

## Oil Shale Ex-Situ Process - Leaching Study of Spent Shale

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**Abstract:** During the past decade, significant advancement has been made on various extraction technologies to develop U.S. oil shale resources in an environmentally and economically sustainable fashion. This work has been driven by the increasing demand for domestic transportation fuels and the need to improve U.S. energy security. Although conventional hydrocarbon deposits are becoming more difficult to find and limited in volume, unconventional reserves are relatively easy to locate and plentiful. Hence, development of unconventional resources, particularly shale gas, oil sands, and shale oil continues to receive tremendous attention.

The present work shows ex-situ process of oil shale in a five ton/day externally heated horizontal rotary reactor, and discuss the process parameters and yield. The main focus of this article is:

A) Effect of reactor bed temperature, rotation speed and feed rate on the residence time, fuel consumption and process yield

B) Hazardous environmental issue related to leaching of heavy metals and metalloids from spent shale by underground and/or surface water, which prevent further commercialization of this process.

In addition, an Aspen diagram of the overall oil shale process is presented as ongoing work focusing on key mechanical issues that affect online reliability and process efficiency including particle size, bed temperature and solid/gas mixing efficiency.

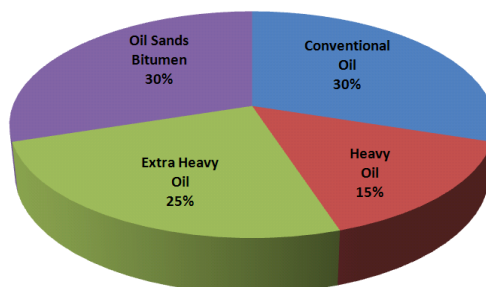
**Keywords:** environmental hazards, ground water pollution, heavy metals leaching, horizontal ex-situ oil shale processing, unconventional hydrocarbon

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### I. Introduction And Background

Conventional petroleum sources are typically those sources which flow freely, when accessed by drilling, due to the pressures in the reservoirs, but Unconventional hydrocarbon sources require additional processing steps to recover the oils including hydro-treating to upgrade the crude into useable liquid transportation fuels. Unconventional petroleum reserves include:

*Heavy oils*, which can be pumped and refined just like conventional petroleum except that they are thicker and have more sulfur and heavy metal contamination, necessitating more extensive refining. *Tar Sands*, which can be recovered via surface mining or in-situ collection techniques. Again, this is more expensive than lifting conventional petroleum but not prohibitively so. Canada's Athabasca tar sands is the best known example of this kind of unconventional reserve. *Oil Shale*, which requires extensive processing and consumes large amounts of water. Still, reserves far exceed supplies of conventional oil [1]. A comparison of world conventional and unconventional oil reserves is shown in Fig 1 [2]. Even though heavy oils and oil sands require extra processing which leads to higher costs of recovery, there is still great interest in recovering these resources because of the vast quantities of unconventional oil found in the U.S. comparing to other foreign oil reserves, shown in Figure 2[3]. This enormous amount of unconventional oil deposits, such as U.S. oil shale, Canadian tar sands and Venezuela's extra-heavy oil, could greatly affect the world's oil inventory. Several ex-situ processes have been developed and used to produce syn-crude from oil sands/oil shale including hot water extraction, external hot gas, indirect heating, and internal combustion. More recently, several in-situ methods have been explored including Shell's in-Situ conversion Process (ICP), ExxonMobil's electrofrac process, Petro Probe superheated air method, and IEP Geothermal Fuel Cell (GFC).



**Figure 1** – comparison of world conventional and unconventional oil reserves

The major challenge for any new ex-situ or in-situ process is transforming it from a pilot demonstration project to a fully commercial plant. This challenge includes several environmental issues including water usage and treatment, greenhouse gas emissions, and land reclamation. Although tremendous advances have been, many challenges remain including energy efficiency, net water demand, CO<sub>2</sub> generation, reclaiming the land, resource recoverability but most importantly, the many mechanical and operational issues related to on-line reliability. One issue related to the environmental impact of this ex-situ process is how to use the spent shale (sands). Generally, this material is an excellent source for making road base or to be used as an additive to concrete. However, heavy metals leaching from the de-oiled material must be further addressed to reduce production uncertainty. Resolving this uncertainty can help further define the production profile required for commercialization.

