DEVELOPMENT AND VALIDATION OF AN ACID MINE DRAINAGE TREATMENT PROCESS FOR SOURCE WATER

Final Technical Report

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PRINCIPAL INVESTIGATOR

Ann Lane, Battelle
614-424-3266
lanea@battelle.org

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Battelle Memorial Institute
505 King Ave
Columbus, OH 43201

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Abstract

Throughout Northern Appalachia and surrounding regions, hundreds of abandoned mine sites exist which frequently are the source of Acid Mine Drainage (AMD). AMD typically contains metal ions in solution with sulfate ions which have been leached from the mine. These large volumes of water, if treated to a minimum standard, may be of use in Hydraulic Fracturing (HF) or other industrial processes. This project's focus is to evaluate an AMD water treatment technology for the purpose of providing treated AMD as an alternative source of water for HF operations. The HydroFlex™ technology allows the conversion of a previous environmental liability into an asset while reducing stress on potable water sources. The technology achieves greater than 95% water recovery, while removing sulfate to concentrations below 100 mg/L and common metals (e.g., iron and aluminum) below 1 mg/L. The project is intended to demonstrate the capability of the process to provide AMD as alternative source water for HF operations.

The second budget period of the project has been completed during which Battelle conducted two individual test campaigns in the field. The first test campaign demonstrated the ability of the HydroFlex system to remove sulfate to levels below 100 mg/L, meeting the requirements indicated by industry stakeholders for use of the treated AMD as source water. The second test campaign consisted of a series of focused confirmatory tests aimed at gathering additional data to refine the economic projections for the process. Throughout the project, regular communications were held with a group of project stakeholders to ensure alignment of the project objectives with industry requirements. Finally, the process byproduct generated by the HydroFlex process was evaluated for the treatment of produced water against commercial treatment chemicals. It was found that the process byproduct achieved similar results for produced water treatment as the chemicals currently in use. Further, the process byproduct demonstrated better settling characteristics in bench scale testing.

The field testing conducted in the second project budget period demonstrated the ability of the HydroFlex technology to meet industry requirements for AMD water chemical composition so that it can be used as source water in HF activities. System and operational improvements were identified in an additional series of confirmatory tests to achieve competitive cost targets. Finally, the application of the HydroFlex process byproduct in produced water treatment was demonstrated, further supporting the commercial implementation of the technology. Overall, the project results demonstrate a path to the economic treatment of AMD to support its increased use as source water in HF, particularly in regions with limited local freshwater availability.

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1.0 Executive Summary

Hydraulic Fracturing (HF) is becoming an increasingly common method of unconventional resource extraction allowing access to previously uneconomical natural gas reserves. This has contributed to increasing utilization and potential strain on local freshwater sources due to the large volume of water required during the fracturing process. This project's focus is to evaluate an Acid Mine Drainage (AMD) water treatment technology for the purpose of providing treated AMD as an alternative source of water for HF operations. The HydroFlex™ technology allows the conversion of a previous environmental liability into an asset while reducing stress on potable water sources. The technology achieves greater than 95% water recovery, while removing sulfate to concentrations below 100 mg/L and common metals (e.g., iron and aluminum) below 1 mg/L. This project demonstrated the capability of the process to provide AMD as alternative source water for HF operations.

During the second budget period both a structured test campaign, Test Campaign 1 (TC1), as well as shorter, discrete confirmatory tests, Confirmatory Test Campaign (CTC), were performed using the field demonstration HydroFlex system. Data collected as part of the field trials was used to project costs for operation of the system. During TC1, several extractant to aqueous (E:A) ratios were evaluated to determine which would remove sulfate most effectively. It was determined that a 1:2 E:A ratio provided the best sulfate removal. Therefore, this ratio was used during long duration, steady state operation to evaluate the long term ability of the HydroFlex system to remove sulfate. A summary of the sulfate removal capabilities at this E:A ratio is shown in Figure 1. Using a single extraction stage, sulfate levels in the AMD were reduced to less than 150 mg/L from an average influent concentration of approximately 550 mg/L. When a second extraction stage was brought online at 120 hours, the sulfate levels in the AMD were reduced to below 100 mg/L.

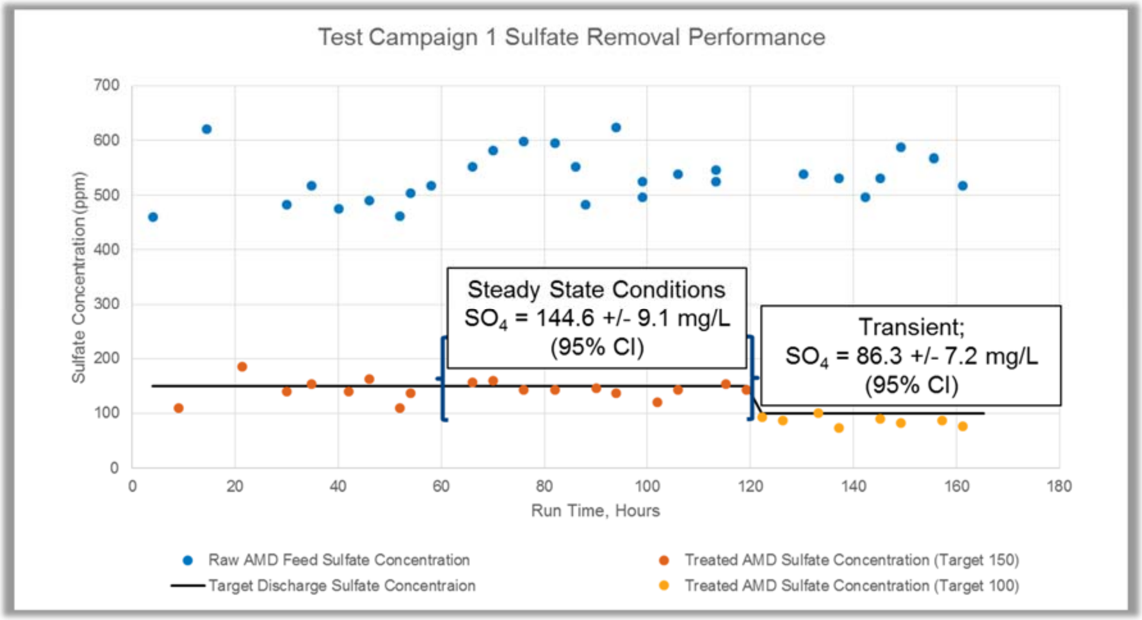

Figure 1. TC1 sulfate removal performance of the HydroFlex system.

The CTC was conducted after the conclusion of TC1 to further evaluate the primary system economic drivers. Process chemical feed rates, filtration requirements, and chemical makeup procedures were identified to be primary system cost drivers and were tested to determine if operational cost savings could be realized. It was found that system operational costs were able

to be reduced approximately 50% during the CTC when compared to operating costs during TC1. Further operational cost projections from the CTC results indicate that an additional 30% reduction may be realized through additional process modifications.

As part of the BP2 activities, regular project updates were provided to the project stakeholder committee. The results of the test campaigns were shared with the project stakeholders, who confirmed that reducing sulfate levels to less than 100 mg/L achieves the requirements for use of treated AMD as a source water.

Byproducts which were generated as part of field testing were characterized and evaluated against industrial chemicals used in the treatment of produced water. It was found that the sodium sulfate solution which the HydroFlex process produces is effective at treating produced water for the removal of barium and strontium, which are constituents of concern when recycling or disposing of produced water. While both the HydroFlex process byproduct and the industrial softening solution provided adequate reduction of barium and strontium levels in produced water samples, the HydroFlex process byproduct reduced turbidity in the treated effluent samples substantially when compared against the commercially available solution.

Results of the comparative testing conducted were shared with companies currently providing treatment for produced water to determine the interest in using the HydroFlex byproduct as a softening agent for removal of barium. It is believed that the increased settling rate will translate into smaller settling tanks required for the treatment of produced water.

The field testing conducted in the second project budget period demonstrated the ability of the HydroFlex technology to treat AMD to meet industry requirements for source water in HF activities. System and operational improvements were identified during the confirmatory test campaign to achieve competitive cost targets. Finally, the application of the HydroFlex process byproduct in produced water treatment was evaluated, further supporting the commercial implementation of the technology by validating an outlet for the process byproduct stream. Overall, the project results demonstrate a path to the economic treatment of AMD to support its use as source water in HF, particularly in regions with limited local freshwater availability and proximity of HF operations to AMD sources.

2.0 Project Background

The National Petroleum Council estimates the volume of recoverable gas from tight sands, gas shales, and coal bed methane within the United States is in excess of 25 trillion cubic meters (900 trillion cubic feet) (National Petroleum Council, 2011). The United States, on average, consumes 0.6 trillion cubic meters (22 trillion cubic feet) of natural gas per year, substantiating the largely untapped resource as a critical contributor to the nation’s energy portfolio (National Petroleum Council, 2011). A method employed and improved upon for more than 60 years to access the gas from the underground formations is hydraulic fracturing (HF). HF is a well-stimulation process that aims to maximize the extraction of underground resources, including oil and natural gas, by injecting large volumes (on the order of millions of liters) of pressurized water with a mixture of chemicals and proppant into underground rock formations to create pathways for the natural gas and oil to migrate from the rock pores to the surface.

