

Oil & Natural Gas Technology

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Quarterly Report October – December 2011 Post Retort, Pre Hydro-treat Upgrading of Shale Oil

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1. EXECUTIVE SUMMARY

In the 9th quarter considerable progress was made. A presentation was made at the Oil Shale Symposium titled, “Novel process for shale oil upgrading without using hydrogen”. The presentation was well attended and raised awareness of the technology. Reactor experiments were conducted on coker diesel which contained refractory nature sulfur compounds originally in coke with 1.8% sulfur content, nearly 98% sulfur was removed. Also bitumen from the McKay River in Alberta was processed under various conditions which had an initial API of about 8 and starting sulfur content 5.1%. Up to 97% of the sulfur was removed and API was increased to 19, and TAN was reduced from 5 to 0. These are encouraging results both for refinery stream processing as well as pre-processing feedstocks which may be sent to the USA by pipeline. Meanwhile the electrolysis process is still looking very encouraging. The long term test cell has longed over 5000 hours with no apparent membrane degradation. The program is expected to have funds remaining at the scheduled program end so a no cost extension has been requested.

2. PROGRESS, RESULTS AND DISCUSSION

2.1 Task 1.0 -- Project Management Plan

The PMP was updated within 30 days and submitted to the Project Manager (Quarter 1).

2.2 Task 2.0 -- Upgrading Development

It is explained in detail in 6th Quarterly report.

2.3 Task 3.0 -- Electrolysis Development

It is explained in detail in 6th Quarterly report.

2.4 Task 4.0 -- Analysis

It is explained in detail in 6th Quarterly report.

Budget Period 2

2.5 Task 5.0 – Upgrading Development

This task is related to developing the process of treating shale oil, or heavy oil at elevated temperature and pressure in the presence of an alkali metal, either sodium or lithium and also a hydrogen source, either hydrogen gas or methane (natural gas) to form an oil stream with reduced levels of sulfur, nitrogen and heavy metals and also in the process reducing the viscosity and increasing the API gravity. The object here is to determine the impact of various reaction parameters on product quality.

2.5.1 Subtask 5.1 – Analytical Capability

Analytical laboratory set up

There are no major changes in analytical capability.

2.5.2 Subtask 5.2 – Upgrading reactor and Separation setup

Additional experiments were performed with bitumen and Coker diesel as feedstocks. The bitumen specifications are shown in Table 1 below.

Table 1: Specifications of Bitumen

| C | H | N | S | API | TAN | Ni | V | Fe |
|------|-------|-----|-----|------|-----|----|-----|----|
| 83.7 | 10.03 | 0.4 | 5.1 | 7.97 | 5.2 | 77 | 213 | 3 |
| | | | | | | | | |

The various different runs are listed in Table 2 below. The results of the runs are shown in Table 3. As observed from the Table, 96.5% sulfur was removed from the bitumen (Run B1) in presence of hydrogen whereas methane as a cover gas resulted into maximum 88.6% sulfur removal (Run B4). API of the upgraded product was 19 and 17 respectively. It can be concluded that, hydrogen as a cover gas is more effective at upgrading compared to methane.

Table 2: List of upgrading experiments for Bitumen

| Run ID | Gas | Actual Na/ Theoretical Na | Reaction Time | Reactor Pressure | Reactor Temp. |
|--------|-----|---------------------------|---------------|------------------|---------------|
| | | | Min. | Psig. | C |
| B1 | H2 | 0.98 | 127 | 1049 | 391 |
| B2 | CH4 | 0.63 | 123 | 1324 | 395 |
| B3 | H2 | 1.01 | 39 | 1136 | 383 |
| B4 | CH4 | 1.00 | 40 | 1431 | 403 |
| B5 | H2 | 1.08 | 245 | 980 | 385 |

Table 3: Results of Bitumen upgrading

| Run ID | Sulfur moved | Re- | Nitrogen Rem | Liquid Fraction Mass Yield | API | Hydrogen Consumption | Light ends (C1-C6) Formed |
|--------|--------------|-----|--------------|----------------------------|------|----------------------|---------------------------|
| | % | | % | % | | scf/barrel | scf/barrel |
| B1 | 96.5% | | 43.5% | 89.1% | 19.1 | 335.5 | 153.9 |
| B2 | 76.2% | | 26.8% | 91.4% | 13.0 | N/A | 145.2 |
| B3 | 63.6% | | 5.4% | 91.6% | 13.0 | 114.4 | 154.6 |
| B4 | 88.6% | | 28.5% | 73.2% | 17.0 | N/A | 206.4 |
| B5 | 95.6% | | 12.9% | 84.1% | 16.8 | 193.1 | 154.6 |

Sulfur Removal from Coker Diesel

Coker diesel was treated in the upgrading reactor with Nitrogen and hydrogen as cover gases respectively. The initial coker diesel has a sulfur content of 1.8%. The experiments were performed at 275 C and 450 psig gas pressure.