Back in 80's and early 90's lots of researches have been done regarding spent oil shale leaching experiment. Most of them used the EPA extraction procedure [4,5,6] and ASTM D3987 [7] and two column test methods as reference method. Also, ground water simulation and organic residual leaching were done at the time. Another issue required for commercialization is having a comprehensive process model that describes the full process. A fully verified tool is necessary to establish a detailed and clear business plan. This tool, based on process testing, is necessary to establish an acceptable economic risk profile for process commercialization.

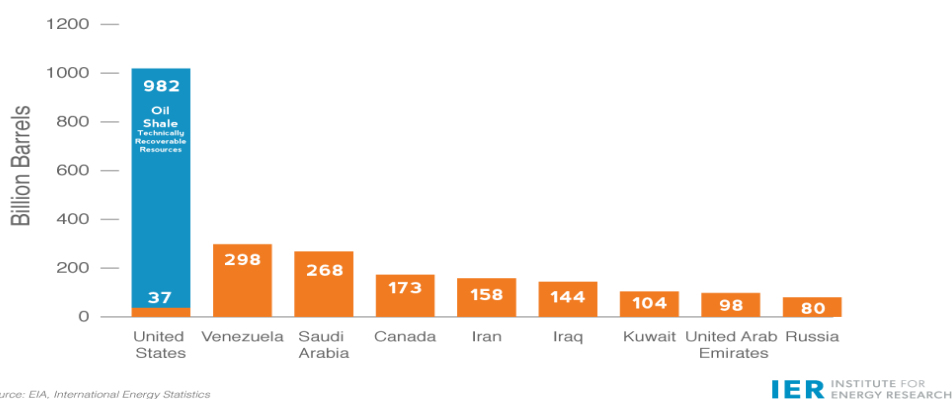


Figure 2 – U.S. oil shale resource vs. foreign oil reserve

## II. Oil Shale Ex-Situ Process Unit

The “two-concentric cylinders” reactor which was used for this experiment is shown in Fig 3. The outer cylinder of this reactor is fixed while the inner one rotates by an electric motor. This 7ft long reactor is utilized with six natural gas burners which are located in the shell space between the two cylinders and heat up the outside surface of the rotary inner cylinder. The first two burners are the large ones supplying lots of heat at the feed entrance following with four lower capacity burners for midstream and downstream as oil shale particles move through the bed. The whole reactor has 1 degree angle to the horizon in order for oil shale particles to move as inner cylinder is rotating. This angle is adjustable from 1 to 3 degree. The rotation speed of inner cylinder could be adjusted from 6 to 20 rpm. Based on the required residence time for specific oil shale type and particle size, the rotation speed and slope of the reactor could be calculated and adjusted.

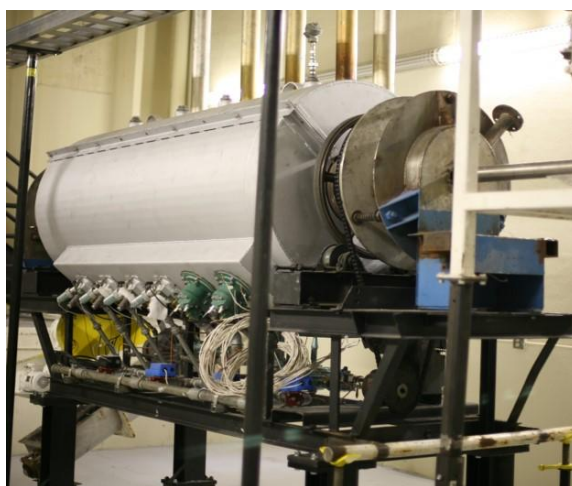


Figure 3 – externally heated horizontal rotary oil shale reactor

The feeding system contains a conveyer which could be fed by buckets and take the oil shale particles 20 ft above to a hopper. This process continues until hopper is filled. After the start up process and heating up the reactor, the shale particles inside the hopper will flow by gravity through an adjustable knife valve at the bottom of the hopper to a screw auger conveyer. The screw conveyer will take the feed into the inner cylinder of the reactor.

