A Solvent Extraction Process for Tar Sand

by

R. J. Graham, J. J. Helstrom, and R. L. Mehlberg
Amoco Oil Company
Naperville, Illinois 60566

Abstract

A process has been investigated for solvent extraction of bitumen from Sunnyside, Utah, tar sands. The Sunnyside deposit, in east central Utah, has 1 to 2 billion barrels of geological reserves with a richness of 6 to 10 wt% bitumen.

In this process, the ore is crushed and the bitumen is dissolved from the mineral in mix tanks. The bitumen and oil is separated from the coarse mineral by hydrocyclones and from the fines by pentane-deasphaltening. The solvent is recovered from the mineral by centrifuges, filters, and rotary kiln steam strippers, and from the bitumen by multi-effect evaporators.

Hydrocyclone tests with one- and four-inch hydrocyclones demonstrated that 97% of the mineral matter could be removed from the bitumen. The experimental data were used to develop mathematical models for predicting feed rate and cut size. Since the accuracy of the model was quite good, it was used to design the hydrocyclones for the process.

Introduction

Tar sands have received worldwide attention since the commercial development of the giant Athabasca deposit in Canada, but relatively little has been said about U. S. tar sands.

A look at estimated domestic reserves (Figure 1) for coal, oil shale, and tar sands shows why. While different estimating bases can give different absolute values, one conclusion is clear. The estimated reserves of U. S. tar sands are only a small fraction of those for the other two resources. In spite of the low reserves, there are good reasons to consider production of synfuels from domestic tar sands.

First, the heavy oil, or bitumen, produced from most U. S. tar sands is relatively low in sulfur, nitrogen and metals compared to synfuels from coal and shale and high sulfur foreign crudes. Secondly, although the domestic deposits are too small to be of much importance on a national scale, some tar sand deposits could be a significant part of the domestic supply of hydrocarbons for an oil company like Amoco.

Figure 1. Domestic Reserves

As a result, Amoco is currently investigating processes to recover bitumen from a Utah tar sand deposit. Water flotation, pyrolysis, and solvent
extraction processes have been reviewed, but most of our attention has been placed on solvent extraction. One of the conceptual solvent extraction designs that Amoco has investigated from bench scale, test stand, and vendor test data is the subject of this paper.

The Sunnyside Tar Sand Deposit

One of the largest mineable tar sands deposits in the U. S. is located about 120 miles southeast of Salt Lake City in the mountains of Carbon County, Utah (Figure 2). Geologic reserves have been estimated at 1 to 2 billion barrels of bitumen, enough to provide up to 100,000 Bbl/day of syncrude for over 30 years. Amoco owns or controls approximately 50% of the Sunnyside deposit.

Salt Lake City

Sunnyside

Amoco Sunnyside Project

Utah

Figure 2. Location of Sunnyside Tar Sand Deposit

Figure 3 is a schematic of the mountain which contains the Sunnyside resource. Delta systems of ancient rivers emptying into Lake Uinta deposited porous beds of sand interlaced with non-porous beds of shale. At later times bitumen accumulated in the porous sand beds to produce the tar zones shown here. The bitumen is present at 4 to 13 wt% in the porous sand beds. Because of the high viscosity of the bitumen, conventional oil recovery techniques such as steam flooding will not work on this resource. Instead, the tar sand must be mined and processed to recover the bitumen.

The Sunnyside bitumen is a low-gravity, low-sulfur and nitrogen tar like hydrocarbon (Table 1). Only 25 wt% has a boiling point of less than 1,000°F.