An estimated 117 billion liters (31 billion gallons) of freshwater was used from 2011 to 2013 for HF among eight major shale plays, as shown in Figure 2 (Lux Research, 2012). In 2013, the hydraulic fracturing industry was projected to double its freshwater usage over the next 3 years, Source: Lux Research,“Risk and Reward in the Frac Water Market”, Mar 2012

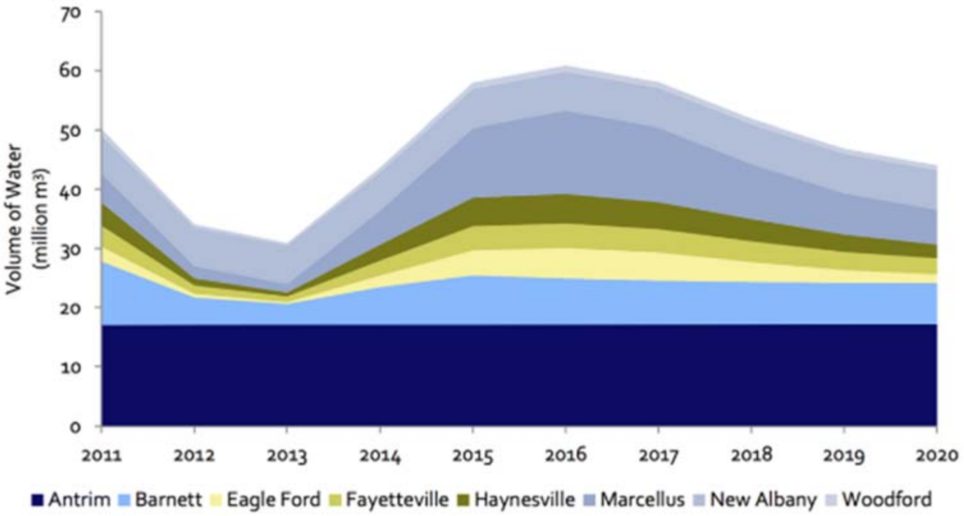

Figure 2. The yearly volume of water injected during hydraulic fracturing at the eight major shale plays was previously projected to exceed the volume used in 2013 each year through 2020.

and, if the current freshwater sources continue to be used, the strain on public water supplies may negatively impact the price and availability of natural gas and oil for residential and industrial consumers. The Department of Energy (DOE) has a goal to advance the environmentally sound development of unconventional, domestic natural gas and oil resources that is dependent on identifying novel technologies that reduce water usage and resource degradation through less freshwater-intensive approaches.

Acid mine drainage (AMD) is an abundant source of marginal water that is frequently disregarded as a water source for hydraulic fracturing. As the US looks to the future of energy with unconventional resource development, the legacy of pollution from past resource extraction is noted with AMD. AMD is formed by a natural process that generates sulfates and metals in solution in almost any case where large volumes of earth or rock are disturbed. It is especially prevalent in areas of coal and mineral mining, and the highly contaminated drainage can continue for centuries after mining production is discontinued. In Pennsylvania alone AMD affects more than 8,800 kilometers (5,500 miles) of waterways (Cavazza & Cavazza, 2011). AMD’s main components, iron and sulfate, are not suitable for fracing water due to downhole considerations; however, under the right conditions, iron is a powerful water treatment chemical, and sulfate an effective precipitant for scale- causing metals.

Current AMD remediation technologies are not able to provide water that meets the quality standards desired by the oil and gas industry. Limestone neutralization (i.e., liming) is the most common method currently employed for treating AMD water; however, it does not meet the stringent HF water standards for sulfate concentration. Liming is dependent on the water solubility of calcium sulfate and is frequently unable to remove sulfates below the level of 1,000 (mg/L). Nanofiltration with liming in succession helps to improve the water quality, but adds a level of complexity and is not a “one solution fits all” approach that can meet the sulfate discharge level requirement of less than 250 mg/L. Another drawback of liming is that it generates large volumes of high-density waste that may require costly disposal.

The HydroFlex process is a platform technology that is capable of meeting the sulfate discharge requirements of <250 mg/L (and potentially lower concentrations) and common metals to levels below surface discharge requirements. It recovers greater than 95 percent of the incoming water, produces byproducts of potential use to the industry rather than a waste sludge, and operates with modified commercial off-the-shelf (COTS) equipment.

The HydroFlex process for the remediation of AMD has been tested in the lab and demonstrated successfully in the field at St. Michael, PA. The previous laboratory and field testing confirmed the consistent removal of aluminum, iron, and sulfate from AMD source water for a range of starting concentrations. These demonstration efforts were targeted solely for AMD remediation for discharge. The work conducted under this project is focused on adaptation of the process to provide water and byproducts useful for the unconventional resource development industry. This DOE program supports the optimization and large scale demonstration of the HydroFlex technology for treatment of AMD water for use in hydraulic fracturing.

3.0 Project Objectives and Approach

The objectives of this project are: (1) to develop an innovative treatment process to support the use of AMD water for HF, (2) to evaluate the use of the process byproducts in produced water treatment processes, and (3) to determine the environmental, regulatory and commercial implications of using treated AMD as source water in HF.

These objectives were met through a multi-task technical approach conducted in two budget periods. As part of the activities of the first budget period, laboratory testing was conducted to determine process operating parameters, and field shakedown testing was conducted by Battelle's subcontractor, Winner Water Services (Winner). During the second budget period the field demonstration HydroFlex system was operated in support of both a structured test campaign as well as shorter, discrete confirmatory tests aimed at supporting specific process development objectives. Data collected as part of the field trials was used to project costs associated with operation of the system, providing estimated costs for the treatment of AMD for source water use using the HydroFlex technology.

The byproduct generated during field testing were characterized and evaluated against industrial chemicals used in the treatment of produced water. Use of the byproduct solution in other oil and gas applications reduces the cost for operation of the system by avoiding waste disposal costs and potentially providing an additional revenue stream. Results of the comparative testing conducted were shared with companies currently operating produced water treatment facilities to determine their interest in using the HydroFlex byproduct as a softening agent for removal of barium.

As part of BP2 activities, regular project updates were provided to the project stakeholder committee. Throughout the project, the committee provided valuable information regarding required water quality characteristics for source water as well as acceptable economics for water treatment. Additionally, stakeholders were able to share challenges that the industry is currently struggling with regarding use of AMD as source water in HF operations.

4.0 Technical Tasks

4.1 Task 1.0: Project Management and Planning

Project management activities were conducted throughout the second budget period (BP2). Battelle managed the project as stated in the Project Management Plan (PMP) which was updated whenever a significant change was made to the project. All work conducted under BP2's

Tasks 3 and 4 was planned and managed through the project management task, including the field work and reporting completed by Winner Water Services (Winner). Throughout BP2, project scope and budget was managed against the PMP.

Due to concerns with the system's ability to successfully execute a second test campaign, a GO/NO GO decision point was added between the first and second planned test campaigns to ensure that the data collected during the first test campaign supported the need for a second test campaign. The SOPO and PMP were updated to reflect these changes. At the conclusion of the first test campaign, a NO GO decision was made regarding execution of the second test campaign. Instead of a second test campaign, the remaining project funding was used to complete the originally proposed tasks associated with byproduct testing and stakeholder outreach. In addition, a series of discrete confirmatory field tests were conducted to further refine the system operating cost projections. The report submitted as part of the GO/NO GO decision point included the Field Testing Report for the first test campaign and replaced this deliverable as per the SOPO.

4.2 Task 3.1: AMD Treatment Process Testing and Evaluation-Test Plan Development

A test plan was created for the first field test campaign using data from the lab and shakedown testing performed in Budget Period 1. The test plan is included as Appendix B. Test campaign one (TC1) consisted of two parts; the first part encompassed initial testing of extractant to AMD (E:A) ratios to select the ratio most likely to achieve sulfate removal goals. The second portion included a steady-state run performed at the E:A ratio selected during the range-finding portion of TC1.

Sulfate removal results from the continuous lab testing in Phase 1 were used to estimate the sampling requirements for the E:A range finding tests. It was determined that the data are not likely to be normally distributed, so the data was treated as nonparametric. Using two different standard deviations, one a pooled value and the other a worst case estimate, power calculations were performed assuming a nonparametric test for a difference in medians of two populations. In Figure 3, the estimated number of samples per E:A to detect a difference in medians between two E:A groups is shown. Based on this graph, it was decided that 5 samples would be collected and run at each E:A to detect a difference of 50 mg/L achieved in the sulfate concentration between test conditions.

Additionally, during the process shakedown testing of Phase 1, sulfate samples were collected from the extraction stage. The number of samples needed to provide a confident assessment of steady state sulfate removal during the second portion of TC1 was predicted using the variability in the sulfate values from Phase 1. In Table 1 the estimated 95% confidence interval precision is provided by sample size. It should be noted that in both these cases, the actual confidence interval width and test power changed according to the variability actually realized in the samples collected.

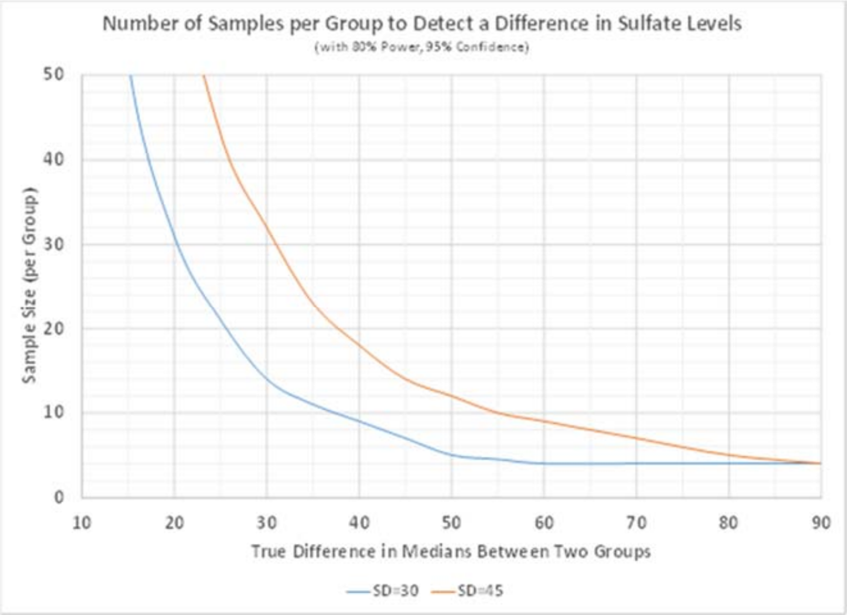

Figure 3. This graph shows the estimated number of samples required to detect a difference in medians between two E:A test cases during the first portion of test campaign one.

Table 1. The estimated 95% confidence interval precision by sample size is shown.

Sample Size	Confidence Interval Precision
3	Mean +/- 147
4	Mean +/- 43
5	Mean +/- 28
10	Mean +/- 15
25	Mean +/- 8
50	Mean +/- 6
100	Mean +/- 4

The number of samples required to draw statistical conclusions from the test data was used to inform the number of tests and required test length of the E:A ratio screening test runs, as well as the overall duration and sampling frequency of the steady state portion of TC1.

4.3 Task 3.2: AMD Treatment Process Testing and Evaluation-Process Testing

4.3.1 EXTRACTANT EVALUATION

Prior to the start of the first test campaign in BP2, additional laboratory and field testing was performed to evaluate the extractant material on the field system and identify any adjustments needed to the process operation based on the extractant's anticipated performance. The primary concern behind these tests were losses of the organic extractant observed in the Phase 1 testing due to entrainment of the extractant in the treated AMD water. Because organic loss has a significant impact on the process economics, several tests were run to reduce organic losses from the system. These tests identified changes to the process chemistry, unit operations operating parameters, and maintenance procedures. More detailed results of the testing performed are included in Appendix A.