There are different types of sweep gas used in this type of processes such as carbon dioxide, nitrogen or superheated steam. In our experiment, steam was used as the sweep gas to push the produced hydrocarbon gas out of the reactor to the distillation unit. The feed rate of superheated steam in our test was 0.05 kg per 1 kg of raw shale and its temperature ranged from 400°C to 550°C. As one of the product coming out of distillation unit, steam was recovered and recycled to the boiler to be sent into the reactor gain. Distillation unit in this experiment has five stages, starting from very heavy hydrocarbon distillate to the light ones as the gas flow through these stages. At the end, the remaining gas which could not be liquefied is sucked by a 5 hp induced draft (ID) fan which pulls the produced HC gas all the way from the reactor through distillation unit and send it to a storage tank. Sometimes a portion of this light gas will be mixed with natural gas to supply fuels for gas burners and reduce natural gas consumption. Distillation unit along with ID fan and gas storage tank are shown in Fig 4.



Figure 4 – distillation unit, induced draft fan and storage tank

## 2.1 Feed Specification And Operational Parameters

The oil shale used in this test was transported from white river basin in eastern Utah where they were retorted in a horizontal kiln at a flue gas temperature of (650°C) at Combustion Resources Inc. in Provo, Utah. White river shale (medium grade) used as feed to this kiln had a bulk density of 78.6 lb/ft<sup>3</sup> with a mean particle size of 1.89 mm which had a gross heating value of 2831 BTU/lb. This shale had a Fischer assay efficiency of 30 gal/ton. A comparison of heating value of raw shale to syn-crude and crude oil is shown in Table 1 [8]. Heat of pyrolysis was reported as 524 KJ/Kg. 11.09% by weight of the wet shale turned to shale oil with 2.4% water and 1.84% gas. 84.67% by weight were reported as spent oil shale shown in Table 2. The produced shale oil from this pyrolysis process had heating value of 15161 BTU/lb.

Table 1 - Comparison of heating values

| Type                       | Raw Shale | Syn-Crude | Crude Oil (Average) |
|----------------------------|-----------|-----------|---------------------|
| Gross Heating Value btu/lb | 2831      | 15161     | 18352               |

The product of oil shale process contain oil, gas, water and spent shale (solid residue). A dry and wet basis weight percentage of the composition of this type of shale are shown in Table 2. These numbers could vary based on the type of the shale being processed. The reactor rotates at 14 rpm and a total of 360 lb of shale was processed at an average feed rate of 6 lb/min. Based on the feed rate of raw shale, the required fuel and air flow rate to supply natural gas burners are shown in Table 3.

**Table 2 – Product wt% composition**

| Component   | Wt% (Wet) | Wt% (dry) | Gal/ton of raw shale |
|-------------|-----------|-----------|----------------------|
| Oil         | 11.09     | 11.36     | 29.46                |
| Gas         | 1.84      | 1.89      |                      |
| Spent Shale | 84.67     | 86.75     |                      |
| Water       | 2.4       |           | 11.11                |

**Table 3 – Natural gas burners’ fuel and air flow rate**

|                                      |      |
|--------------------------------------|------|
| Mass flow rate of raw shale (lb/min) | 6.78 |
| Fuel flow rate (lb/min)              | 0.16 |
| Air flow rate (lb/min)               | 2.82 |

## 2.2 Results

To find out the effect of bed temperature, two tests were performed with different reactor wall temperature. During the first run, the wall temperature started at 750°C at the entrance of the reactor and decreased down along the 7ft length of the reactor to 500°C as it reached the outlet. On the second run, the temperature was kept constant at 500°C along the bed. The corresponding results for these two cases are shown in Fig 5 and Fig 6, respectively. Temperature of the solid residue (spent shale) coming out of the other end of the reactor is shown as  $T_s$  in blue while the produced HC gas temperature  $T_g$  is illustrated in yellow. The red curve shows the reactor wall temperature. The yield of the process was considered as the fraction of raw shale which reacted and is shown in black. Finally, the dark brown and purple line show percentage of carbon dioxide released during the process which in both cases are very low and negligible.