Table 1. Properties of Bitumen

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>API Gravity</td>
<td>7.5-10.0</td>
</tr>
<tr>
<td>Carbon wt%</td>
<td>83.3-95.8</td>
</tr>
<tr>
<td>Hydrogen wt%</td>
<td>10.3-11.1</td>
</tr>
<tr>
<td>Nitrogen wt%</td>
<td>0.70-0.79</td>
</tr>
<tr>
<td>Sulfur wt%</td>
<td>0.36-0.46</td>
</tr>
<tr>
<td>Oxygen wt%</td>
<td>0.64-2.49</td>
</tr>
<tr>
<td>Fe wt%</td>
<td>0.08-0.28</td>
</tr>
<tr>
<td>V ppm</td>
<td>0-53</td>
</tr>
<tr>
<td>Ni ppm</td>
<td>45</td>
</tr>
<tr>
<td>Cr ppm</td>
<td>6</td>
</tr>
<tr>
<td>As ppm</td>
<td>2</td>
</tr>
<tr>
<td>Ramsbottom Carbon wt%</td>
<td>12.3-18.5</td>
</tr>
<tr>
<td>OPA Recovery</td>
<td></td>
</tr>
<tr>
<td>Oils wt%</td>
<td>25-30</td>
</tr>
<tr>
<td>Resins wt%</td>
<td>52-58</td>
</tr>
<tr>
<td>Asphaltene wsX</td>
<td>12-23</td>
</tr>
<tr>
<td>Distillation Cuts</td>
<td></td>
</tr>
<tr>
<td>IBP Deg F</td>
<td>560</td>
</tr>
<tr>
<td>1 wt% Deg F</td>
<td>575</td>
</tr>
<tr>
<td>5 wt% Deg F</td>
<td>670</td>
</tr>
<tr>
<td>10 wt% Deg F</td>
<td>760</td>
</tr>
<tr>
<td>20 wt% Deg F</td>
<td>900</td>
</tr>
<tr>
<td>1000°F+ wt%</td>
<td>75</td>
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</table>

The mineral is composed of fine to very fine grained sandstone averaging about 50 wt% quartz, 44 wt% feldspar and 6 wt% other. The small size of the mineral particles and the high viscosity of the tar like bitumen make separation of the mineral from the bitumen difficult. A size distribution plot (Figure 4) shows just how small the Sunnyside mineral particles are. About 50 wt% of the particles are smaller than beach sand; 10 wt% are smaller than human blood cells, and 1 wt% are actually as small as particles in cigarette smoke.

Figure 3. Schematic of Sunnyside Deposit

Figure 4. Sunnyside Tar Sand Particle Sizes

Bitumen Recovery Processes

There are three general types of processes for bitumen recovery from tar sands: Water flotation, pyrolysis, and solvent extraction.
Water flotation, i.e., the hot water process, has been successfully commercialized in Canada, where Suncor and Syncrude produce a total of 175,000 Bbl/day from the rich Athabasca deposit. The hot water process takes advantage of a film of water, which is found between the sand and the bitumen. Tar sand and additional water are contacted with intense mixing, and the bitumen is actually melted and abraded off the sand particles. This process has proven much less successful on Utah tar sands, which lack the thin film of water surrounding each particle and contain bitumen of very high viscosity. Also, the abrasion process can produce a stable, aqueous suspension of clay minerals as a waste stream, causing disposal problems.

In pyrolysis processes, unlike the other options, the bitumen is recovered through chemical conversion. The tar sand is retorted by heating it by mixing with hot recycled sand, to crack and vaporize the bitumen. A cyclone system removes the sand from the product vapor. The coked sand then travels to a combustor, where the coke is burned to provide the heat needed for the pyrolysis reaction. This process suffers when the bitumen content of the tar sands is low, because of the high energy demand of heating the sand. The process upgrades the quality of the bitumen product, but at a substantial yield penalty, because of coke and gas production. Also, high temperatures and pressures make pyrolysis processing technically more demanding.

The third option, solvent extraction, has several attractive features. First, recoveries of bitumen can theoretically approach 100%. Second, for the Sunnyside deposit, operating in the arid western U.S., water usage is lower than for flotation processes. Finally, process tailings contain little residual solvent and no chemical additives, and leave at temperatures only slightly above ambient, simplifying waste disposal. Of these three options, Amoco has concentrated our recent efforts on solvent extraction. One of the conceptual solvent extraction designs that has been backed by bench scale, test stand, and vendor test data is described in this paper.