First, the type and quantity of extractant material that was not recovered during the operation of the process in BP1 was estimated. Next, the impact of various process changes on the amount of unrecovered material was evaluated, and finally operational changes were made to capture the extractant for recovery and return to the system. The following sections outline the testing performed to quantify and reduce the loss of entrained extractant.

QUANTIFICATION OF LOSSES

In the laboratory testing conducted as part of BP1, total organic carbon (TOC) tests were used to estimate the amount of extractant carry over observed during testing. Operational parameters, such as mixing speed, were configured to minimize TOC readings in the process discharge, thus maximizing phase disengagement and recovery of extractant material. Similarly during the field shakedown testing, TOC tests allowed quick characterization of the level of entrained extractant. However, TOC tests do not measure the concentrations of individual constituents in the extractant mixture, rather they determine the bulk organic concentration.

To identify which process changes would best reduce extractant carry over during B2 testing, information regarding the carry-over of individual extractant components was needed. Therefore, additional analysis of organics both in the aqueous phase as well as bulk analysis of the extractant remaining on the system was conducted. In both cases, Battelle ran samples taken from the field system using 2-dimensional gas chromatography with time-of-flight mass spectral detection. Samples of treated AMD were pulled immediately after the final extraction stage (upstream of GAC beds), as well as at the overall process discharge point (downstream of GAC beds). Response factors for the extractant components were determined by analyzing neat samples of each, and blank and spiked samples were also run. For analysis of components in the aqueous phase, the sample was first extracted with dichloromethane to concentrate the target compounds.

The extractant mixture is composed of three components in a predetermined ratio; the components include the active ingredient, the diluent and a modifier to improve phase disengagement. At the beginning of TC1, the bulk extractant was analyzed and compared to virgin extractant to determine how the composition had changed during the previous year of operation, using the method described above.

This analysis indicated that concentration of the active ingredient had decreased by approximately 20% with relation to the diluent and modifier components. This indicates that organic losses tend to favor the active ingredient rather than being an entrained mixture of composition similar to the bulk extractant. Figure 4 shows a 3-dimensional plot representing the spectra obtained in the two dimensional GC work. The active ingredient is a mixture of amines, which tend to elute from the GC column later due to their ionic nature. It should be noted that the alcohol modifier used in the extractant could not be separately distinguished from the hydrocarbons in the spectra. Additionally, recovery of hydrocarbons was low, at around 4%, versus an 89% recovery for the amine components.

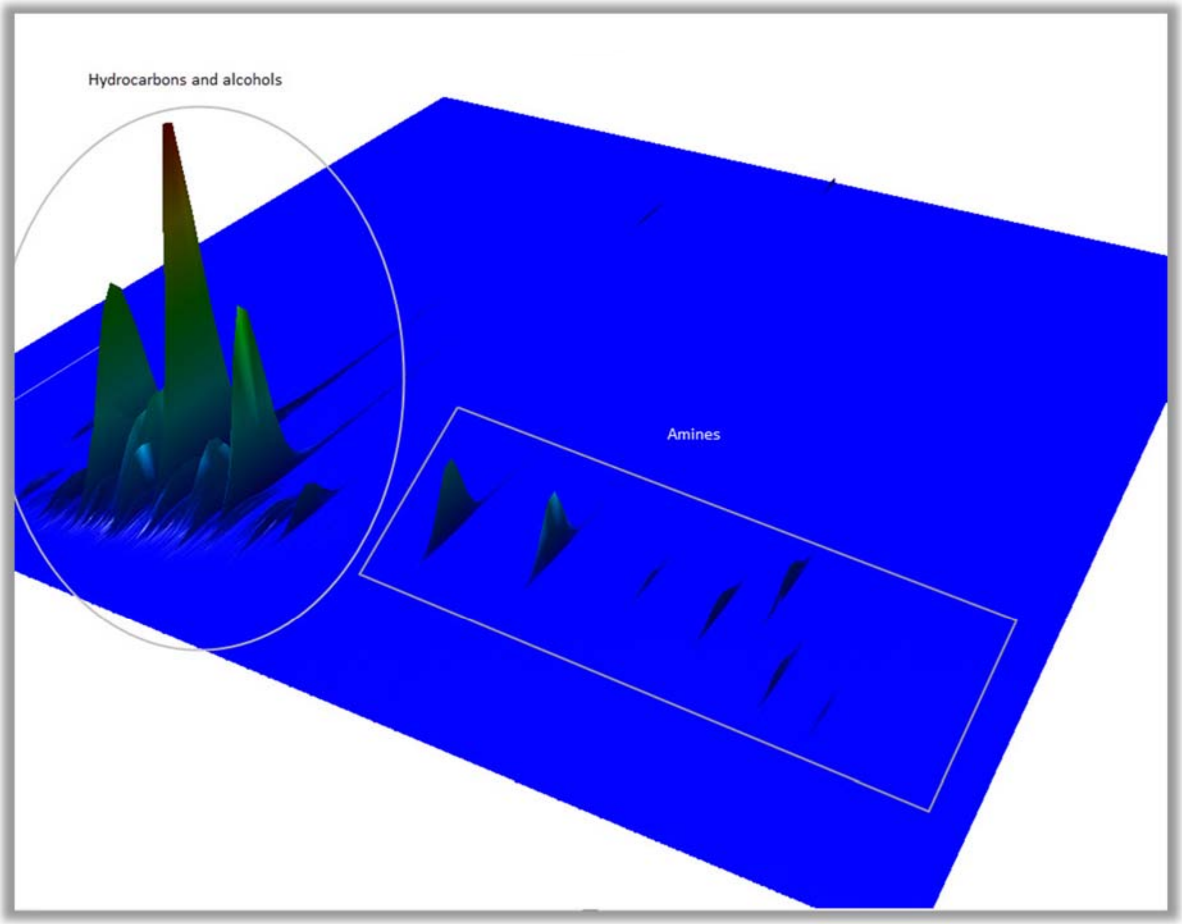

Figure 4. 3 dimensional plot of 2 dimensional GC spectra of the extractant used for the testing.

Analysis of the organic components extracted from the process water using dichloromethane also indicated an enrichment of active ingredient in the aqueous phase. The observed concentrations were adjusted by the recovery observed in the positive control, and showed that the concentration of active ingredient in the aqueous phase relative to the modifier and diluent is higher than in the bulk extractant (see Appendix A). Another finding from the GC work was that the concentration of extractant components was slightly higher after the activated charcoal filter than before, indicating that the extractant had built up in the charcoal beds, blinding the media and resulting in bypass of the media. A change in the maintenance procedures for the charcoal beds to incorporate more frequent backwashing was incorporated to prevent bypass of the filters by the extractant components. More detailed results of the GC work are provided in Appendix A.

PROCESS CHANGES

Several process changes were investigated to prevent extractant loss in the HydroFlex system, including changing the extractant formulation, filtration/centrifugation of the treated AMD effluent, adjustment of the pH of the extraction step, addition of iron coagulant, extraction of residual extractant using individual extractant components, use of bicarbonate instead of carbonate to regenerate the extractant, and ion exchange for polishing of the AMD effluent.

Extractant formulation was investigated during Phase 1 testing; however, some of the higher ratios of modifier to extractant were not previously tested. These values were examined to see if a more beneficial effect could be realized. In this testing, the linear relationship between extractant:modifier ratio and residual TOC in the AMD was consistent with that observed previously, as shown in Figure 5. Since adjustment of the extractant:modifier ratio was determined to provide only a limited benefit to the process economics, no change was made to the extractant formulation prior to the continuous run in TC1.

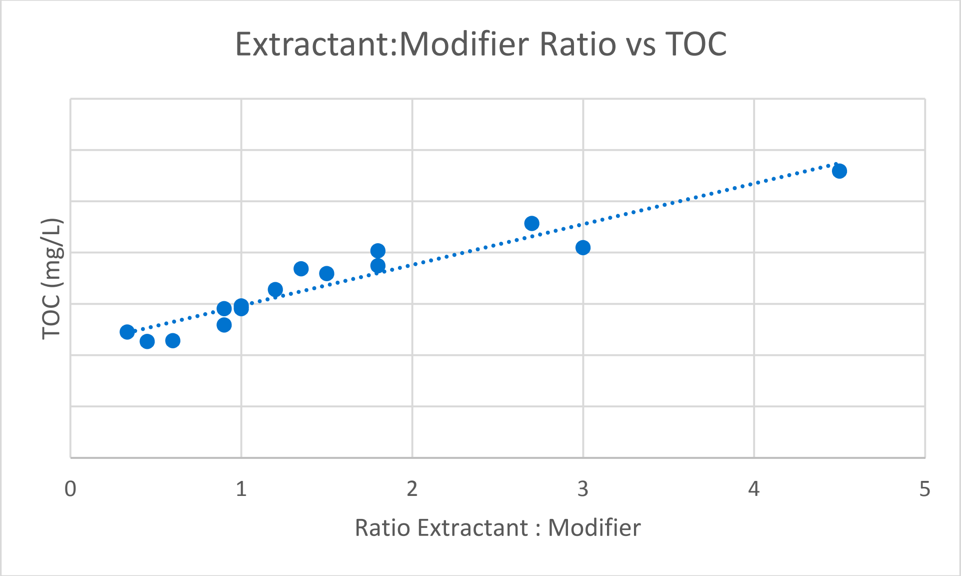

Figure 5. A linear correlation was observed between the ratio of active ingredient to modifier in the extractant to the residual TOC in the AMD water.

The pH was also adjusted in the extraction stages to lower levels in expectation that a lower pH would affect the surface chemistry of both the organic colloids and the calcium carbonate solids. There was no significant effect of pH on the residual TOC in the AMD water, and so pH adjustment was not investigated further or implemented in TC1. Results of the pH adjustment testing can be found in Appendix A.

As a possible process change filtration and centrifugation of the effluent water was tested. A polysulfone micro filter was used to filter the effluent to judge whether extractant was entrained preferentially in the calcium carbonate solids formed due to the high calcium content in the AMD source. Centrifugation was also employed to fully clarify the effluent. Analysis of the effluent for TOC pre and post centrifugation indicated that a portion of the organic material was associated with the calcium carbonate solids, and a portion was not removed through filtration or centrifugation, remaining in the treated effluent. These results helped to guide future evaluation of methods to reduce extractant entrainment, but were not implemented due to the relatively low concentration of extractant present in the recovered calcium carbonate solids.