Table 4 shows the detailed results. The sodium charge was approximately 6 g. The results show upto 97.7% sulfur removal using hydrogen as a cover gas. With nitrogen as a cover gas, up to 90% sulfur was removed.

Table 4: Results of Coker diesel desulfurization

| Run ID | Gas | Actual Na/ Theoretical Na | Sulfur Removed | Liquid Fraction Mass Yield | API |
|--------|-----|------------------------------|----------------|----------------------------|-------|
| | | | % | % | |
| CR1 | N2 | 1.10 | 83.2 | 88.5 | 35.20 |
| CR2 | N2 | 1.07 | 90.3 | 86.05 | 35.24 |
| CR3 | N2 | 1.10 | 94.1 | 85.2 | 35.31 |
| CR4 | H2 | 0.66 | 72.4 | 94.5 | 33.12 |
| CR5 | H2 | 0.94 | 90.4 | 92.5 | 34.46 |
| CR6 | H2 | 1.16 | 97.7 | 89.8 | 35.79 |

Additional flow meters, and data loggers were installed to provide better mass balance data and monitoring of process parameters during runs.

2.6 Task 6.0 - Electrolysis development

To reduce the overall cost of the upgrading process, an electrolysis process will be developed to regenerate sodium or lithium from the respective polysulfide. The process will feature ceramic ion conductive membranes developed at Ceramatec. The energy cost to regenerate the alkali metals from the polysulfide is expected to be about half that of producing the metals from their respective chlorides.

2.6.1 Subtask 6.1 – Membrane fabrication

The Recipient shall fabricate and characterize sodium conductive and lithium conductive membranes.

2.6.2 Subtask 6.2 – Seal testing

Ceramatec shall evaluate various seal approaches for compatibility with the alkali metal and the metal polysulfide at various temperatures.

2.6.3 Subtask 6.3 – Cell design and set up

Ceramatec shall design benchtop cells for two types of operation, one where the alkali metal is molten and one where it plates onto a current collector. Reactors and catholyte transfer means will be provided to prepare alkali metal sulfide of differing composition and transfer to the cell. The cells will be designed to accommodate multiple reference electrodes, operate at various elevated temperatures. The cells will have features designed to facilitate sulfur removal and be designed to operate within a dry enclosure.

2.6.4 Subtask 6.4 – Cell operation

The Recipient shall operate cells under various conditions including variation of the current density, electrode gap, temperature, electrolyte, polysulfide order, and alkali metal. Current will

be measured as a function of applied voltage. Periodically cell operation will be interrupted and cell contents analyzed to determine current efficiency. In Phase 1 the alkali metal polysulfide will be synthesized from alkali metal and sulfur and will not contain appreciable impurities which may flow through from an actual upgrading process as will occur in Phase 2.

Long term test cell results

One cell has been operated as long term test during this reporting period. Table 5 summarizes the properties of the cell. Sodium polysulfide (Na_2S_4) is periodically added to the anolyte solution in the cell to replenish the sodium as the ions are transported and reduced to sodium metal in the cathode. Cell *Na_molten_20110504* has been in continuous operation for 5044 hours (210 days) at a constant current density of 60 mA/cm^2 . Figure 1 shows the cell voltage and the Open Circuit Voltage (OCV) during the test. The average cell voltage for the run is equal to 3.10V. The cell's Nernst potential or OCV (Open Circuit Voltage) varied between 2.1 and 2.3V, with an average around 2.2V. Table 6 summarizes the cell's key metrics. A total of 1933 grams of sodium tetrasulfide Na_2S_4 has been added to the cell, from which 442 grams of sodium have been recovered. This represents approximately 86.5% of the total sodium added as sodium polysulfide to the cell.

