnyside sample may be related simply to the viscosity of the bitumen rather than to bitumen phase continuity as previously suggested (Sepulveda and Miller, 1978). If the high viscosity of the Sunnyside bitumen is responsible for the inferior quality of separation then it should be possible to improve the separation by reducing the bitumen viscosity with the addition of a diluent. In this regard, a controlled solvent-assisted hot water process was developed. Presumably, the diluent dissolves into the bitumen and reduces its viscosity, such that with the aide of a high shear, stirred tank reactor phase disengagement should be improved.

*Effect of Soda Ash.* It was established by Sepulveda and Miller (1978) that wetting agent addition is a critical factor for the success of the hot water process. Therefore, the coefficient of separation for the Sunnyside sample was determined as a function of sodium carbonate concentration with and without diluent addition as shown in Fig. 7. The coefficient of separation provides a unique "one parameter" description of the efficiency, or quality, of the separation process. It is defined as the fraction of the feed material which undergoes a perfect separation (Schulz, 1970). The coefficient of separation can be expressed in terms of recovery and for a binary system can be shown to be equal to the difference between the recovery of the bitumen and the recovery of sand in the same concentrate. It can be realized from Fig. 7 that diluent addition has increased the coefficient of separation significantly and that the best separation can be achieved at about 0.2 M sodium carbonate addition for a diluent to bitumen volume ratio of 0.2. Low wetting agent additions $< 0.1$ $\underline{M}$, produce a sticky concentrate and much of the bitumen is lost in the tailing product. At higher additions of sodium carbonate in the presence of diluent, the effectiveness of the separation is impaired apparently due to a tendency of the system towards emulsification.

In these experiments with the addition of a diluent, about 95% of the bitumen in the feed material was recovered in the concentrate and 72% of the sand was rejected in the tailings product containing less than 0.5% bitumen. However, from 5% to 10% of the feed was recovered as aggregates in a middling product which contained 10.5% bitumen. Typical results for hot water processing of a Sunnyside sample are presented in Table 5. Coefficients of separation are calculated based on a combined concentrate-middling product.

In all of the above experiments 30 minutes digestion time was used. In view of the middling stream produced, it was thought that a longer digestion time might minimize the production of this intermediate product. In order to test this hypothesis, a series of experiments at a diluent/bitumen volume ratio 0.2

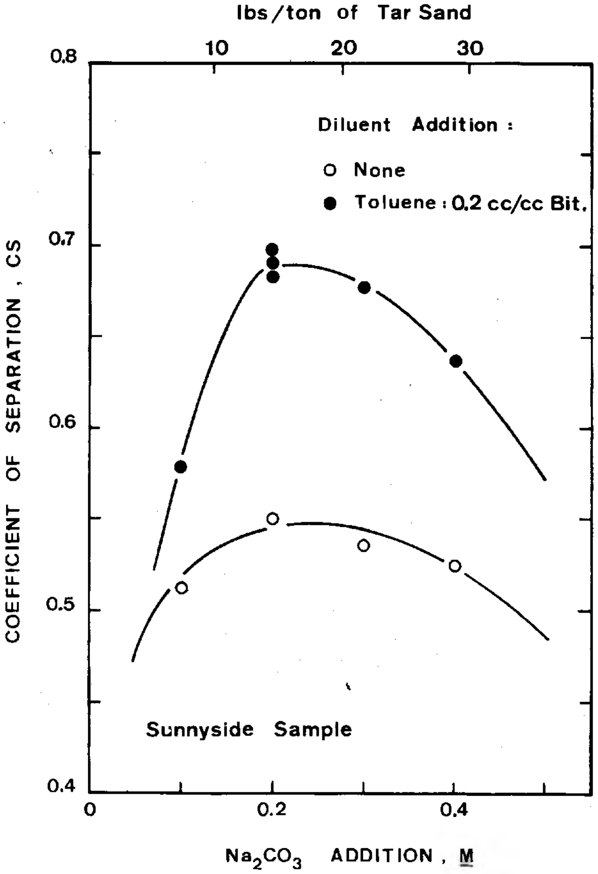

Fig. 7—Quality of separation as a function of sodium carbonate concentration for the Sunnyside sample at a diluent to bitumen volume ratio of 0.2.