An additional process change evaluated was the dosage of iron to assist in clarifying the effluent. The HydroFlex process was originally designed to remove iron from water streams by formation of an oxyhydroxide floc that is floated by the organic extractant. At correct iron doses and process conditions, the floc collects the entrained portion of extractant and floats it to the top of the sample. The floc would then require processing through the currently unused metal stripping section of the HydroFlex system, designed to separate the iron from the extractant, recovering the entrained extractant and producing a separate ferric sulfate solution.

This mechanism was tested for recovery of both the soluble and insoluble entrained extractant by addition of ferric sulfate pentahydrate solution. Figure 6 shows iron applied to processed AMD water in varying conditions, with the best performance seen in samples -11 and -13, where the floc is floated and the water is clarified.

Figure 6. Several tested conditions of iron addition for recovery of entrained extractant.

Although the iron was effective at recovering the entrained portion of extractant, the associated cost of dosing the iron and recovering it in the metal stripping stages outweighed the benefit of reclaiming the extractant. Accordingly, the approach of iron addition was omitted from further consideration.

Another method evaluated to recover extractant material associated with the treated AMD was to perform a second extraction step using another organic based extractant to pull the dilute amount of organic material out of the bulk treated AMD. Re-extraction of the entrained organic was tested using specific fractions of the extractant mixture. The expectation was for contact with the diluent to break any emulsions and pull the organic from the aqueous stream with a clean phase break. This diluent could then be reused in the process. However, testing indicated that this approach would not be feasible for this application, as it recovered only a small fraction of the extractant, and would be difficult to implement during the field test campaign.

Normally, the extractant used in the HydroFlex process is loaded in a carbonate form to exchange for and remove sulfates, but can also be loaded with other salts or bicarbonate. Bicarbonate was tested to determine whether it would improve phase disengagement in the extraction section versus carbonate-loaded extractant. A small improvement was observed in the entrainment with bicarbonate-loaded extractant. However, use of bicarbonate requires twice the stoichiometric dose to regenerate the extractant compared to carbonate, and additional testing would have been required to validate efficient stripping performance. The measured improvement in entrainment did not justify these expenses, so no changes were made to the form of salt loaded on the extractant.

Finally, ion exchange resins were tested for their efficiency in recovering soluble active ingredient from the extractant. The ion exchange testing considered four separate resins, ultimately settling upon a weak acid cationic resin due to its relatively high loading for extractant and economical regeneration. Initial screening tests of the resins were performed by mixing a gram of resin with 40 mL of HydroFlex effluent and comparing the TOC removal. Loading capacities for the resins were then found by making flow through columns to pass an excess of effluent over the bed. Effluent samples were collected at fixed intervals to calculate the TOC loading on the resins. Resin for the regeneration tests was also loaded in the flow through columns, then regenerated with sulfuric acid according to the engineering specification sheets for each resin. The TOC removal capability for each resin was then assessed after regeneration and compared to a virgin resin sample to judge the efficiency of the regeneration.

No resins appeared to be economical for this recovery when sulfuric acid is used as the regenerant due to high volumes of waste, but if hydrochloric acid is used, value could be realized from an ion exchange (IX) bed. The demonstration site was not permitted for hydrochloric acid, so this was not implemented in the testing, but can be considered to improve the process economics going forward.

Overall, most of the process changes evaluated did not produce a large enough reduction of the organic carry over to be economically applied to the field test unit while maintaining the project scope and schedule guidelines.

OPERATIONAL CHANGES

In addition to process changes, operational changes were investigated to determine their effect on reducing the quantity of organic carryover including changing the mixing parameters and the scheduled maintenance of the activated charcoal beds installed as a polishing step downstream of the HydroFlex process. Sufficient mixing to remove the sulfate from the AMD was achieved with the mixer off in the extraction stage, but this left very little room for adjustment. Options to improve the range of adjustability in the mixer were investigated. Additionally, as the process was operated, it was noted that the TOC levels in the effluent became higher after the granular activated charcoal (GAC) beds than before, so the maintenance schedule for the GAC was amended. Finally, additional residence time was added to the system to facilitate additional phase break time.

In BP1 testing, it was found that operation of the extraction stage without the mixer operating allowed for sufficient contact between the extractant and AMD to remove sulfates. The mixing in this case came from the turbulence of the two flows entering the false bottom in the mixer and passing by the vanes in the impeller. However, despite effective sulfate removal, this layout did not allow flexibility in adjusting the mixing as flows or sulfate levels changed. Accordingly, a simple modification to the mixing compartment was tested. This modification is shown schematically in Figure 7, where the feed was redirected through a two inch pipe which could be slid up and down inside a pre-existing three inch perforated pipe. A gasket at the bottom of the two inch pipe sealed the interior and directed the height at which extractant entered the mixing chamber, which in turn affected the level of mixing. When the pipe was at its lowest, the fluids experienced higher mixing, while the fluids experienced the least mixing at the highest pipe location.

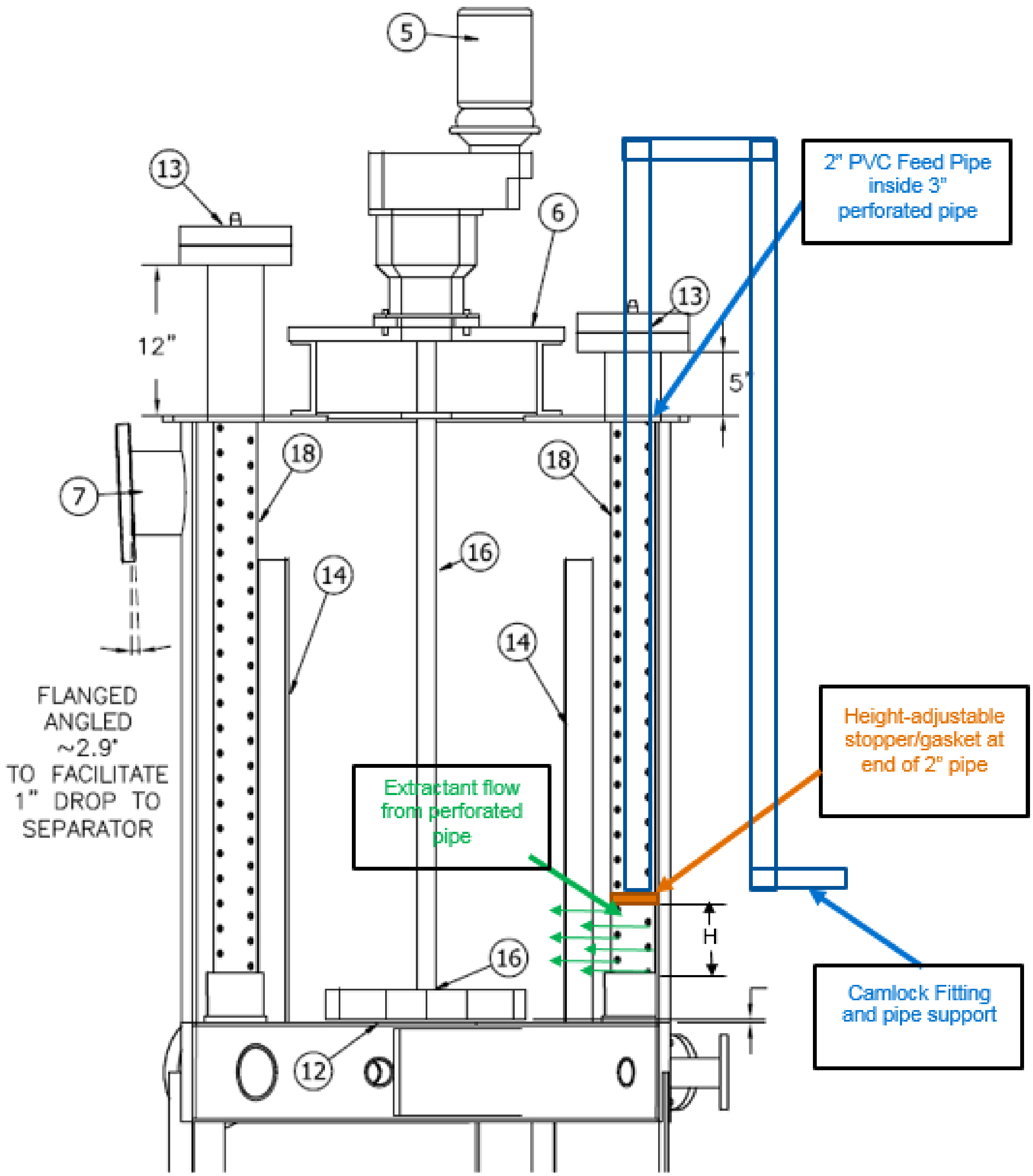

Figure 7. Conceptual diagram of the modification made to the mixing compartment during mixing tests.

With the modified mixing compartment, equivalent sulfate removal was achieved but with lower entrainment, as measured by turbidity in the sample as it overflowed from the mixer. However, the TOC measurements from the aqueous overflow after the settling section were not meaningfully affected by changes in the mixing compartment. Due to these results, the mixer was reverted to its original design with the mixer turned off.

The original design of the HydroFlex system included a GAC unit to ensure that volatile organic carbon (VOC) compounds were not released by the process. During regular VOC sampling around the GAC, it was discovered that the total organic carbon in the effluent increased across the carbon bed. Inspection revealed that a layer of organic had accumulated in the carbon vessels and was adding to the TOC in the effluent. To remedy this, backwashing of the GAC beds was added as a weekly maintenance item. Following this modification to the GAC maintenance procedures, no additional TOC was detected in samples of GAC effluent during the remainder of the field testing. Appendix A contains additional information of the operational changes made to the process.

4.3.2 TEST CAMPAIGN ONE: EVALUATION OF SULFATE REMOVAL CAPABILITIES

The first test campaign was started on August 3, 2015. The test campaign was comprised of two phases. During the first phase, the E:A ratio of the system was varied to determine its impact on sulfate removal. In the second phase, the system was run for three weeks under steady state conditions at the E:A ratio which resulted in the best sulfate removal. The average flow rate of AMD through the treatment system was 45 gallons per minute, driven primarily by the availability of the AMD blend on site.

E:A RATIO EVALUATION DISCUSSION

The system was operated at a single E:A ratio each day for the first week of operation. Sulfate samples were pulled after several hours of run time at each E:A ratio. Figure 8 shows the sulfate extraction performance observed at each E:A ratio. As expected, at higher E:A ratios, when the ratio of extractant to aqueous phases was increased, more sulfate was extracted. Analysis of variance indicated that there was significant difference between the test groups, and pairwise comparisons showed that all results were significantly different at 95% confidence.