As documented in past quarterly reports, we have detected small amounts hydrogen sulfide gas being generated in the cell. Since the cells are operated inside a glove box in a dry nitrogen environment, we have speculated that the gas comes from electrochemical decomposition reactions between the anolyte solvent and the sodium polysulfide. Aging of the anolyte solvent is evident as the test goes on and it is manifested as an increase in viscosity and in the reduction of the ionic conductivity. This explains the slow but steady increase in the cell operating voltage with each new anolyte, as shown in Figure 1. We have measured the sulfur composition of the anolyte solution during the test. Figure 2 compares the total cumulative sulfur added and the total sulfur measured, expressed as weight percent of the anolyte solution, during the long term test with Anolyte #4. The difference between the sulfur added and the sulfur measured is equal to the sulfur lost as H_2S gas plus the elemental sulfur, which precipitates out of the anolyte solution. The figure indicates that it takes approximately 240 hours (after adding fresh anolyte solution) to reach a 46% difference between sulfur added and measured. Then, this difference is maintained for the remaining of the anolyte life. If we discount the amount of elemental sulfur recovered from the anolyte after separation, the sulfur losses account for 37% of the total sulfur added. A total of six anolyte solutions (five replacements) have been used so far during the 5044 hours of the test. After replacing the anolyte, the cell voltage decreases every time to a value similar to that of the beginning of the test. This is proof of the good condition of the membrane since little or no over potential due to the NaSICON solid electrolyte has been measured.

In the next quarter, sodium tetrasulfide (Na_2S_4) will be replaced with sodium sulfide (Na_2S) in the long term test cell.

Table 5: Summary of sodium recovery test cells run in long-term mode

| Cell ID# | Anolyte | Anode Electrode | Membrane | Catholyte | Cathode Electrode | Seal Type | Operating Conditions |
|---------------------------|--------------------------------------|---------------------------------|-------------------------------------|-----------|-------------------------------------|--------------------|--|
| Na Recovery Cell 20110504 | Na ₂ S ₄ in MF | Platinized Ti mesh (1.1" diam.) | NaSICON GY (1 mm thick, 0.8" diam.) | Molten Na | Molten Na, Ti rod current collector | Silica-Boria Glass | Temperature=130C Anolyte agitation Cte Current=60 mA/cm ² |

Table 6: Summary of performance of sodium recovery test cells run in long-term mode at 60 mA/cm² current density

| Cell ID | Total Run Time | Avg. Voltage | Avg. OCV | Number of Anolyte Solution Replacements | Total Na ₂ S ₄ Added | Na Recovered | | Notes |
|---------------------------|----------------|--------------|----------|---|--|--------------|-------------------|--|
| | Hours (days) | V | V | | | g | % out of Na added | |
| Na Recovery Cell 20110504 | 5044* (210) | 3.10 | 2.20 | 5 | 1933 | 442 | 86.5 | Cell is still in operation with membrane in excellent conditions |