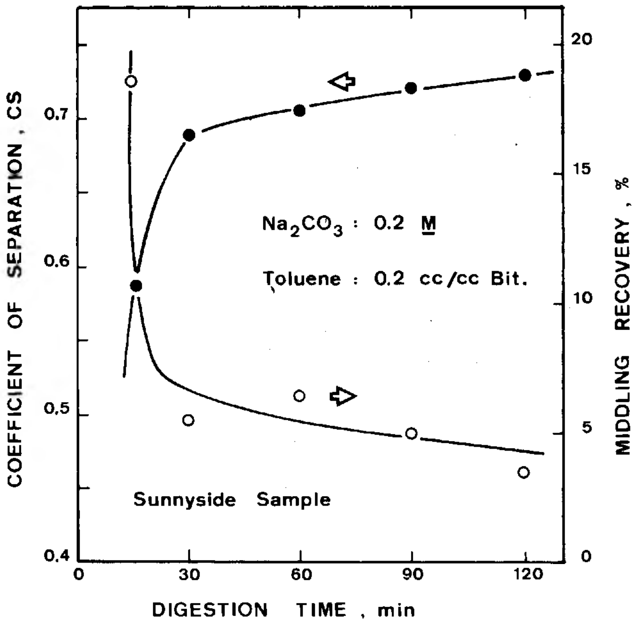

Fig. 8—Quality of separation and recovery of middling as a function of digestion time at 0.2 *M* sodium carbonate and a diluent to bitumen volume ratio of 0.2.

were performed for longer digestion times. Experimental results presented in Fig. 8, indicate that increased digestion beyond 30 minutes causes the quality of separation (coefficient of separation) to increase slightly. In addition, the recovery of the middling stream shows a corresponding decrease. It appears that a 30 minute digestion time is reasonable. This middling stream which was not produced in the hot water processing of the Asphalt Ridge sample, will have to be redigested in order to effect bitumen recovery.

*Effect of Diluent.* Preliminary results with controlled addition of diluents, in which separation coefficients greater than 0.69 were obtained, have encouraged further investigation in this area. In this regard, the effect of different diluent types on the separation coefficient was determined as a function of diluent to bitumen volume ratio. As can be observed from Fig. 9 toluene and No. 1 fuel oil are most effective when compared to the response obtained using kerosene. Tetraline, a hydrogen donor solvent, is not nearly as effective as the other diluents.

The diluent to bitumen volume ratio is critical from the processing standpoint. Even a low diluent to bitumen volume ratio of 0.01 has a significant effect on the separation coefficient. However, a better separation is achieved at a higher diluent to bitumen volume ratio such as 0.2. Excessive diluent additions (volume ratios above 0.40) have a detrimental effect on the separation due to a tendency of the system to emulsify. Further research in this area of diluent addition is comtemplated as a part of the future research program.

Product Characterization—Analysis of the products from the separation experiments provides further insight regarding the nature of the phase disengagement-displacement process.

*Particle Size Analysis.* The products in the hot water separation of the Sunnyside sample differ significantly from those that had been obtained in the hot water processing of the higher grade feed stocks. In the processing of the high grade Asphalt Ridge and P.R. Spring samples, essentially no middling product was obtained and the sand content of both the concentrate and the tailing consisted of free non-aggregated particles. Further, it was shown that sand was partitioned between the concentrate and tailing on the basis of size, i.e., size classification occurred during the process sequence with the fine sand being preferentially recovered in the concentrate and the coarse sand being rejected in the tailing as shown in Fig. 10.

The character of these products should be contrasted to the products obtained in the hot water processing of the lower grade, Sunnyside sample at 0.3 M sodium carbonate addition and for a diluent to bitumen volume ratio of 0.2. A significant middling stream (~ 10% of the feed) is produced consisting largely of aggregates which remain undigested during the processing sequence. The interparticle strength is such that the aggregates persist even after extraction of bitumen during Dean-Stark analysis. In addition, the aggregates can be identified in the feed, a phenomenon that was rarely observed in the Asphalt Ridge and P.R. Spring feed stocks. These aggregates have been separated from the feed and XRD analysis clearly shows the presence of calcite and dolomite which enhance the aggregate strength by acting as an interparticle cementing agent.