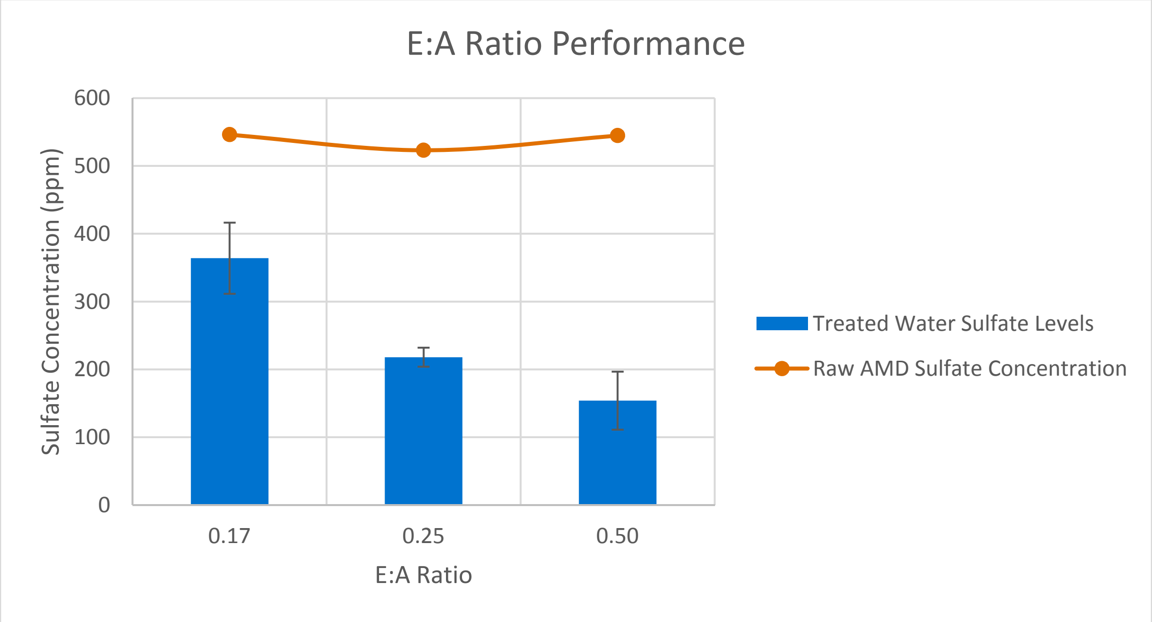

Figure 8. Comparison of E:A ratios evaluated during the first week of the test campaign. Error bars represent a 95% confidence interval.

In general, the lowest E:A ratio capable of providing the sulfate removal required is optimal, as a smaller overall volume of extractant is required to treat a given water source, resulting in lower capital and operating expenses. From the run data collected, it was determined that a 1:2 E:A ratio would be required to reduce sulfate to the required levels. This E:A ratio differs from the 1:10 E:A ratio previously used during field test runs conducted during the first budget period.

The original design for the process included a small makeup feed stream to address minor extractant losses. Due to the complexity of incorporating this makeup feed in daily operations, extractant makeup was not routinely performed during the field testing conducted prior to the first test campaign. It is believed that gradual degradation to the extractant health over a year of operations contributed to the need to run at a 1:2 E:A to achieve sulfate removal similar to that of the 1:10 E:A used when the extractant was fresh. Prior to the start of the second phase of TC1, additional extractant was added to the system to enhance the sulfate removal capabilities of the system; however, the extractant already loaded on the system was not able to be restored to its original strength observed during the two-30 hour demonstration runs conducted in BP1.

STEADY STATE RUN DISCUSSION

The system was operated for approximately 150 hours to collect performance data during steady state operation at the 1:2 E:A ratio. During the three weeks of run time, the process feed to the HydroFlex process was maintained, on average, between 500 and 600 mg/L sulfate. For the majority of the test campaign (approximately 120 operational hours), a single extraction stage achieved a sulfate level of below 150 mg/L. This equates to a sulfate removal of an average of 403 mg/L or a 74% reduction in sulfate content. Figure 9 and Figure 10 show the sulfate concentration of influent AMD stream and the process discharge stream.

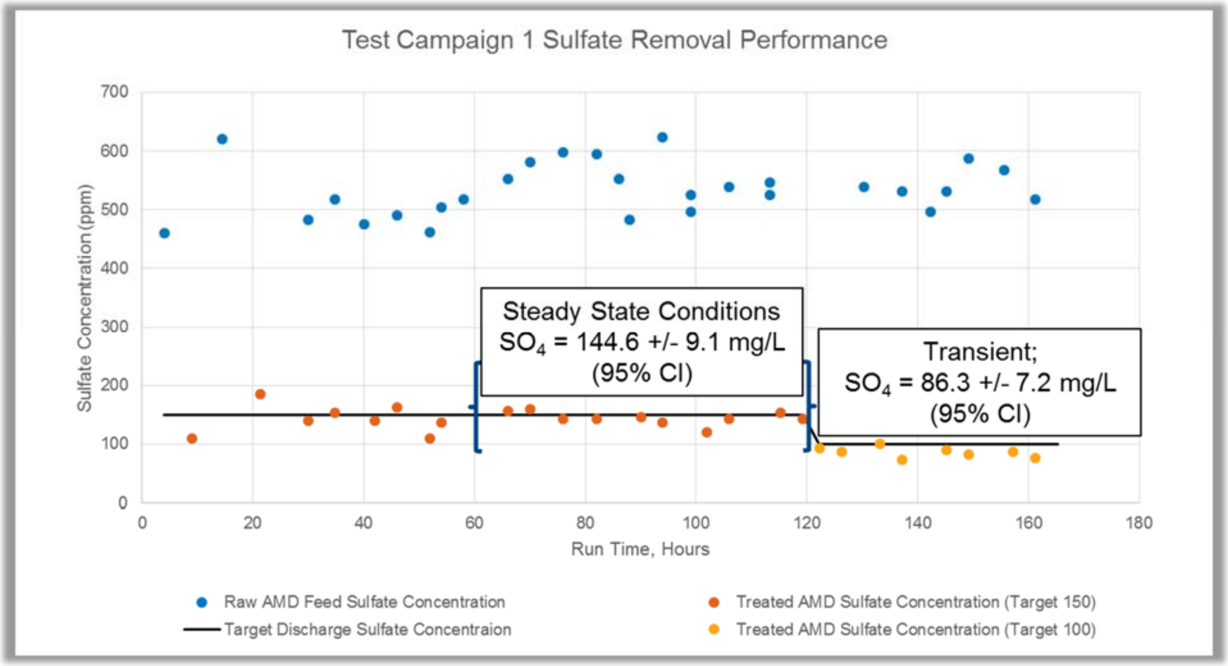

Figure 9. AMD feed sulfate concentration and treated water sulfate concentration for both 150 mg/L and 100 mg/L targets.

After approximately 120 hours of operation, a second extraction stage (E2) was brought online to provide additional sulfate removal capability, achieving sulfate levels below 100 mg/L per the guidance of our stakeholder committee. The impact of the second extraction stage is illustrated by the step change in the sulfate removal targets shown in Figure 9 and Figure 10. Under these conditions, sulfate was consistently reduced to levels below 100 mg/L, removing an average of 448 mg/L of sulfate or a reduction of sulfate by 85%.

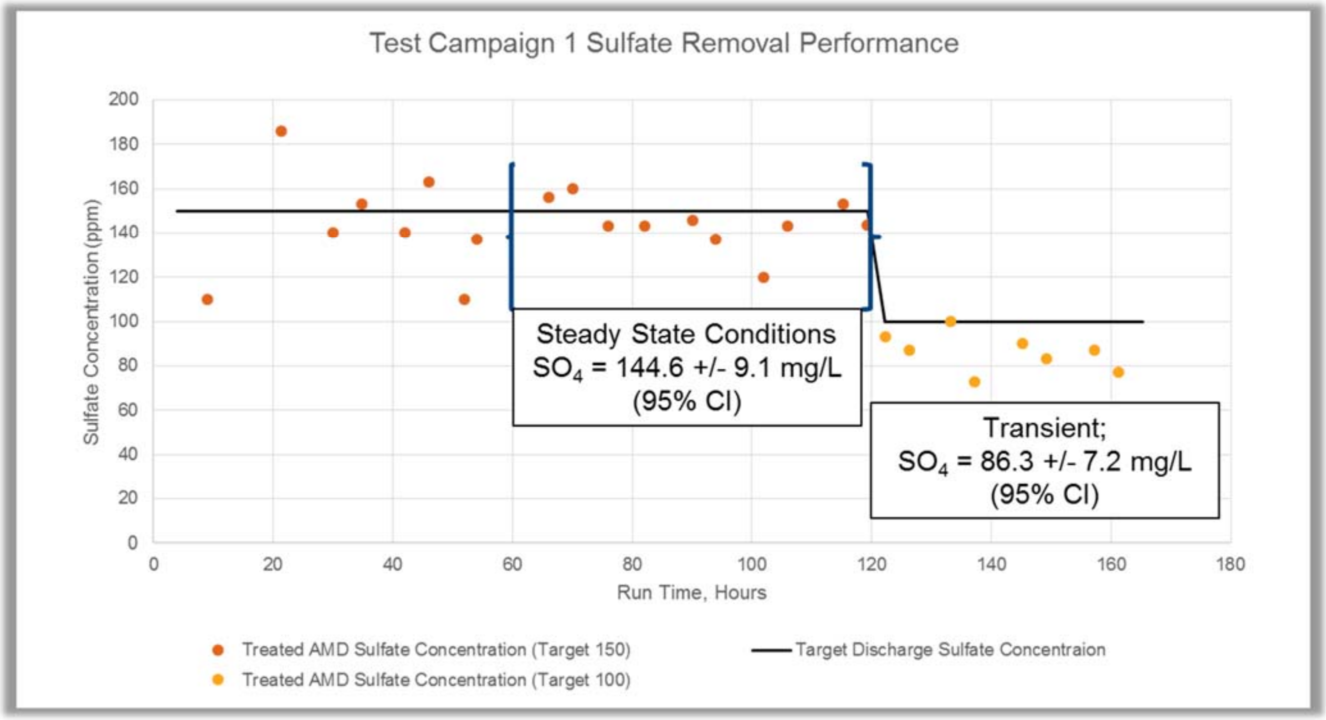

Figure 10. Zoomed view of Figure 2, showing only discharge water sulfate concentration.