* Total time includes short time periods to measure the cell OCV

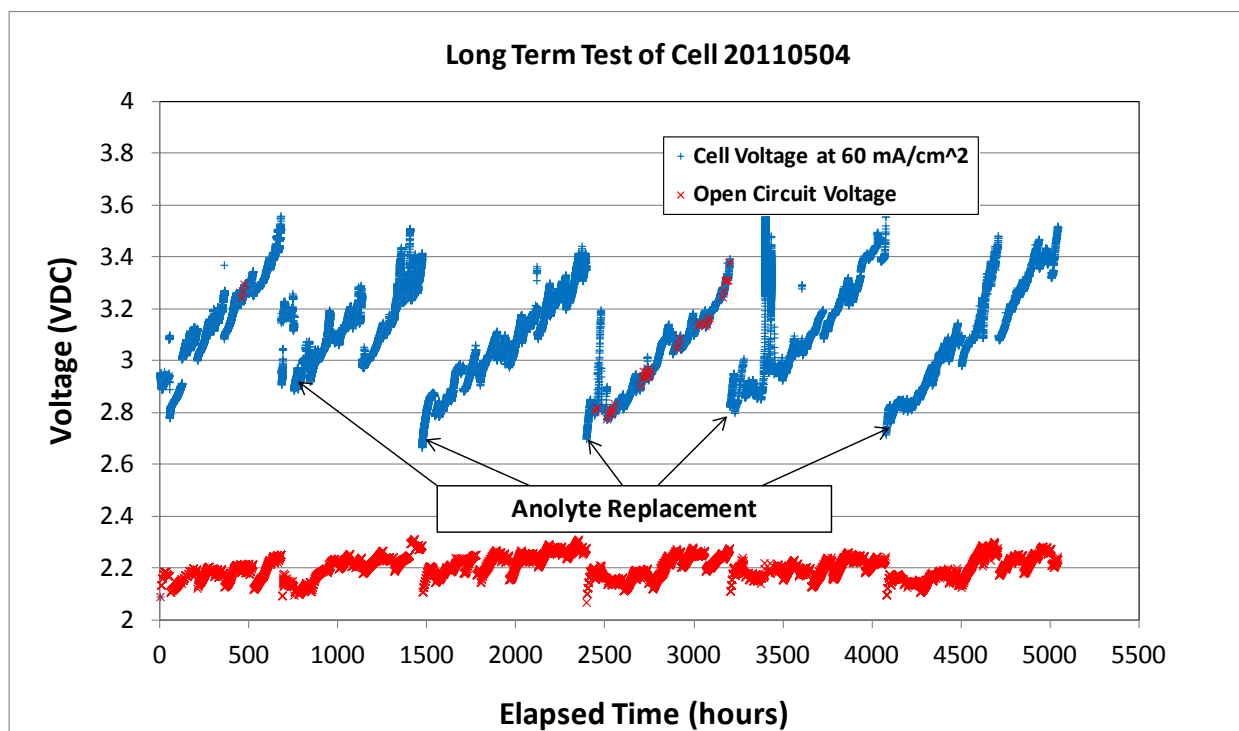


Figure 1: Cell voltage at 60 mA/cm² and Open Circuit Voltage (OCV) versus run time for sodium recovery cell Na_molten_20110504. Run time includes time periods for OCV measurements (no current).