These results account for the lack of size classification during hot water processing of the Sunnyside sample. As can be seen in Fig. 11, the sand from the middling product has the coarsest size distribution, followed by the sand from the concentrate, and finally the sand from the tailing which has the finest size distribution and consists of essentially free, non aggregated particles, whereas the sand from the middling product consists almost exclusively of aggregates with very few free particles. As might be expected, no carbonate cementing compounds could be detected in the tailing product by XRD analysis.

These apparently anomalous results, for which no size classification is observed, indicate that sand is not transported to the concentrate by mechanical entrapment as appears to be the case for the Asphalt Ridge and P.R. Spring samples, but rather some undigested aggregates, of a relatively coarse size, are trans-

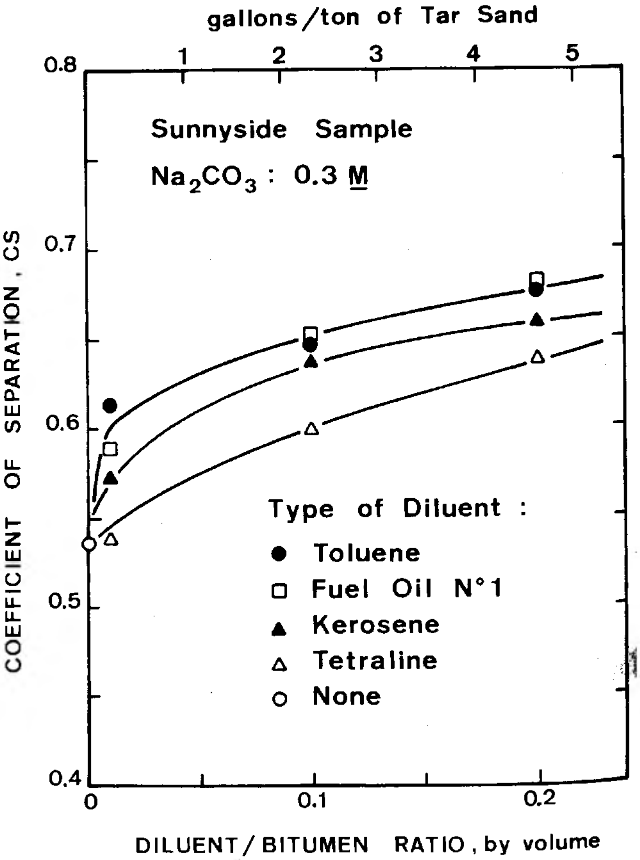

Fig. 9—Quality of separation for the Sunnyside sample as a function of diluent to bitumen volume ratio for different types of diluent at 0.3 *M* sodium carbonate.

ported to the concentrate due to their partial hydrophobicity. This hydrophobic character arises from the residual bitumen which together with cementing agents bond the sand particles together and account for the aggregate's strength.

*Bitumen Viscosity.* It is clear from the results of hot water processing of the Sunnyside sample that diluent addition is necessary in order to achieve a better separation. The addition of diluent seems to be an appropriate strategy because the viscosity of the Sunnyside bitumen was found to be significantly higher than the viscosities of the Asphalt Ridge and P.R. Spring samples as shown in Fig. 4. Bitumen viscosity measurements of the concentrate and middling streams from the solvent-assisted hot water separation of the Sunnyside sample support the previous discussion regarding the presence and behavior of aggregates in the Sunnyside feed stock. Note in Fig. 12 that the bitumen associated with the middling product has the same viscosity as the bitumen from the feed, indicating that the diluent has had no effect on the viscosity of the bitumen in the middling stream, a result which is consistent with the observation that the middling consists of undigested, (apparently impervious) tightly bonded aggregates. On the other hand, the bitumen associated with the concentrate product apparently has dissolved the diluent and as a result is an order of magnitude less viscous than either the middling or the feed bitumen. Interestingly, the viscosity of the bitumen from the Sunnyside concentrate shown in Fig. 12 has a viscosity profile essentially the same as the viscosity profile for the Asphalt Ridge and P.R. Spring feed stocks, both of which can be processed without diluent addition.

These results demonstrate that for high viscosity feed stocks diluent additions are necessary to allow for effective digestion. Further, at least in the case of the high viscosity Sunnyside sample, the tenacious interparticle bonding results in the stabilization of non-reactive aggregates which report to the concentrate and middling product accounting for the less efficient separation relative to that which had been achieved with the Asphalt Ridge and P.R. Spring samples.