The Solvent Extraction Process

Figure 5 shows a simplified block diagram of a conceptual solvent extraction process which utilizes hydrocyclones as the primary means of separation. Ore from an open pit mine is crushed in mills. In the extraction block, bitumen is dissolved in heptane solvent, the coarse sand is separated from the solvent and bitumen by hydrocyclones, centrifuges, and belt filters. The fine mineral is removed by pentane deasphalting in the fines removal block. Solvent is recovered from the coarse tailings by steam stripping and from the fine tailings by drying. After the solvent has been recovered from the bitumen in multi-effect evaporators, the bitumen is topped in a crude unit. One option for upgrading is to cokex the 1050°F fraction and hydrotreat the coker products and virgin distillate and gas oil. Coke from the fluid coker and asphaltene sludge from the fine tailings dryer are burned in a circulating fluid bed combustor, CFBC. High pressure steam from the CFBC provides electric power and medium and low pressure process steam.

Figure 5. Block Diagram for Solvent Extraction Process

Crushing

The ore is mined in an open pit mine, crushed in the pit with feeder-breaker crushers to 8-inch top size, and conveyed to the process plant. The ore is fed to crushers and crushed to 3/8-inch top size. Oversized product from the crushers is screened out by a trommel on the end of each unit and conveyed back to the feed stream.

Solvant Extraction (Figure 6)

The extraction section is the heart of the process. Ore from the crushers is fed to the dissolution tank through two stages of rotary air locks to prevent air from entering the tanks. In addition, CO is used to purge the feed and blanket the tanks.

Figure 6. Solvent Extraction

The bitumen is dissolved and the slurry is diluted to 50 wt% solids with recycle solvent in a dissolution tank followed by the hydrocyclone feed tank.

The slurry from the hydrocyclone feed tank is pumped to extraction cascades consisting of eight stages of 10-inch hydrocyclones for removal of the coarse mineral and seven stages of 1-inch hydrocyclones for removal of the middlings. The 10-inch hydrocyclones remove 74% of the mineral and the 1-inch hydrocyclones remove 24% of the mineral leaving only 2% of the original mineral in the extract.

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The coarse mineral underflow from the first stage 10-inch hydrocyclone is counter-currently washed in seven stages of 10-inch hydrocyclones. The 70 wt% solids in the underflow from the eight stage is filtered on a belt filter to recover solvent from the coarse tailings and to produce a cake with only 7 wt% solvent. The ten hydrocyclones and filters recovery all but 0.6 wt% of the soluble bitumen. An additional 3.5 wt% of the bitumen is insoluble and is left on the coarse tailings.

The coarse extract from the first stage of 10-inch hydrocyclone is pumped to a cascade of seven stages of 1-inch hydrocyclones to separate the bitumen-solvent from the middlings and to counter-currently wash bitumen from the middlings.

The 60 wt% solids underflow from the seventh stage 1-inch hydrocyclone flows to decanter centrifuge to recover more solvent and to produce a cake containing 12 wt% solvent. Only 0.3 wt% of the soluble bitumen is lost in the decanter centrifuge cake, but 1.1 wt% of the bitumen is insoluble and is lost with the centrifuge cake.

Equipment sizes and flowrates for the process design were estimated from data from bench scale, test stand, and vendor tests. Because hydrocyclones play such a key role in the process, the emphasis was on obtaining data to design the hydrocyclone cascades.

The hydrocyclones were modeled using the ASPEN process simulation model. The original hydrocyclone equation in the ASPEN model proved to be inadequate for the Sunnyside ore-hydrocarbon solvent system; therefore, it was necessary to build a test stand to evaluate hydrocyclone performance and develop equations for the ASPEN model. Tests were made on Krebs one-inch (Model PC-1 Alumina) and four-inch (Model DAB-12°-834 with Hycar-Liner) hydrocyclones with Sunnyside tar sands in hexane solvent.

The experimental data was used to obtain parameters for L. Svarovsky empirical equations for feed rate and cut size. Cut size is the diameter of those particle which have a 50% chance of being captured in the hydrocyclone underflow. Because the equations are non-linear the parameters were estimated using weighted least squares with the Marquardt algorithm.