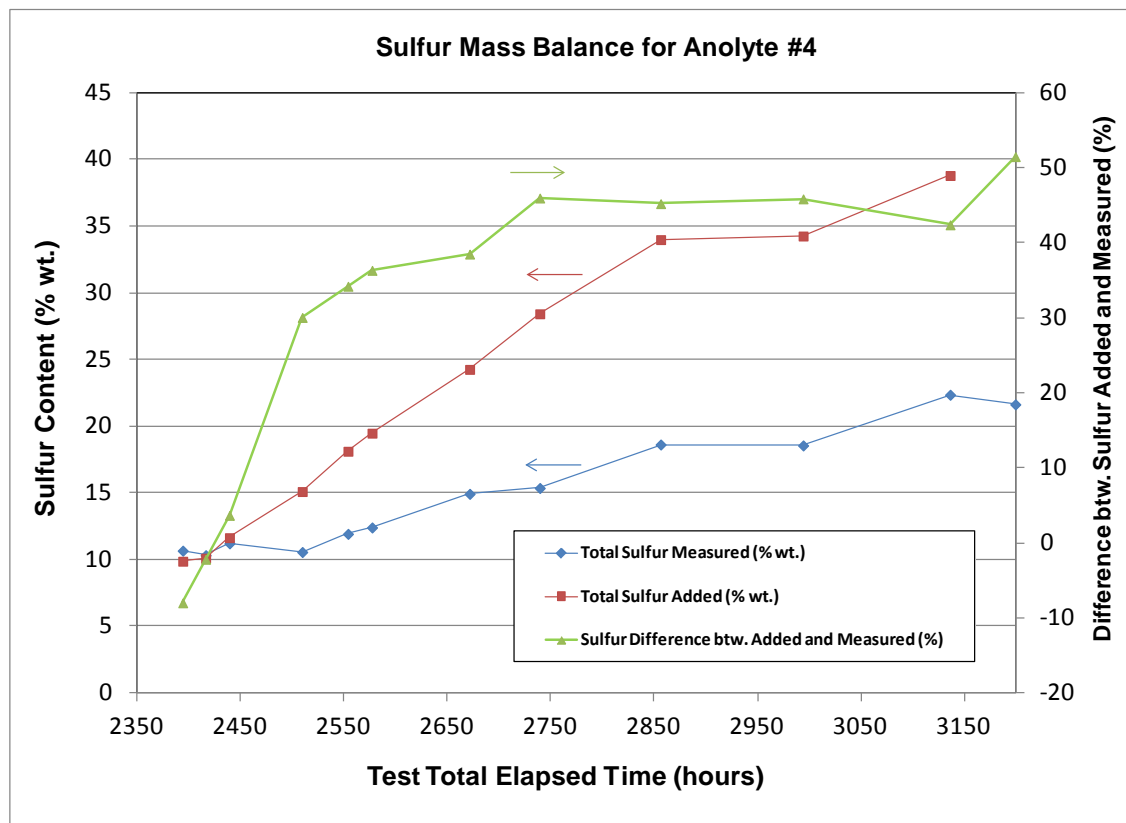


Figure 2: Comparison between total sulfur added and measured for Anolyte #4 during long term test for cell Na Recovery Cell 20110504

Sodium recovery test cells results using solids from processed oil samples:

A sample of Coker Diesel feedstock was procured in this quarter. A total of six upgrading tests, using sodium and hydrogen or nitrogen as covered gas, were planned and conducted. The reaction was done at a temperature and pressure of 280°C and 425 psig, respectively. At the end of the reaction batch time, the reaction products were decanted to separate the solid and liquid fractions. The solids were washed with hexane to dissolve the remaining oil and then centrifuged to separate the washed solids from the liquid. The washed solids were dried at 80°C to evaporate the remaining hexane solvent. Figure 3 shows pictures of the dried solids for four of the runs (CR2, CR3, CR4, and CR5). CR2 and CR3 were run using nitrogen as covered gas, whereas CR4 and CR5 used hydrogen. There is a clear difference in color and composition between the two set of solids. While they all have sodium sulfide (Na₂S), the amount of sulfide in the hydrogen run solid samples is significantly higher. Moreover, the higher carbon content of the nitrogen run solid samples evidences the larger extent of thermal cracking that occurs when hydrogen gas is not present. The electrolyte solution for the electrolysis runs is prepared by dissolving the dried solids in our preferred polar organic solvent. The electrolyte solutions obtained from the hydrogen runs had higher ionic conductivities and were less viscous than the solutions from the nitrogen runs. A total of four cells were assembled and run during the reporting period (see Table 7). Figure 4 shows the cell voltage and the current density versus test elapsed time for cell

Coker_Diesel_CR2_20111108. The cell was run in constant voltage mode, initially at 3V for 10 hours and then at 3.5V for the remaining of the 140 hr test. A maximum current density of 30 mA/cm² was only achieved at 3.5V, with a significant cell deactivation as shown by the quick current density drop in Figure 4. Test cells *Coker_Diesel_CR4_20111110*, *Coker_Diesel_CR5_20111111*, and *Coker_Diesel_CR6_20111116* run much better reaching and maintaining in all cases the optimum current density of 60 mA/cm². However cell deactivation, as evidenced by carbon rich deposits on the surface of the anode electrode, was still observed towards the end of the runs.



Figure 3: Picture of the dried solid samples of two nitrogen Coker Diesel runs (CR2, CR3) and two Coker Diesel hydrogen runs (CR4, CR5)

Table 7: Sodium recovery test cells, using solids from processed Coker Diesel runs, assembled and tested during the reporting period

| Cell ID# | Anolyte | Anode Electrode | Membrane | Catholyte | Cathode Electrode | Cell Operating Conditions |
|---------------------------|---|--|---|-----------|--------------------------|--|
| Coker_Diesel_CR2_20111108 | 273.6 g of solution with 6.8 wt.% solids in MF | Platinized Ti mesh (1" diameter, 3 mm from membrane) | NaS GY pressed disk (0.51 mm thick) Active area=1.86 cm ² | Molten Na | Ti rod current collector | Temperature=130C Cte Votage=3.5V |
| Coker_Diesel_CR4_20111110 | 238.6 g of 4.1 wt% CR4 solids in MF | Platinized Ti mesh (1" diameter, 3 mm from membrane) | NaS GY pressed disk (1 mm thick) Active area=1.84 cm ² | Molten Na | Ti rod current collector | Temperature=130C Cte Current=60 mA/cm ² Cte Votage=3.5V |
| Coker_Diesel_CR5_20111111 | Added 14.3 g of CR5 solids to anolyte of Coker_Diesel_CR4_20111110 Cell. 5.8% wt. solids solution in MF | Platinized Ti mesh (1" diameter, 3 mm from membrane) | NaS GY pressed disk (1 mm thick) Active area=1.84 cm ² | Molten Na | Ti rod current collector | Temperature=130C Cte Current=60 mA/cm ² Cte Votage=3.5V |
| Coker_Diesel_CR6_20111116 | 314.9 g of 5.4wt% CR6 solids in MF | Platinized Ti mesh (1" diameter, 3 mm from membrane) | NaS GY pressed disk (0.41 mm thick) Active area=2.0 cm ² | Molten Na | Ti rod current collector | Temperature=130C Cte Current=60 mA/cm ² Cte Votage=3.5V |