PROCESS BYPRODUCT GENERATION

The HydroFlex process generates a process byproduct which is a mixture of sodium carbonate and sodium sulfate. A 6% sodium carbonate solution is fed to the S4 stage of the sulfate stripping stages to regenerate the extractant for reuse in the extraction stages. The stripping stages generate a byproduct which is concentrated and collected from the S1 stage. To preserve the process mass balance, the volume of byproduct dosed to the system is equivalent to the volume of carbonate dosed. Every 12 hours of operation, the sulfate concentration of the produced byproduct was sampled and recorded. The sulfate concentration of the generated byproduct, as well as the carbonate feed rate to the sulfate stripping stages is shown in Figure 11 below.

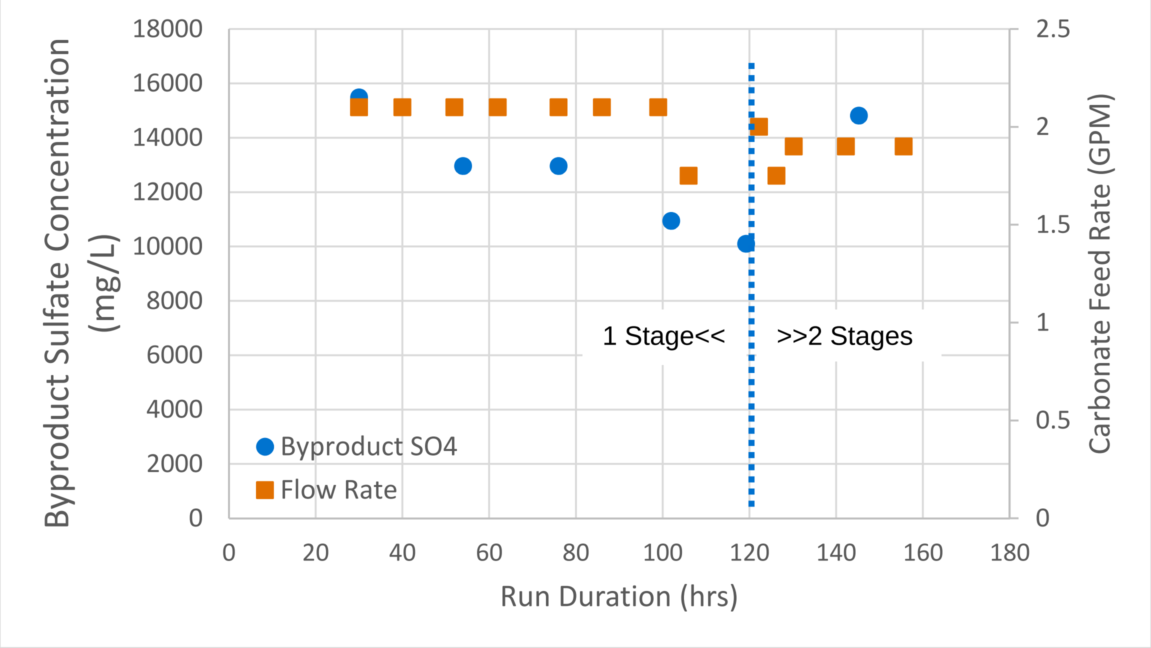

Figure 11. Byproduct sodium sulfate concentration and process sodium carbonate feed rate shown for steady state portion of TC1.

The volume of byproduct produced is equivalent to the volume of sodium carbonate fed to the process, and is directly proportional to the concentration of the contaminants being removed from the AMD source. Pumps and level controllers ensure that the flow rates of carbonate solution fed to the process and byproduct solution which leaves the process are equal. Throughout the test campaign, the concentration of the byproduct decreased, indicating that carbonate was dosed in excess during this phase of testing, diluting the sulfate concentration in the byproduct stream. This was intentional to maintain extractant performance at the highest possible level during TC1 to provide maximum sulfate extraction, so during TC1 sodium carbonate dosing rate was maintained at approximately 2.1 GPM throughout the test. TC2 was originally intended to identify optimal carbonate dosing rates for best possible process economics, but to evaluate the benefit of a lower carbonate feed in TC1, the sodium carbonate dosing rate was decreased to 1.9 GPM near the end of the second phase of TC1 after the second extraction stage was brought online. With the incorporation of the second extraction stage, the test results determined that the extraction performance was maintained at this lower carbonate feed rate.

EXTRACTANT CARRY OVER

Throughout the test campaign, TOC was measured at the E1 aqueous discharge to estimate the amount of net extractant losses (or carryover into the aqueous phase) during operation of the system. Minor extractant losses are expected, and would be compensated for in a production system by dosing a small volume of makeup extractant to the process on a continual basis. It should be noted that TOC measures all organic-based carbon sources in the water, so it can only be used as a comparative indicator of extractant losses rather than an accurate measurement.

During the E:A screening runs, TOC was measured at each E:A ratio to determine the potential impact of E:A ratio on extractant carry over. The results are shown in Figure 12.

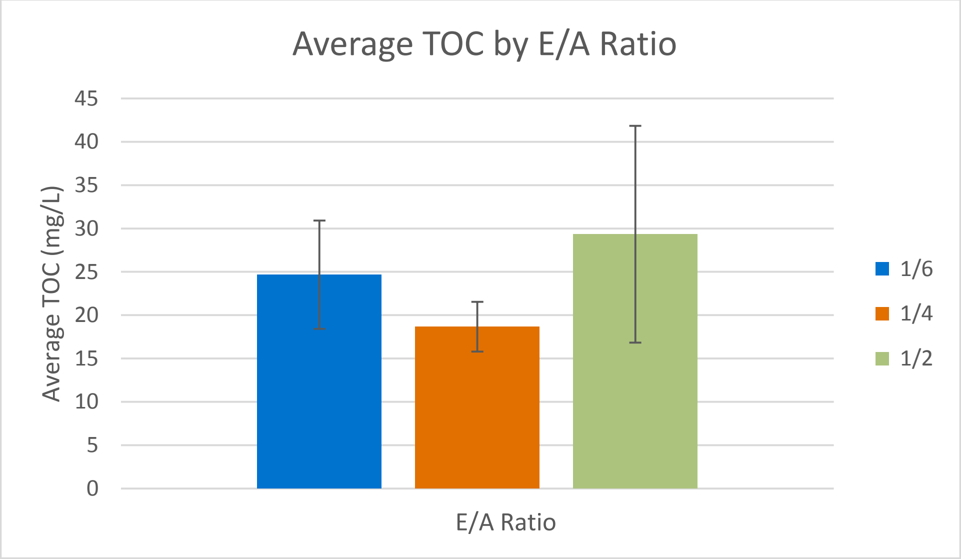

Figure 12. E:A ratio vs average TOC reading, error bars represent the 95% confidence interval.

It can be seen that the highest level of TOC observed occurs at the maximum E:A ratio tested (1:2). The highest amount of sulfate removal was also observed at this E:A ratio. During BP1 laboratory testing, it was also found that the higher E:A ratios exhibited greater TOC values, while simultaneously providing better sulfate removal. These data are aligned with projections, as it is believed that there is improved contact between the aqueous and extractant phases at higher E:A ratios. This allows for more efficient exchange of sulfate ions from the aqueous phase into the extractant phase, but also increases the potential for extractant to become entrained in the aqueous phase and be discharged from the system. Although the TOC is lowest at a 1:4 E:A, this reading is suspected to be within the error range of the measurement instruments. Based on this data and observations from previous laboratory testing, it is believed that TOC content generally increases as E:A ratio increases.

At the end of the first test campaign, the decision was made to increase the sulfate removal to provide treated AMD with less than 100 mg/L of sulfate. To achieve this, the process was adjusted to incorporate a second extraction stage. While the sulfate removal targets of less than 100 mg/L were met, a large spike in the TOC of the product water was observed. Figure 13 shows how the addition a second extraction stage approximately doubled the residual TOC in the treated AMD.

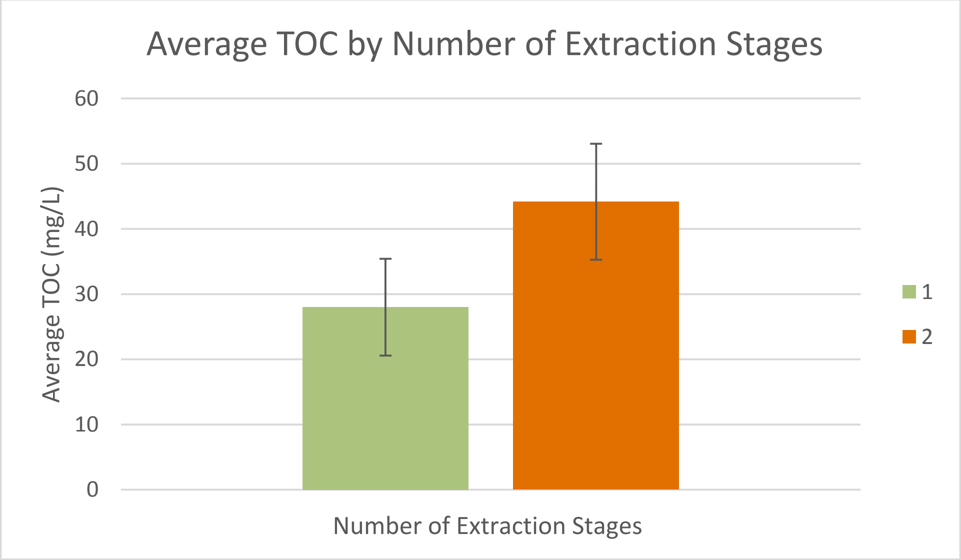

Figure 13. TOC vs Number of Extraction Stages.

It is therefore expected that the extractant losses of the system are elevated when operating a second extraction stage. This likely occurs because a second extraction stage requires operation of a mixer at high RPM to achieve inter-stage liquid transfer. Once again, it can be seen that a balance must be struck between the degree of sulfate removal required and the financial costs associated with the extractant lost from the system. This information is important design information for future commercial implementation of the HydroFlex process, as an appropriate balance between sulfate removal and extractant carryover must be found for the specific operation.

In general, throughout TC1 the TOC of the treated AMD decreased as the run duration increased, as shown in Figure 14. This suggests that if the run were to have continued past the planned duration, the TOC in the treated discharge may have further decreased before reaching some steady state minimum TOC level. This indicates that estimates for extractant makeup based on the TOC results measured during TC1 will result in conservative economic projections.