The equations with their estimated parameters are:

**Feed Rate**

\[ Q' \sim n_0 \frac{P}{S^*} \]

Where:
- \( K = 1.328 \)
- \( 2 = 0.1 \)
- \( S \) (1-inch hydrocyclones) \( \times 0.56 \text{ m}^2/\text{sec} \)
- \( S^* \) (4-inch hydrocyclones) \( = 4.72 \text{ m}^3/\text{sec} \)

Range for \( Q', 3.0 \times 10^{-1} \text{ to } 40 \times 10^{-4} \text{ m}^3/\text{sec} \) (5.0 to 65 gpm)

**Cut Size**

\[ X_{50} \sim \left( \frac{Q}{(1-d_i)} \right)^{\frac{1}{5}} \]

Where:
- \( K = 1.370 \)
- \( K = 16.91 \)
- \( S \) (1-inch hydrocyclones)

Range for \( X_{50} \), 0.9 to 9.9

All variables are in SI units except for cut size, \( X_{50} \), which is in microns.

The variables are defined in Table 2.

<table>
<thead>
<tr>
<th>Table 2. Variables</th>
</tr>
</thead>
<tbody>
<tr>
<td>( X_{50} ) - cut size, microns</td>
</tr>
<tr>
<td>( d ) - density of particles, kg/m^3</td>
</tr>
<tr>
<td>( d ) - density of bitumen solution, kg/m^3</td>
</tr>
<tr>
<td>( \nu ) - viscosity of bitumen solution, Pa<em>sec or kg/ms</em>sec</td>
</tr>
<tr>
<td>( d ) - diameter of hydrocyclone, m</td>
</tr>
<tr>
<td>( f ) - fraction of liquid in feed reporting to underflow</td>
</tr>
<tr>
<td>( s ) - volume fraction of solids in hydrocyclone feed</td>
</tr>
<tr>
<td>( \sigma ) - standard deviation</td>
</tr>
</tbody>
</table>

Svarovsky’s equations proved to be quite successful in describing the hydrocyclone behavior (refer to Figures 7 and 8).

![Figure 7. Flow Rate Equation for 1 Inch and 4 Inch Hydrocyclones](image1)

![Figure 8. Cut Size Equation for 4 Inch Hydrocyclones](image2)
The feed rate equation was used to fit the data for both the one-inch and the four-inch hydrocyclones, but the cut size equation was used to fit only the four-inch cyclone data. The cut size model could not be tested for the one-inch hydrocyclone because the mineral particles in the one-inch overflow were too small to be measured.

**Fines Removal (Figure 9)**

The middlings extract from the 1-inch hydrocyclone cascade contains 18 wt% bitumen and 8% very fine mineral that must be removed before the bitumen can be upgraded. Removal of the fine sand from the bitumen solvent is a difficult problem, but one solution is to remove the fine sand by pentane deasphalting.

**Figure 9. Fines Removal**

When pentane is added to the slurry, asphaltenes come out of solution and agglomerate the fine mineral to form a heavy sludge. Approximately one pound of pentane is required for each pound of heptane in the bitumen-heptane slurry. After the pentane is mixed with the slurry in an in-line mixer, the fine mineral-asphaltene sludge is settled out in gravity settlers. The settler overflow contains only 4 wt% mineral in the bitumen on a solvent free basis.

The sludge from the settler can be concentrated further to 74 wt% mineral with centrifuges. Only 1.7 wt% of the bitumen that was in the ore is left in the sludge. The centrate is recycled to the hydrocyclone feed tank.

**Tailings Stripping (Figure 10)**

The centrifuge and filter cakes from the extraction section are steam stripped in a rotary kiln. The kiln has lifters that lift the sand up and drop it into a stream of low pressure steam that flows counter-currently from the discharge end of the kiln. The solvent vapor and steam leave the feed end of the kiln and are condensed, separated, and returned to the process. The stripper operates at atmospheric pressure.