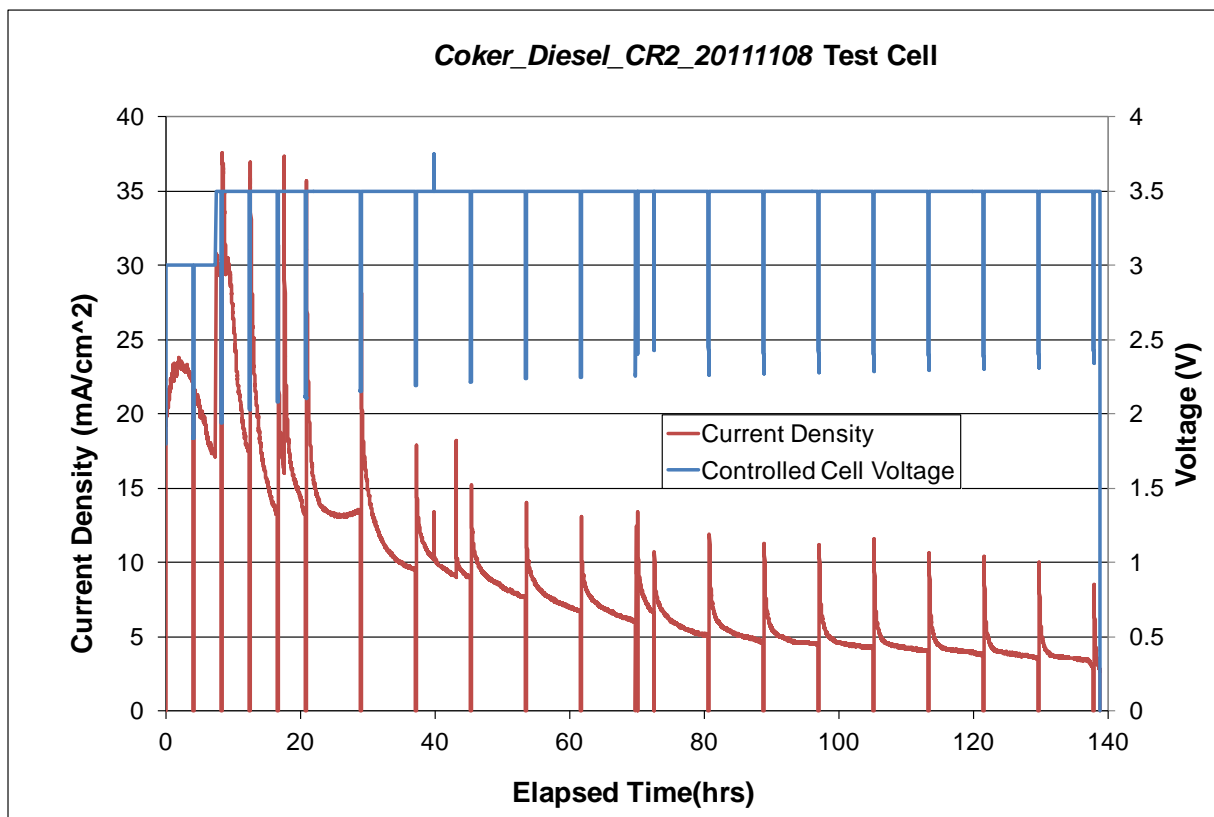


Figure 4: Cell voltage and current density versus elapsed time for sodium recovery test cell Coker_Diesel_CR2_20111108

Task 7.0 – Analysis

Subtask 7.1 – Develop electrolysis process model

Ceramatec shall analyze data from Task 3 and a performance model will be developed. Various factors such as membrane thickness, type of alkali metal, electrode configuration and cell design would be included in the model considerations.