From the results, it is observed that a decrease in temperature at entrance would result in a dramatic decrease in process yield from 94% to 16%. On the other hand, Table 4 shows the effect of initial wall temperature  $T_w$ , rotation speed  $N$  and feed rate on the natural gas (NG) consumption and residence time (RT). The composition of non-condensable oil shale gas is shown in Fig 7. This analysis was based on wt% of the component and shows that methane and ethane are the major components of this gas. From starting point of the process with raw feed to the final refined product are shown in Fig 8. From right to left: crushed raw shale, low temperature solid residue, high temperature solid residue, shale oil, and refined light oil.

**Table 4 – Sensitivity analyses**

| Input    |          |         | Output    |        |
|----------|----------|---------|-----------|--------|
| Feed TPD | $T_w$ °C | $N$ rpm | NG lb/min | RT min |
| 3        | 600      | 6       | 0.09      | 13.8   |
| 3        | 600      | 12      | 0.09      | 6.9    |
| 3        | 600      | 18      | 0.09      | 4.6    |
| 4        | 600      | 6       | 0.12      | 13.8   |
| 4        | 600      | 12      | 0.12      | 6.9    |
| 4        | 600      | 18      | 0.12      | 4.6    |
| 5        | 600      | 6       | 0.15      | 13.8   |
| 5        | 600      | 12      | 0.15      | 6.9    |
| 5        | 600      | 18      | 0.15      | 4.6    |
| 6        | 600      | 6       | 0.18      | 13.8   |
| 6        | 600      | 12      | 0.18      | 6.9    |
| 6        | 600      | 18      | 0.18      | 4.6    |
| 3        | 800      | 6       | 0.09      | 13.8   |
| 3        | 800      | 12      | 0.09      | 6.9    |
| 3        | 800      | 18      | 0.09      | 4.6    |
| 4        | 800      | 6       | 0.12      | 13.8   |
| 4        | 800      | 12      | 0.12      | 6.9    |
| 4        | 800      | 18      | 0.12      | 4.6    |
| 5        | 800      | 6       | 0.15      | 13.8   |
| 5        | 800      | 12      | 0.15      | 6.9    |
| 5        | 800      | 18      | 0.15      | 4.6    |
| 6        | 800      | 6       | 0.18      | 13.8   |
| 6        | 800      | 12      | 0.18      | 6.9    |
| 6        | 800      | 18      | 0.18      | 4.6    |