Water is added to the tailings to make water concentration approximately 13 wt% to stabilize the tailings. The wet tailings are conveyed to the tailings dump area and revegetated.

**Fines Tailing Drying (Figure 11)**

Solvent and some of the water in the asphaltene-fines sludge from the fines removal block is recovered in a Bethlehem Porcupine Dryer. The dried sludge is conveyed to a circulating fluid combustor to incinerate the asphaltenes and make process steam.

**Figure 11. Fines Tailing Drying**

**Solvent Evaporation (Figure 12)**

The solvent in the settler overflow from the fines removal block is recovered in three stages of rising film evaporators followed by one stage of a forced circulation evaporator. The heptane is separated from the pentane in a splitter and both are returned to the process. Refrigeration equipment is required for the pentane from the splitter. The bitumen, containing 4.0% mineral and 0.5% residual solvent, from the final stage evaporator is pumped to upgrading.

**Figure 12. Solvent Evaporation**
Upgrading (Figure 13)

In the upgrading block, a crude unit splits the bitumen into three cuts; virgin distillate, virgin gas oil, and virgin resid. One way to upgrade the virgin resid is to coke it to produce light and heavy coker naphtha, coker gas oil, and coke. Part of the coke is burned in a coke combustor—CO boiler to provide steam for the upgrading section. The excess coke along with the asphaltene sludge from the fine tailings dryer is burned in a circulating fluid bed combustor.

![Figure 13. Upgrading](image)

Two hydrotreaters are used; a Catalytic Feed Hydrotreater Unit (CFHU) and a Distillate Hydrotreater Unit (DHTU). The CFHU hydrotreats the virgin gas oil from the crude unit and the heavy coker gas oil. The DHTU treats virgin distillate from the crude unit, and the coker naphtha and light coker gas oil. A Vapor Recovery Unit, VRU, recovers C_3 and C_4 from coker gas. The fuel gas from the VRU and the fuel gas from the hydrotreaters go to a hydrogen plant where hydrogen for hydrotreating is manufactured. H,S and ammonia are removed from acid gas and water from a sour water stripper by Stretford gas treating unit with a Claus sulfur plant and amine regenerator.

Syncrude Quality

The hydrotreated product, based on our laboratory tests, is a 27 °API syncrude with a 975° end point (Table 3). Sulfur is less than 200 ppm and nitrogen is less than 400 ppm. Approximately one-half the syncrude is gas oil, one-third is distillate and the remainder is naphtha.

<table>
<thead>
<tr>
<th>Basis: 1 Barrel of C + Syncrude</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gravity</td>
</tr>
<tr>
<td>Light Naphtha</td>
</tr>
<tr>
<td>Heavy Naphtha</td>
</tr>
<tr>
<td>Distillate</td>
</tr>
<tr>
<td>Gas Oil</td>
</tr>
<tr>
<td>Total</td>
</tr>
</tbody>
</table>

Bitumen Recovery and Product Yields

Refined petroleum products (C_+ syncrude and LPG) account for 77.7 wt% of the bitumen (refer to Figure 14). Coke (15 wt% of the bitumen) is burned to produce steam and so is the sludge (1.6 wt% of the bitumen) left on the fine tailings. Fuel gas, 3.6 wt%, is burned or converted to hydrogen for upgrading. Only 5.4 wt% of the bitumen is lost with the coarse tailings. Chemical water, ammonia, and sulfur account for the other one percent.

![Figure 14. Bitumen Recovery](image)

Figure 15 shows what goes into and comes out of the plant. For each barrel of liquid product, 5,300 pounds of tar sand ore (8 wt% bitumen) are required. Water, 200 gallons, is required for wetting down the tailings and to make up for losses in cooling towers, etc. Natural gas, 240 SCF, are required for fuel. By-products are 1.3 pounds of ammonia, 1.0 pounds of sulfur, and 5.2 KWH of electricity; 5,390 pounds of coarse tailings and 230 pounds of ash containing 13 wt% water must be dumped.