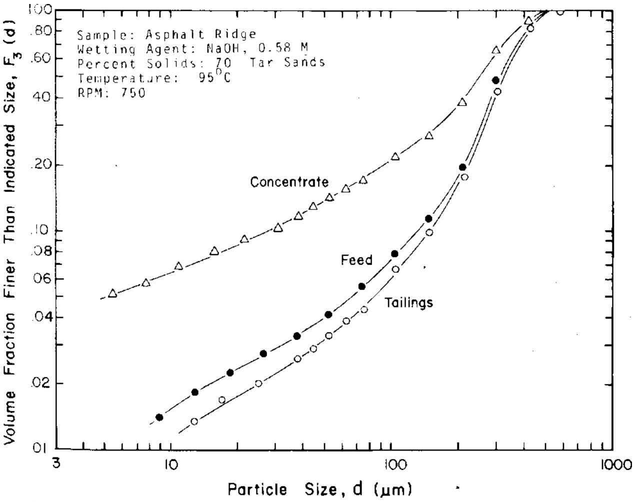

Fig. 10—Particle size distribution of the sand in the feed and products from a typical hot water separation of the Asphalt Ridge sample.

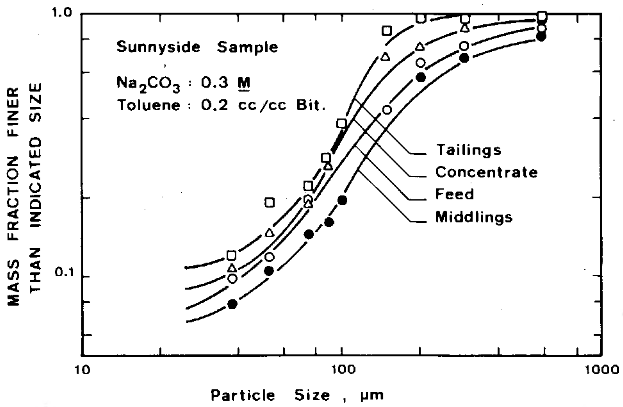

Fig. 11—Particle size distribution of the sand in the feed and products from a typical hot water separation of the Sunnyside sample.

## Summary and Conclusions

Consideration of previous results and hypotheses (Sepulveda and Miller, 1978) regarding the phenomenological description of the phase disengagement-displacement process together with the results of this current study indicate that the criteria for an effective separation remain the same for the hot water processing of Utah tar sands. These criteria are:

- High shear force field
- Appropriate level of wetting agent addition, and
- High temperature digestion

However, it appears the previous hypothesis (Sepulveda and Miller, 1978) that bitumen phase continuity (> 10 weight percent bitumen) is required for effective hot water separation must be dismissed. In view of the results reported in this investigation it would seem that the correlation between bitumen content of the feed and the effectiveness of the separation is due to two major factors:

- Viscosity of the bitumen, and
- Interfacial bonding between sand particles

For low grade tar sands such as the Sunnyside sample, the bitumen viscosity is at least an order of magnitude greater than the bitumen viscosity for high grade tar sands such as Asphalt Ridge. In addition, the low grade tar sands exhibit strong interparticle bonding accounting for the presence of aggregates in the feed which maintain their integrity during digestion and subsequent processing. These factors explain the inferior separations obtained with the Sunnyside sample while much better separations are obtained with high grade samples. At this point it would appear that the bitumen viscosity and the strength of the interparticle bonding may be related to feed grade via the genesis of the deposit. The insufficient bitumen content of the low grade material results in the formation of strong interparticle bonds during the rock forming process, whereas high grade material has sufficient bitumen between the sand particles to prevent this strong interparticle bonding and as a result facilitates the phase disengagement-displacement process in the hot water separation of high grade samples.

The conclusion regarding the importance of bitumen viscosity is based on the necessity of adding diluents to the Sunnyside feed for effective separations and the fact that the bitumen viscosities of the separation products (concentrate and middling) reflect a marked difference between that bitumen which had been separated into the concentrate successfully and that bitumen which was not separated successfully and was recovered in the middling. These results are contrasted to the nature of the bitumen of the higher grade tar sands (Asphalt Ridge and P. R. Spring) the viscosity of which is equivalent to the viscosity of the diluted Sunnyside bitumen recovered in the concentrate.