Subtask 7.2 – Preliminary cost analysis

The Recipient shall incorporate the models from Subtasks 4.1 and 4.2 into a preliminary cost model. Based on the preliminary cost analysis, a selection will be made between sodium and lithium as the most promising alkali metal for further pursuit in Phase 2. Sodium was selected because of higher efficiency of removing sulfur and lower cell voltage in electrolysis compared to Lithium.

3. CONCLUSION

Our conclusion at this point is that high levels of both sulfur and nitrogen can be removed from shale oil, heavy oil, coker diesel and bitumen with the process tested. Both methane and hydrogen are effective in removal of sulfur, nitrogen, heavy metals, and increasing API gravity.

4. COST STATUS

The projected costs stated in the Project Management Plan and the monthly costs of the 9th quarter are shown in Table 12, along with the projected costs stated in the Project Management Plan.

Table 8: Project costing profile for the 9th Quarter

| Note: Benefits, Overhead, & G&A rates char | Oct-11 | | Nov-11 | | Dec-11 | | Q9 | | |
|--|-----------|-----------|------------|-----------|------------|-----------|------------|------------|------------|
| | Projected | Actual | Projected | Actual | Projected | Actual | Projected | Actual | |
| Direct Labor | | 31,996.53 | | 28,543.54 | | 29,094.80 | | 89,634.87 | |
| Benefits | 29.76% | 9,522.17 | | 8,494.56 | | 8,658.61 | | 26,675.34 | |
| Overhead | 43.71% | 13,985.68 | | 12,476.38 | | 12,717.34 | | 39,179.40 | |
| Total Burdened Labor | | 55,504.38 | | 49,514.48 | | 50,470.75 | | 155,489.61 | |
| Direct Materials / Spec Test | | 7,013.98 | | 15,124.03 | | 14,900.75 | | 37,038.76 | |
| Equipment | | | | | | - | | - | |
| Consulting | | 16,864.08 | | 9,885.84 | | 13,374.96 | | | |
| Travel | | 119.00 | | 4,737.06 | | 1,915.00 | | 6,771.06 | |
| Subtotal | | 79,501.44 | | 79,261.41 | | 80,661.46 | | 239,424.31 | |
| G&A | 31.44% | 24,995.25 | | 24,919.78 | | 25,359.96 | | 75,274.99 | |
| Total monthly | | - | 104,496.69 | - | 104,181.19 | - | 106,021.42 | - | 314,699.30 |

Figure 5 shows a plot of the total monthly costs and the initially projected costs versus time and Figure 6 shows the cumulative monthly costs versus time. Also shown in the figure is the fraction of actual over planned cumulative expenses.