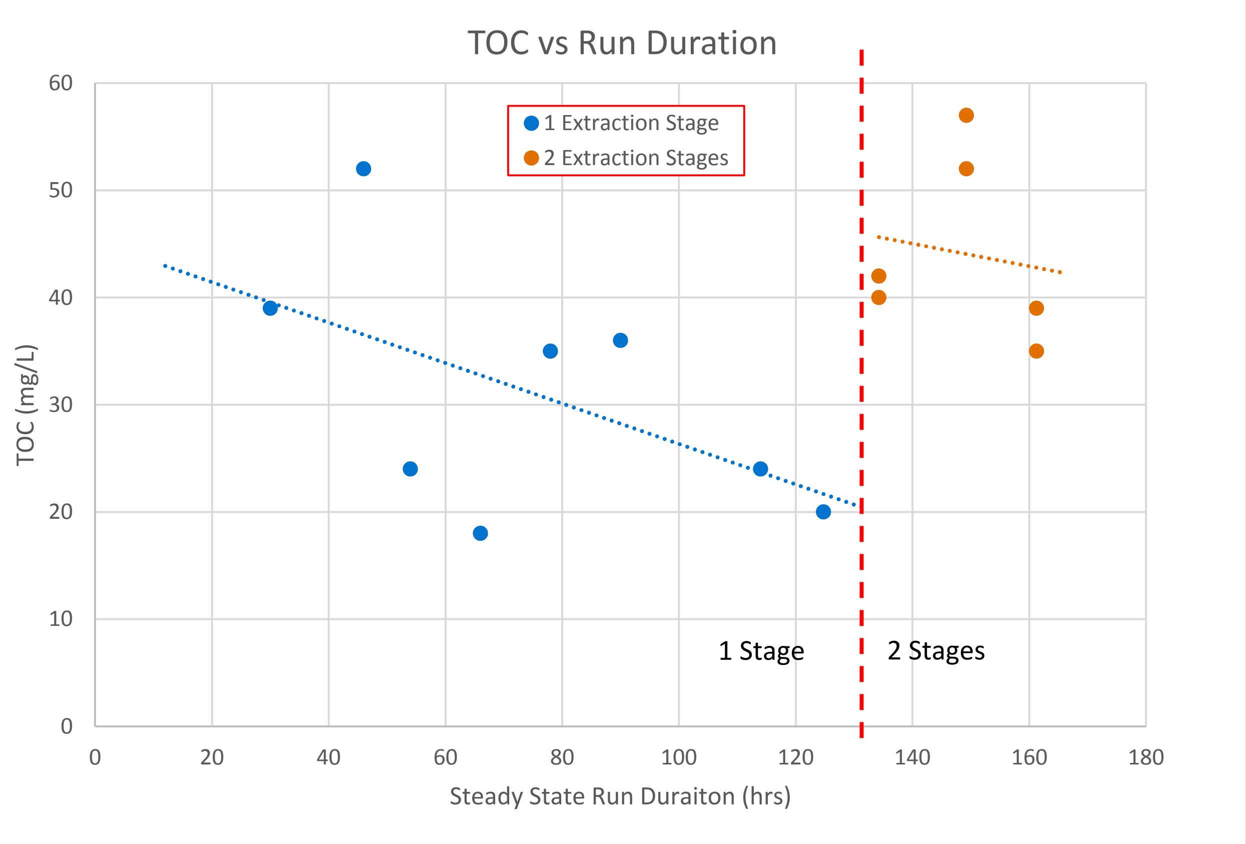

Figure 14. TOC vs Run Duration for operation of a single and dual extraction stages.

TEST CAMPAIGN 1 CONCLUSIONS

Test Campaign 1 demonstrated long term, steady state system behavior. Data from the first test campaign indicates that sulfate levels are effectively reduced through operation of the HydroFlex platform. An initial screening of the E:A ratio effect on sulfate removal performance indicated that to achieve the required sulfate levels under the Sarver, PA site conditions requires operation at an E:A approaching 1:2. A lower E:A ratio is sufficient at higher extractant strength, as evidenced by the results from the demonstration runs conducted in BP1 with fresh extractant.

Operation of a second extraction stage resulted in the reduction of the sulfate levels to below 100 mg/L during the test campaign, but increased extractant entrainment in the AMD discharge. Over TC1, sulfate removal and TOC measurements consistently improved throughout the entire test campaign, indicating that the results may be further improved as the system is operated over a longer period of time.

At the conclusion of TC1, a preliminary estimate of the process economics was conducted and the key cost drivers for the process were identified. These preliminary estimates, illustrated in Figure 15, indicate that carbonate consumption, extractant maintenance and filtration are key cost drivers. If an outlet for the byproduct cannot be established, then waste disposal will also be a key cost, but it is expected that the byproduct can be dispositioned at cost as a treatment chemical for produced water (see Sections 4.5 through 4.7). Additional discussion of the process economics can be found in Section 4.4.1.

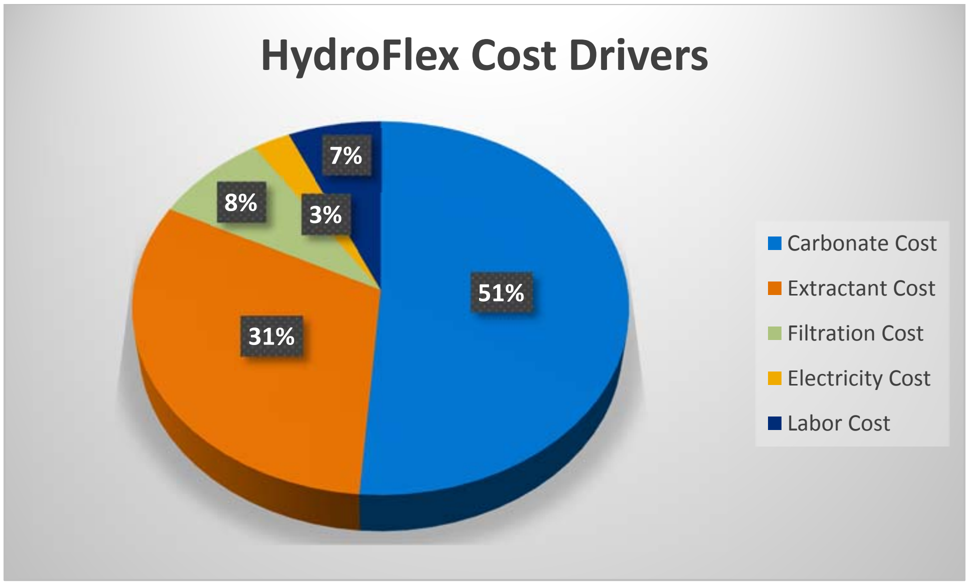

Figure 15. Key cost drivers for the HydroFlex process based on TC1 results.

4.3.3 CONFIRMATORY TESTING: LIMITED OPERATIONAL COST REDUCTION TESTS

Based on these results, the original objectives of test campaign two were redirected toward reducing the process cost drivers; specifically carbonate usage and filtration costs. The field unit was used for additional runs to evaluate the impact of reduced carbonate feed rate on the process performance. Additionally, centrifugal pumps in the system susceptible to fouling were replaced with diaphragm pumps and the filters removed to determine whether filtration costs could be eliminated. A full test plan and report for the Confirmatory Test Campaign (CTC) is included in Appendix C.

This additional testing was performed over the course of four weeks, during which the feed rate of carbonate was reduced from roughly 1.9-1.7 gpm at the end of the TC1 testing to 0.73 gpm. As in TC1, AMD water from both the mine and leach pile were blended to provide a feed between 500 and 600 mg/L SO_4, and two countercurrent extraction stages were operated to target a sulfate residual of 100 mg/L SO_4 or less. Titrations were done on each batch of sodium carbonate feed solution to determine the concentration, and removal was tracked by mass of sodium carbonate fed per mass of SO_4 ion removed. This accounted for variability of the feed carbonate concentration during the test run.

During the four week test, the sulfate feed concentration was 523 +/- 20 mg/L SO_4. The sulfate residual was 103 +/- 18 mg/L SO_4. This was accomplished with a specific utilization of 2.84 +/- 0.29 lb Na_2CO_3 feed per lb of SO_4 removed, almost half that of the 5.25 lb Na_2CO_3 feed per lb of SO_4 removed during the TC1 testing.

Besides carbonate consumption, the omission of fabric filters to prevent pump fouling in the output was evaluated. During BP1 testing and TC1, sub 100 micron bag filters were used prior to the product water pumps to prevent calcium carbonate scale from plugging the centrifugal pumps. Prior to TC1 the centrifugal pumps were replaced with diaphragm pumps, but the filters left in line as a safety precaution to prevent plugging of the oil water separator. Replacement of bag filters is a significant cost driver as shown in Figure 15. Use of 200 micron filter bags prior to the confirmatory tests resulted in no observed impact in oil water separator performance, so it was hypothesized that removal of the bags completely would still allow good performance at improved cost.

The bag filters were removed from the system during the second week of the confirmatory testing and the oil water separator monitored for plugging by solids. The diaphragm pumps did not suffer from scaling without the filters in place. The oil water separator did experience some fouling with precipitated solids at the leading edge of the packing, but it is anticipated that pressure washing of the packing on a regular schedule will allow for continued operation.

The confirmatory testing also included investigation of an alternate means to measure the level of extractant present in the treated AMD entering the granular activated carbon beds. Total organic carbon tests were previously used as an indicator of all organic compounds, but in this round of field testing a traditional Gas Chromatography-Mass Spectroscopy (GC-MS) method was evaluated for extractant component identification. This GC-MS work did not identify any components of the extractant compounds in the effluent water, which suggests that actual losses could be quite low. However, this work is not conclusive as the GC method used needs further validation to properly identify the extractant components, including running of reference materials. A discussion of how the findings from the confirmatory testing affected the process economics is given in Section 4.4.1.

4.4 Task 3.3: AMD Treatment Process Testing and Evaluation-Evaluation of Process Performance

4.4.1 PROCESS ECONOMIC DISCUSSION

Among the primary considerations behind adoption of the HydroFlex process by industry are the costs associated with the operation of the system. Data collected during the testing conducted as part of TC1 and the CTC were used to determine operating cost estimates for the process. As discussed in Section 4.3.3, operational cost projections were first made based on the results of TC1, which were the basis of the test objectives for the CTC. CTC results were then used to project the anticipated operating costs for a commercial system.