The importance of interfacial bonding between sand particles has been demonstrated in the case of the low grade Sunnyside sample by the identification of relatively strong aggregates which report to the middling product and persist both in the feed and in the middling even after bitumen removal during

Table 5—Hot Water Separation of Sunnyside Tar Sand

**Experimental Conditions:**

**Digestion:** Feed source: Sunnyside
Wetting agent: $Na_2CO_3$
Temperature; 95°C
Diluent, (Toulene): Diluent/Bitumen volume ratio of .2
Percent solids: 73.5% by weight tar sands
Digestion time: 30 minutes
$Na_2CO_3$ concentration: 0.2 Mole/liter
Feed size: Minus 4 mesh
Agitation: 750 rpm

**Flotation:** Cell design: Cylindrical
Percent solids: 20 weight percent tar sands
Agitation: 900 rpm
Temperature: 15°C
Air flowrate: 3000 (cc/min)

**Calculated Mass Balance:**

| | Weight (%) | Grade (%) | | Recovery (%) | |
|---|---|---|---|---|---|
| | | Bitumen | Sand | Bitumen | Sand |
| Concentrate | 28.49 | 30.41 | 69.58 | 90.94 | 21.91 |
| Tail | 66.08 | 0.45 | 99.55 | 3.11 | 72.71 |
| Middling | 5.43 | 10.44 | 89.56 | 5.95 | 5.38 |
| Feed | 100.00 | 9.52 | 90.48 | 100.00 | 100.00 |

Coefficient of Separation = 0.69

analysis by the Dean-Stark technique. This phenomenon is revealed in the particle size analysis of the products of the hot water separation experiments. Further, it appears that some of the bonding strength of the aggregates arises due to cementation of the sand particles by calcareous compounds such as calcite and dolomite which were identified by XRD. Aggregates such as these were only rarely encountered in the processing of the Asphalt Ridge sample.

Even with this understanding and making the necessary provisions to improve the process, the quality of separation for the Sunnyside sample is still inferior to that which can be achieved for the Asphalt Ridge sample.

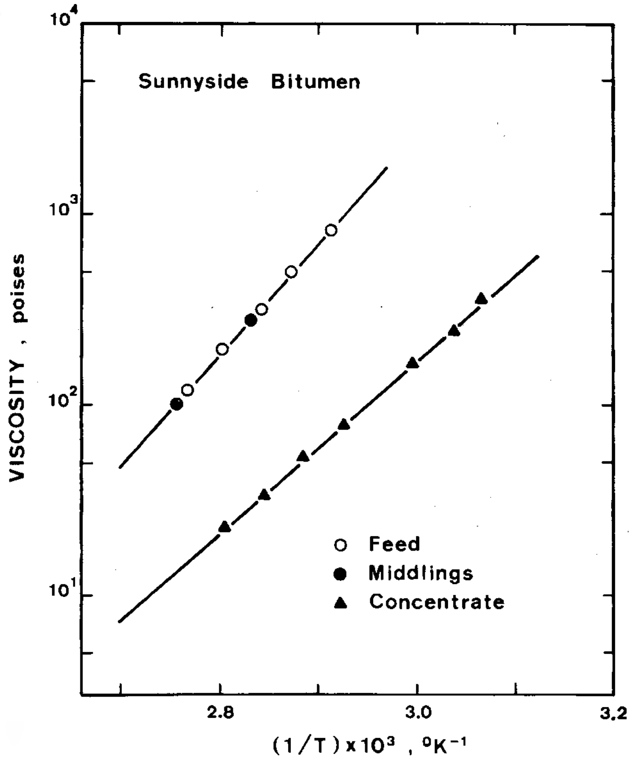

Fig. 12—Arrhenius-type plot illustrating the effect of temperature on bitumen viscosity for products from hot water separation of the Sunnyside sample.

| | Weight % Bitumen in Concentrate | Recovery Bitumen in Concentrate | Coefficient of Separation |
|---|---|---|---|
| Asphalt Ridge | 64.82 | 96.39 | 0.88 |
| Sunnyside | 27.20 | 96.43 | 0.69 |

## Acknowledgments

The authors wish to acknowledge the financial support of DOE Contract No. ET-77-S-03-1762, under the direction of Dr. A. G. Oblad. In addition, the assistance of Maurits Van Camp and J.E. Sepulveda on the execution of this investigation was sincerely appreciated.