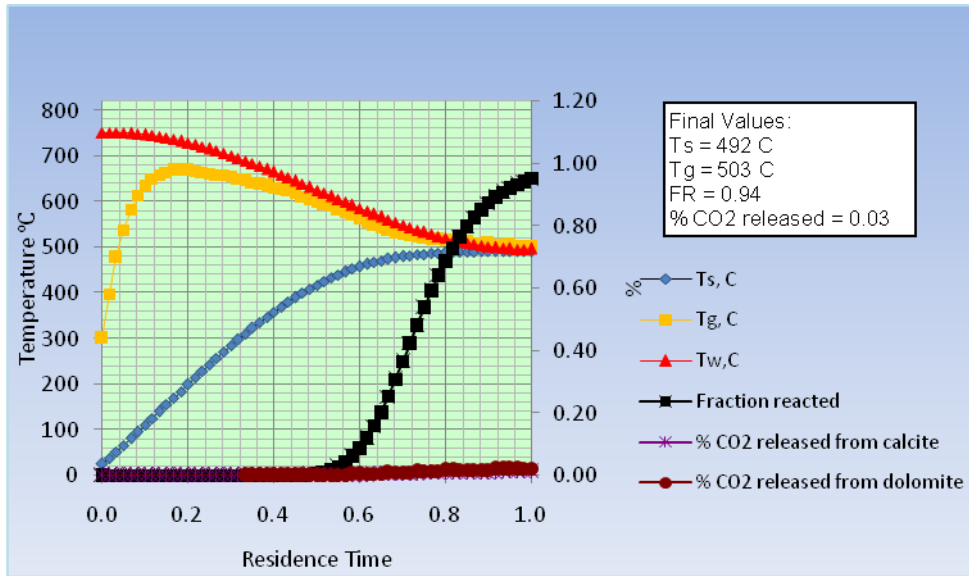


Figure 5 – results for run 1

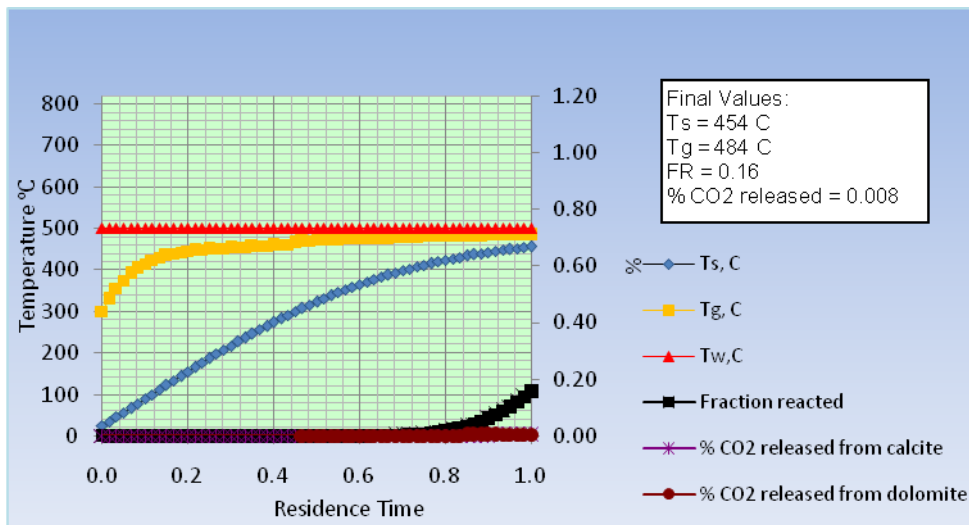


Figure 6 – results for run 2

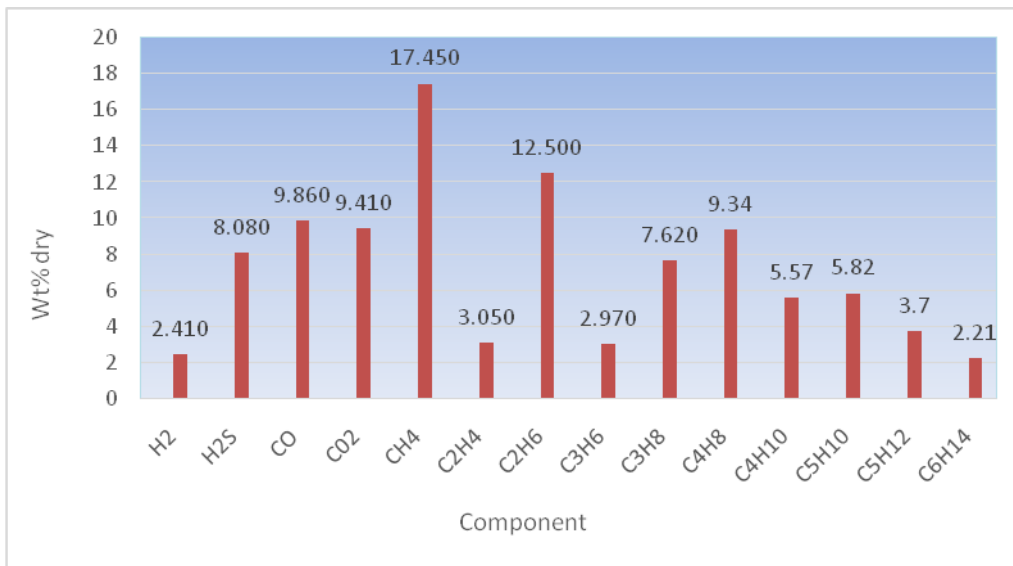


Figure 7 – oil shale non-condensable gas composition





Figure 8 – right to left: raw oil shale, low temp spent shale, high temp spent shale, shale oil, refined oil

### III. Leaching Methodology

Disposal or reuse of solid residue (spent shale) represents an environmental challenge for oil shale (oil sands) processing. These solid residues contain various kinds and levels of heavy metals and metalloids which could be hazardous to human and agricultural life if they enter the underground (and/or surface) water supply. The environmental hazard for surface water is the possibility of ground water movement through retorted oil shale produced by either Ex-situ or In-situ processing. This ground water could leach harmful minerals, heavy metals and salts from the spent process waste (oil shale) and contaminate the potable water supply [9,10]. For this reason, the potential impact on water pollution caused by leaching spent oil shale by ground water was studied. Results of this work is critical and must be considered before commercial production is attempted.