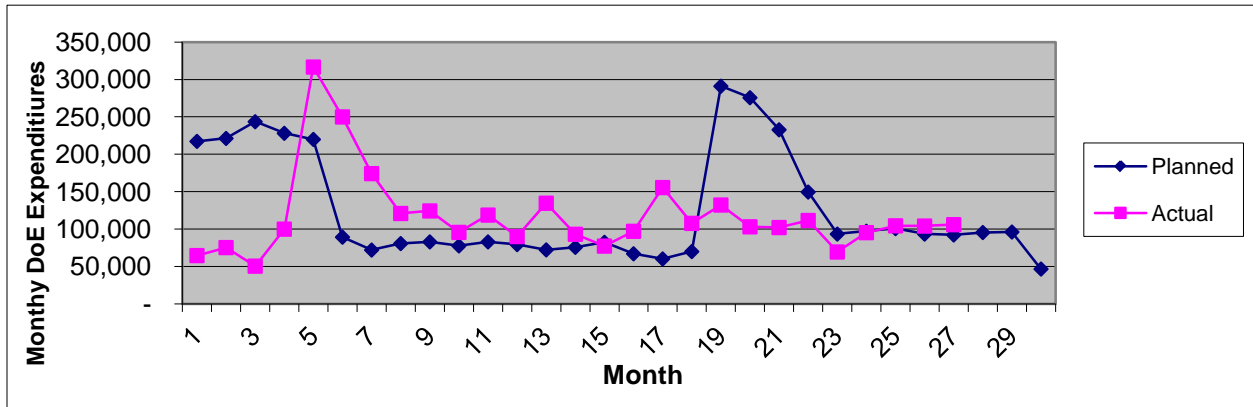


Figure 5: Projected and actual monthly costs over time

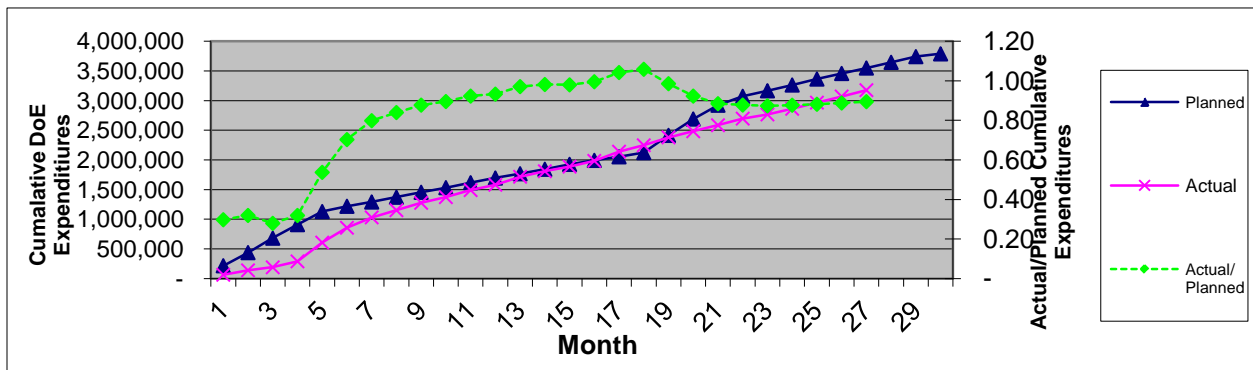


Figure 6: Projected and actual cumulative costs over time

5. MILESTONE STATUS

New milestones need to be set for the current Budget Period.

Table 9: Milestone log for 6rd Quarter

| Milestone No. | Task / Sub-task | Project Milestone Description | Planned Start Date: | Planned End Date: | Actual Start Date: | Actual End Date: | Comments |
|---------------|-----------------|---------------------------------|---------------------|-------------------|--------------------|------------------|---|
| 1 | 1 | Updated PMP | 9/29/09 | 10/29/09 | 9/29/09 | 10/26/09 | |
| 2 | 2.1 | Analytic capability established | 9/29/09 | 3/1/10 | 9/29/09 | 3/23/10 | Analytical capability has been established as stated in the PMP. Operators have been trained on GC. ICP and CHNS are operational |
| 3 | 2.2 | Complete upgrading exp. Setup | 9/29/09 | 3/29/09 | 9/29/09 | 3/26/10 | Upgrading set-up has been completed including HAZOP and pre-start up safety review. The reactor set up has been ready to be operational as of Friday, March 26, 2010. |
| 4 | 2.3 | Complete process runs | 3/30/10 | 1/3/11 | 3/26/10 | 3/31/11 | Process runs underway |
| 5 | 3.1.1 | Complete membranes for Phase 1 | 9/29/09 | 7/5/10 | 9/29/09 | 9/20/10 | Membrane fabrication has exceeded demand for fabrication. Mechanical characterization was complete on September 20, 2010. |
| 6 | 3.3 | Cells ready for operation | 4/13/10 | 2/28/11 | 4/13/10 | 3/31/11 | Cells were ready for operation on time. Initial cells testing began running 4/26/10 when sufficient sodium polysulfide was synthesized. |
| 7 | 4.3 | Preliminary cost model complete | 2/8/11 | 3/14/11 | 1/4/11 | 3/18/11 | A preliminary cost model was completed and reviewed internally. Adjustments were recommended by the reviewers and additional cases suggested for updated cost models. |

6. ACCOMPLISHMENTS

- Electrolysis of sodium sulfide at temperatures of molten sodium have begun and are showing very encouraging results.
- Reactor tests with methane have continued with encouraging results
- Process models of both the reactor and electrolysis processes have begun which will lead to the preliminary cost model.
- Additional input and output measurement has improved the accuracy of mass balances
- Electrolysis of Na₂S₄ has exceeded 5000 hours at the target current density and temperature.
- **The technology has been demonstrated on multiple feedstocks of different origins: Shale Oil, Heavy Oil, Bitumen (Oil Sands), and Coker Diesel**

7. PROBLEMS OR DELAYS

No problems to report at this time.

8. PRODUCTS

No products to report at this time.

9. LIST OF APPENDICES

None

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