## References

Bichard, J.A., Bowman, C.W., Butler, R.M., and Tiedje, J.L., 1974, "Separation of Oil from the Athabasca Oil Sands by Sand Reduction," the Clark volume, a collection of papers on Athbasca Oil Sands, presented to K.A. Clark on the 75th anniversary of his birthday, ed. by M.A. Carrigy, pp. 171-191.

Camp, F.W., 1974, "The Tar Sands of Alberta, Canada," second edition, Cameron Engineering, Inc., Denver, Colo.

Clark, K.A., 1923, Research Council of Alberta Report 1922, Edmonton, Alberta, Canada, pp. 42-58.

Clark, K.A., and Pasternack, D.S., 1932, "Hot Water Separation of Bitumen from Alberta Bituminous Sands," Ind. and Engg. Chem., Vol. 24, No. 12, p. 1410.

Clark, K.A., 1944, "Hot Water Separation of Alberta Bituminous Sand," Canadian Institute of Mining and Metallurgy, *Trans.*, Vol. 47, p. 257.

Cottrell, J.H., 1974, "Development of an Anhydrous Process for Oil-Sand Extraction," the K.A. Clark volume, a collection of papers on Athbasca Oil Sands, presented to K.A. Clark on the 75th anniversary of his birthday, ed. by M.A. Carrigy, pp. 193-201.

Djingheuzian, L.E., 1951, "Cold Water Method of Separation of Bitumen from Alberta Bituminous Sand," *Proceedings*, First Athabasca Oil Sands Conference, King's Printer, Edmonton, Alberta, Canada, September, pp. 185-199.

Innes, E.D., and Fear, J.V.D., 1967, "Canada's First Commercial Tar Sand Development," *Proc.*, Seventh World Petroleum Congress, Vol. 3, Elsevier Pub. Co.

Kruyer, Jan, 1978, "Oleophillic Society of Alberta, *Edmonton Journal,* Wednesday, March 22.

Lowe, R.M., 1975, "Status of Tar Sand, Exploitation in the United States," paper presented at the 68th Annual Meeting of the AIChE, Los Angeles, Calif., Nov. 16-20.

McConville, L.B., 1975, "The Athabasca Tar Sands, The Outlook for the Future," *Mining Engineering,* Vol. 27, No. 1, p. 37.

Oblad, A.G., et al., 1976, "Recovery of Bitumen from Oil-Impregnated Sandstone Deposits of Utah," *Oil Shale and Tar Sands,* AIChE Symposium Series, Vol. 72, No. 155, p. 69.

Porteous, K.C., 1978, "Oil Mining - The Syncrude Project," Syncrude, Canada, Ltd., Edmonton, Albe., Can., presented at The Alternate Resources and Technologies for Fuel Symposium, Univ. of Pittsburgh, Penn., July 31.

Ritzma, H.R., 1973, "Oil Impregnated Rock Deposits of Utah," Utah Geological and Mineral Survey, map 33.

Schulz, N.F., 1970, "Separation Efficiency," *Trans.,* SME/AIME, Vol. 247, p. 81.

"Separation of Bitumen from Dry Tar Sands," 1978, US Patent No. 4,120,766, Oct. 17.

Sepulveda, J.E., Miller, J.D., and Oblad, A.G., 1977, "Hot Water Extraction of Bitumen from Utah Tar Sands," Fuels Div., ACS. Symposium on Oil Shale, Tar Sands, and Related Materials-Production and Utilization of Synfuels, Div. of Fuel Chemistry, ACS,21, No. 6, p. 110, 1976.

Sepulveda, J.E., and J.D. Miller, 1978, "Extraction of Bitumen from Utah Tar Sands by a Hot Water Digestion—Flotation Technique," *Mining Engineering,* Vol. 30, No. 9, p. 1311.

US Bureau of Mines, Report of Investigation, No. 4004.

Walters, E.J., 1974, "Review of the World's Major Oil Sands Deposits," *Oil Sands; Fuels of the Future,* L.V. Hills, Ed., Canadian Society of Petroleum Geologists, Calgary, Albe., Canada.