#### 3.1 Total Organic Carbon Test

In this experiment, only one type of spent oil shale was used as a resource for all samples, which had an average size of 2 mm and was mostly spherical in shape. An analysis of the total organic carbon (TOC) in the spent oil shale sample used in the leaching investigation was performed and results are presented in Table 5. As a standard reference, glucose carbon test was also carried out. Glucose Carbon standard values represent weights of carbon detected in a glucose test sample (contains approximately 40% carbon). Results are shown in Table 6 and Fig 9.

Table 5 – Total organic carbon of spent shale

| Sample     | Spent shale (mg)     | Peak area | TOC (µg/mg of sample) | Average TOC (µg/mg of sample)                     |
|------------|----------------------|-----------|-----------------------|---|
| 1          | 101.5                | 787.6     | 61.4                  | <b>64.24</b>                                      |
| 2          | 78.32                | 671.2     | 67.8                  |   |
| 3          | 83.43                | 668.6     | 63.4                  |   |
| Spike test | 81.75 <sup>(1)</sup> | 1290      | 10.32 <sup>(2)</sup>  | Conducted after testing to confirm average result |

<sup>(1)</sup> mg of sample + 5 mg carbon

<sup>(2)</sup> Expected value is 10.21 (test result recovery is 101.03%)

Table 6 – Glucose standard carbon test

| Glucose standard carbon (mg) | 0 | 1.25  | 2.5   | 5   | 10   | 15   | 20   |
|------------------------------|---|-------|-------|-----|------|------|------|
| Peak area                    | 0 | 158.3 | 300.5 | 615 | 1213 | 1889 | 2585 |

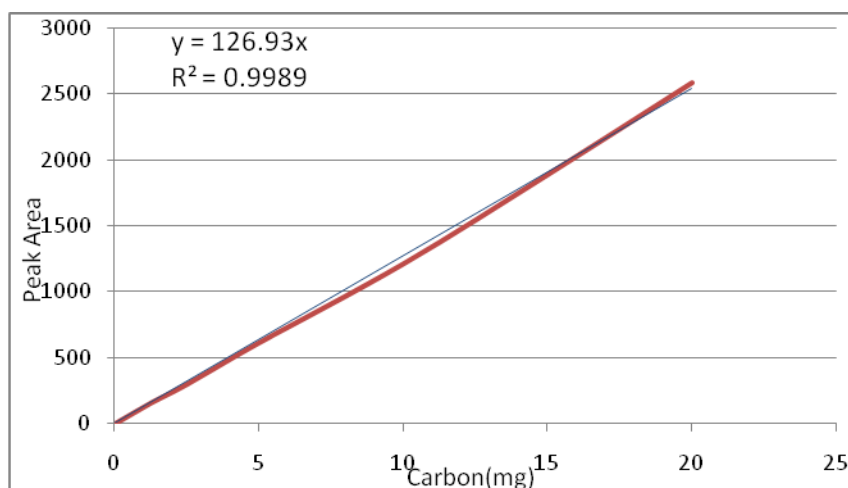


Figure 9 – peak area vs. carbon plot

### 3.2 Experimental Matrix

Test samples were selected randomly from the resource. Water was used as the leachate and a solution of 3% nitric acid as a miscible liquid was mixed with water to approximate ground water [11,12]. Two different pH levels were considered for ground water, pH = 4 and pH = 5, plus two different solid to liquid ratio (S/L) values, S/L = 0.1 and S/L = 0.05 were considered. For the first case, a ratio of 0.1 for solid to liquid was used. To achieve this S/L ratio, 10 grams of spent oil shale (solid) was mixed with 100 grams of water/acid mixture (liquid) with a pH of 4. To get an estimate of experimental error, five replications were completed for each combination. For the second case, the pH was held constant at the same level as in the first test but the S/L was changed to 0.05. This was achieved by mixing 5 grams of spent oil shale with 100 grams of water/nitric acid in the sample container. The leachate pH was then increased to 5 for the third test condition with S/L of 0.1. Finally, a fourth condition was completed with a pH of 5 and a S/L of 0.05. The full test included twenty sample containers which were placed in a shaker for 24 hours, operating at 180 shakes per minute.