<table>
<thead>
<tr>
<th>Basis: 1 BBL syncrude (C_+ and LPG)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
</tr>
<tr>
<td>River water 200 gal.</td>
</tr>
<tr>
<td>B Natural gas 240 cu. ft.</td>
</tr>
<tr>
<td>Power 5.2 kwh</td>
</tr>
<tr>
<td>Product</td>
</tr>
<tr>
<td>By-products</td>
</tr>
<tr>
<td>Plant</td>
</tr>
<tr>
<td>Tailings cake 5390 lbs</td>
</tr>
<tr>
<td>Ash 230 lbs</td>
</tr>
</tbody>
</table>

Summary

Although domestic tar sands reserves are relatively small, some deposits may be large...
enough to provide a secure domestic supply of relatively high quality hydrocarbons in quantities up to 100,000 Bbl/day. Amoco owns or controls 60% of one such deposit in east central Utah, the 1-2 billion barrel Sunnyside deposit.

After considering water flotation, retorting, and solvent extraction, for Sunnyside tar sands, Amoco has concentrated its efforts on solvent extraction. One of the conceptual designs that Amoco is investigating is a process that utilizes hydrocyclones for the primary separation of bitumen and solvent from the mineral. Equations for flowrates and cut size were developed from test stand hydrocyclone data to design hydrocyclone cascades. The process has been extensively tested in bench scale, test stand, and vendor equipment tests.

In the process, the ore is crushed and the bitumen is dissolved from the mineral with heptane solvent. The bitumen and solvent are separated from the coarse mineral by hydrocyclones, and from the fines by pentane deasphalting. The solvent is recovered from the mineral by centrifuges, filters, and rotary kiln steam strippers, and from the bitumen by multi-effect evaporators.

The solvent extraction described in this paper is by no means the final word in solvent extraction processes for Sunnyside tar sand. Research is continuing and new and improved processes are being developed.

References


PROCEEDINGS

1987 EASTERN OIL SHALE SYMPOSIUM

November 18-20, 1987
Hyatt Regency
Lexington, Kentucky

Sponsors:
Commonwealth of Kentucky
Kentucky Energy Cabinet
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Proceedings Publisher:
Kentucky Energy Cabinet Laboratory
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U.S. Department of Defense  
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Indiana Geological Survey  
Bloomington, Indiana

Dr. Roy C. Kepferle  
Eastern Kentucky University  
Richmond, Kentucky

Ms. Patricia A. Lynch  
Institute of Gas Technology  
Washington, D.C.

Ms. Shelia Shelton Medina  
Kentucky Energy Cabinet  
Lexington, Kentucky

Dr. Francis P. Miknis  
Western Research Institute  
Laramie, Wyoming

Mr. Martin C. Noger  
Kentucky Geological Survey  
Lexington, Kentucky

Mr. Paul A. Petzrick  
Oil Shale Association  
Annapolis, Maryland

Mr. Edwin M. Piper  
Stone and Webster Engineering Corporation  
Denver, Colorado

Mr. Raymond C. Rex, Jr.  
HYCRUDE Corporation  
Chicago, Illinois

Dr. Thomas L. Robl  
Kentucky Energy Cabinet Laboratory  
Lexington, Kentucky

Mr. Carl E. Roosmagi  
U.S. Department of Energy  
Laramie Project Office  
Laramie, Wyoming

Ms. Aurora M. Rubel  
Kentucky Energy Cabinet Laboratory  
Lexington, Kentucky

Mr. Gene Tampa  
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Laramie Project Office  
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Lexington, Kentucky

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University of Kentucky  
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Office for Informational Services and Technical Liaison  
Lexington, Kentucky

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Rhonda Pettit  
Proceedings Editor  
Kentucky Energy Cabinet Laboratory  
Lexington, Kentucky

Ellen Spalding  
Layout  
Kentucky Energy Cabinet Laboratory  
Lexington, Kentucky

Betty Gee  
Typesetting  
Kentucky Energy Cabinet Laboratory  
Lexington, Kentucky