### 3.3 Results

After 24 hours, samples are filtered through a special filter (non-sterile syringe filter, pore size 0.2μ) and are placed in Inductively Coupled Plasma Mass Spectrometry or ICP-MS, designed to detect various element types including heavy metals and provide a quantitative measure of their concentration. ICP-MS has many advantages including: a) detection limits for most elements equal to or better than those obtained by Graphite Furnace Atomic Absorption Spectroscopy (GFAAS), b) higher throughput than GFAAS, and c) the ability to obtain isotopic information. Using ICP-MS, twomixtures of nitric acid and water were prepared as blanks for two different pH levels used in these tests. Metal concentration in the blanks are shown in Table 7. Also, the results of each case of our “Leaching” experiment are shown from Table 8 to Table 11.

**Table 7 – Metals concentration in blanks**

| Sample ID       | Concentrations in ppb |      |      |      |      |      |      |      |      |      |      |      |
|-----------------|-----------------------|------|------|------|------|------|------|------|------|------|------|------|
|                 | Be                    | Cr   | Ni   | Cu   | Zn   | As   | Se   | Cd   | Sb   | Ba   | Pb   | Ti   |
| Blank pH 5      | <DL <sup>(1)</sup>    | <DL  | 1.98 | 0.27 | <DL  | <DL  | <DL  | <DL  | <DL  | 0.23 | <DL  | <DL  |
| Blank pH 4      | <DL                   | <DL  | 1.13 | 0.61 | 1.95 | <DL  | <DL  | <DL  | <DL  | 0.62 | 0.09 | <DL  |
| Detection Limit | 0.02                  | 1.50 | 1.00 | 0.20 | 1.00 | 0.20 | 1.00 | 0.02 | 0.20 | 0.05 | 0.05 | 0.05 |

<sup>(1)</sup>DL stands for Detection Limit

**Table 8 – Case 1: pH = 4 and S/L = 0.1**

| Sample ID       | Concentration (ppb) |       |       |       |       |        |        |        |        |        |       |       |
|-----------------|---------------------|-------|-------|-------|-------|--------|--------|--------|--------|--------|-------|-------|
|                 | Be                  | Cr    | Ni    | Cu    | Zn    | As     | Se     | Cd     | Sb     | Ba     | Pb    | Ti    |
| Spike 20ppb     | 14.87               | 17.34 | 17.38 | 16.32 | 17.77 | 55.04  | 31.68  | 22.01  | 33.80  |        | 18.32 | 25.24 |
| Spike recovery% | 74.35               | 81.66 | 77.12 | 77.33 | 86.26 | 105.01 | 118.55 | 103.08 | 108.01 |        | 91.52 | 126.0 |
| 1-10 pH 4 A     | <DL                 | <DL   | 2.14  | 0.95  | <DL   | 32.02  | 9.92   | 1.55   | 13.08  | 118.13 | <DL   | <DL   |
| 1-10 pH 4 B     | <DL                 | <DL   | 1.96  | 0.85  | <DL   | 34.04  | 7.97   | 1.40   | 12.20  | 114.70 | <DL   | <DL   |
| 1-10 pH 4 C     | <DL                 | <DL   | 1.75  | 0.77  | <DL   | 29.25  | 9.65   | 1.67   | 12.69  | 126.64 | 0.08  | 0.05  |
| 1-10 pH 4 D     | <DL                 | <DL   | 2.05  | 0.89  | 1.03  | 29.82  | 10.55  | 1.60   | 12.95  | 120.52 | <DL   | <DL   |
| 1-10 pH 4 E     | <DL                 | <DL   | 1.76  | 0.74  | <DL   | 29.38  | 9.72   | 1.60   | 12.95  | 130.57 | <DL   | <DL   |

**Table 9 – Case 2: pH = 4 and S/L = 0.05**

| Sample ID       | Concentration (ppb) |       |       |       |       |       |        |        |        |        |       |        |
|-----------------|---------------------|-------|-------|-------|-------|-------|--------|--------|--------|--------|-------|--------|
|                 | Be                  | Cr    | Ni    | Cu    | Zn    | As    | Se     | Cd     | Sb     | Ba     | Pb    | Ti     |
| Spike 20ppb     | 16.64               | 16.19 | 16.34 | 15.42 | 17.65 | 56.14 | 30.15  | 21.12  | 27.61  |        | 16.45 | 22.05  |
| Spike recovery% | 83.19               | 77.48 | 74.86 | 73.65 | 86.49 | 93.73 | 114.92 | 101.63 | 102.35 |        | 82.08 | 110.12 |
| 1-20 pH 4 A     | <DL                 | <DL   | 1.37  | 0.69  | <DL   | 37.39 | 7.16   | 0.79   | 7.14   | 96.62  | <DL   | <DL    |
| 1-20 pH 4 B     | 0.03                | <DL   | 1.11  | 0.66  | <DL   | 37.20 | 6.68   | 0.86   | 7.57   | 101.07 | 0.10  | 0.05   |
| 1-20 pH 4 C     | <DL                 | <DL   | 1.03  | 0.71  | <DL   | 34.95 | 7.00   | 0.87   | 7.70   | 103.56 | <DL   | <DL    |
| 1-20 pH 4 D     | <DL                 | <DL   | 1.11  | 0.68  | <DL   | 30.25 | 6.79   | 0.83   | 7.84   | 106.19 | <DL   | <DL    |
| 1-20 pH 4 E     | <DL                 | <DL   | <DL   | 0.61  | <DL   | 28.10 | 6.44   | 0.74   | 7.79   | 111.02 | 0.06  | <DL    |

**Table 10 – Case 3: pH = 5 and S/L = 0.1**

| Sample ID       | Concentration (ppb) |       |       |       |       |        |        |        |        |        |       |        |
|-----------------|---------------------|-------|-------|-------|-------|--------|--------|--------|--------|--------|-------|--------|
|                 | Be                  | Cr    | Ni    | Cu    | Zn    | As     | Se     | Cd     | Sb     | Ba     | Pb    | Ti     |
| Spike 20ppb     | 13.72               | 16.52 | 16.77 | 15.60 | 16.72 | 48.07  | 33.60  | 21.85  | 34.33  |        | 17.58 | 24.22  |
| Spike recovery% | 68.56               | 79.22 | 74.82 | 73.98 | 81.82 | 103.63 | 117.20 | 100.58 | 104.19 |        | 87.74 | 120.90 |
| 1-10 pH 5 A     | 0.04                | <DL   | 1.91  | 0.84  | <DL   | 28.02  | 11.59  | 1.75   | 16.22  | 113.09 | 0.08  | 0.09   |
| 1-10 pH 5 B     | <DL                 | <DL   | 1.81  | 0.80  | <DL   | 27.35  | 10.16  | 1.73   | 13.49  | 113.61 | <DL   | <DL    |
| 1-10 pH 5 C     | <DL                 | <DL   | 1.68  | 0.88  | <DL   | 32.93  | 10.76  | 1.48   | 14.48  | 131.60 | 0.07  | <DL    |
| 1-10 pH 5 D     | <DL                 | <DL   | 2.03  | 0.92  | <DL   | 29.29  | 10.70  | 1.55   | 13.42  | 116.35 | <DL   | <DL    |
| 1-10 pH 5 E     | <DL                 | <DL   | 1.39  | 0.84  | <DL   | 31.27  | 9.15   | 1.38   | 12.93  | 151.28 | <DL   | 0.06   |





## V. Conclusion

Oil shale was processed through an externally heated rotary horizontal reactor and liquid fuel and non-condensable gas were produced. Sensitivity analyses tests were carried out and the effects of operational parameters such as reactor rotational speed, bed temperature and feed rate on the required natural gas, residence time and also the yield of the process were discussed. It was found that CO<sub>2</sub> emission was very low for this process and recycling the superheated steam will result in more sustainability and lower water consumption. Ultimate and proximate analysis of the raw feed and produced non-condensable gas were presented.

The leachability of heavy metals in spent oil shale was studied and it was introduced as an environmental issue. The results of leaching test illustrated the impacts of solid to liquid ratio and pH increase on the leachability of heavy metals and their concentration in the leachate.

Many necessary steps are needed to bring a new process to commercial operation such as developing a detailed process description including detailed flow diagrams with mass and energy balances for the major process variations and feeds. However, this work is only preliminary and a far more detailed and accurate analysis will be possible once better design data can be obtained.

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