Table 2 presents the operational cost estimates determined as an outcome of TC1, the CTC, and production system cost projections. The basis of estimates for each operational parameter were continuously refined for each version of operational cost projections.

Table 3 defines the basis of estimate for each of the cost scenarios examined. Based on the cost analysis presented in Table 2, it is anticipated that the treated AMD costs will range from ~$4-10/kgal for future implementation of the HydroFlex technology.

Table 2. Operational cost projections based on TC1 and CTC results, as well as productions system operational cost estimates.

	Operational Costs		
Parameter	**TC1 Results**	**CTC Results**	**Projected for Commercial Operation**
Carbonate Cost	$5.34	$2.89	$2.48
Extractant Cost	$3.26	$1.94	$0.41*
Filtration Cost	$0.85	$0.00	$0.00
Byproduct Disposal	$10.20	$4.70	$0.00
Electricity Cost	$0.28	$0.24	$0.17
Labor Cost	$0.69	$0.69	$0.69
Total Cost per kgal	**$20.61**	**$10.46**	**$3.76**
Total Cost per bbl	**$0.87**	**$0.44**	**$0.16**

*requires confirmation by long term process operation results.

Table 3. Basis of estimates for each of the costing exercises conducted.

	Basis of Estimate		
Parameter	**TC1 Results**	**CTC Results**	**Projected Production**
Carbonate Cost	TC1 Results, using excess carbonate feed	CTC Results, reduced feed rate; avg for final week of test period	Theoretical utilization of carbonate based on stoichiometric dosing and using bulk carbonate pricing
Extractant Cost	Estimated extractant loss based on TOC measurements during TC1; assumes replacement of active ingredient	CTC TOC Results; Assumes replacement of bulk extractant mixture	Based on 10 ppm target loss; anticipated long term system performance
Filtration Cost	TC1 Results, based on filter replacement cost and change out frequency	CTC Results, assumes no filters required	CTC Results, assumes no filters required
Byproduct Disposal	TC1 Results, $0.30/gal of waste disposed	CTC Results; avg for final week of test period. $0.265/gal of waste disposed	Disposition at no cost
Electricity Cost	TC1 Results, higher electricity rates due to higher tiered rate	CTC Results; based on observed load, lower tiered rate	Typical industrial sector costs
Labor Cost	Engineering estimate for commercial system; assumes operator on site 2 hrs/day	Engineering estimate for commercial system; assumes operator on site 2 hrs/day	Engineering estimate for commercial system; assumes operator on site 2 hrs/day

One of the primary cost drivers for the process is the amount of carbonate fed to the sulfate stripping stages to regenerate the extractant. During TC1, carbonate was fed to the stripping stages at an average flow rate of 2.1-1.9 GPM which results in a contribution of $5.34 to the operational costs associated with TC1. During the CTC, the carbonate flow was reduced to 0.73 GPM, which is less than half of the carbonate flow rate fed during TC1, while still maintaining extraction performance. By achieving more efficient utilization of carbonate fed to the system, operational cost reductions were not only realized in the carbonate cost, but also in the waste disposal of the byproduct stream as the byproduct volume produced during operation of the system was reduced. Under steady state operation, the byproduct generation rate is nominally equal to the carbonate feed rate to the stripping stages. For a production system, the cost for procurement of carbonate is further reduced as carbonate purchased in bulk vs. bagged is less expensive. In addition, it is assumed that the process byproduct will be able to be dispositioned at cost (see byproduct testing discussion in Section 4.5). Additional process improvements are projected to provide carbonate utilization rates which approach theoretical stoichiometric predictions.

Continued process modifications to reduce organic losses from the system are expected to reduce the costs associated with extractant maintenance, with final production system costs consisting of a minor makeup stream for the extractant material.

During the CTC, it was demonstrated that the filtration skid used in the process to protect process equipment was not required to achieve required equipment reliability thresholds. Therefore, filtration costs were removed from the CTC and projected production system cost estimates. Additionally, more refined estimates of electrical consumption were made for the cost of electricity to operate the process, as large industrial processes typically receive lower cost rates.

From previous conversations with stakeholders who utilize source water for HF purposes, water is typically purchased for $0.12-0.50 per barrel (i.e., $2.86-$11.90 per thousand gallons). This indicates that the HydroFlex system has the capability to meet industry cost targets for provision of treated AMD water under the assumptions used in the economic projections. However, adaptations made to the system during the field trials will require permanent incorporation in the commercial system, and a low cost disposal route will need to be identified for disposal of the process byproduct. Finally, the current pilot process requires an operator to be present at all times during system operation. This is due, in part, to the current set up of the HydroFlex system as an experimental pilot facility. Additional automation and process control equipment would reduce operator requirements to only a few hours per shift, translating to reduced labor costs.

4.4.2 STAKEHOLDER OUTREACH DISCUSSION

Regular communications with the project stakeholder committee was maintained throughout BP2 to facilitate feedback on the HydroFlex process performance and economics. There were no changes to the stakeholder group membership during BP2. Table 4 shows the stakeholders who provided feedback throughout the project.

Table 4. Stakeholder committee for source water quality requirements.

Stakeholder	Type
StatOil	Industry
XTO Energy	Industry
PADEP Oil & Gas Program	Regulatory
PADEP Conservation and Restoration	Regulatory
PADEP Office of Surface Mines	Regulatory
Water Research Institute	Academic

The stakeholder committee was briefed approximately quarterly on the project activities. During the stakeholder briefing held at the end of April 2015, the stakeholders were briefed on the planned field test campaigns and were asked to provide feedback to ensure that the testing was aligned with industry requirements. Stakeholders confirmed that the planned testing was an appropriate demonstration of the technology’s ability to meet their requirements for AMD water treatment.

After the BP2 field testing was completed, the stakeholders were briefed on the test results. Water quality characteristics achieved by the HydroFlex process were shared and compared against the requirements for source water defined in BP1. Table 5 shows the water quality characteristics achieved by the HydroFlex process during TC1 compared against the stakeholder committee established source water benchmark characteristics.

Table 5. HydroFlex process water quality compared to projected water requirements for source water.

Parameter	Sarver Site AMD	Slickwater Target	Gel Target	HydroFlex Sarver Site
Chlorides (mg/L)	N/A	< 70,000	<25,000	N/A*
Iron (mg/L)	<1	< 10	< 10	<1
Carbonate/Bicarbonate (mg/L)	< 10	< 400	< 500	350-450
Calcium (mg/L)	<5	< 8,000	< 100	<5
Magnesium (mg/L)	20	< 1,200	< 100	20
Sulfates (mg/L)	550-650	< 400	< 300	<100
pH	8-9	5 to 7	6 to 8	9-10
Total Dissolved Solids (mg/L)	600-800	< 120,000	N/A	500-700

Additionally, stakeholders were briefed on the projected process economics based on the results of TC1. Industry stakeholders agreed that the projected price point based on TC1 data for water treatment of less than $0.50/bbl, which does not include markup for profits or delivery charges, seems like a reasonable cost for treatment. One of the industry stakeholders discussed potential transportation issues associated with using treated AMD for source water. They discussed that they are working toward taking water transportation trucks off the roads wherever possible, as transportation costs associated with trucking and bonding frequently used roads is not economically sustainable. They are transitioning to piping their source water when geography allows, and stated that if the AMD treatment sites were located within a feasible pipeline radius from the well sites, it would make for a more attractive water source. Both industry stakeholders highlighted the importance of process economics as the driving factor behind industry acceptance, and that the overall cost of source water needs to include not only treatment costs, but also transportation and/or storage costs.

4.5 Task 4.1: Process Byproduct Testing and Evaluation-Test Plan Development

The HydroFlex process produces a byproduct which is primarily an aqueous solution of sodium sulfate and sodium carbonate. Exploration of use of this process byproduct in produced water treatment was conducted as part of this project for several reasons. First, reuse of the process byproducts in other industries helps to reduce the costs associated with handling and disposal of the byproduct as a process waste. Second, by providing a cost effective means to recycle produced water, freshwater burdens of HF operations are further reduced. Finally, if the process byproduct is able to be dispositioned at reduced cost or at a profit, the operational economics of the HydroFlex process are further improved.

Preliminary discussions with water treatment facilities revealed that the byproduct from the HydroFlex stripping stages has potential value as a softening/precipitation solution. Of particular concern when recycling produced water is the presence of barium and strontium, which in the presence of sulfate will precipitate out of solution causing scaling of process equipment and formation damage downhole. Additionally, polyvalent metals commonly present in the produced water such as calcium and magnesium can interfere with the crosslinking behavior of gel frac fluid formulations. Battelle tested the effectiveness of the HydroFlex process byproduct for removal of metals and reduction of hardness and turbidity from produced water. The byproduct was compared to solid sodium sulfate which is representative of the treatment chemicals currently used by oil and gas service companies.

A series of experiments were developed to evaluate the performance of the HydroFlex process byproducts against a standard, industrial grade sodium sulfate. Approximately 5 gallons of the sodium sulfate byproduct was collected from the S1 vessel of the HydroFlex plant during TC1 on August 5, 2015 and stored in a sealed, 5 gallon container until analysis/testing. The collection of this sample was timed to ensure that a representative sample of process byproduct was used for the comparative testing. A 5 gallon sample of produced water was collected from a produced water disposal facility site in the Marcellus region and stored in a sealed 5 gallon Nalgene container for analysis and testing. The produced water sample was collected from a well-mixed reservoir of produced water and is considered a representative sample of typical Marcellus Shale produced water.

4.6 Task 4.2: Process Byproduct Testing and Evaluation-Laboratory Testing

The produced water sample was first analyzed for pH, SO_4, alkalinity, turbidity, and concentrations of calcium, magnesium, barium, strontium, and iron. The concentrations of Ba and Sr in the produced water sample were totaled to determine the total moles of target cations that will react and precipitate with SO_4. The sample of HydroFlex process byproduct was analyzed for pH, alkalinity, and SO_4. For each test conducted, byproduct solution (34.1 g/L) was added to 0.35 L of produced water in a 500 mL beaker to equal a stoichiometric ratio of 1 (moles sulfate to moles Ba and Sr). This was repeated for stoichiometric ratios of 0.5 and 2. The entire procedure was repeated using sodium sulfate powder obtained from Sigma Aldrich at the same three stoichiometric ratios. All reaction products were allowed to settle for 5 minutes after which turbidity was analyzed on the clarified sample. Clarified samples were collected and sent to a third party laboratory for total recoverable metals determination by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES).

4.7 Task 4.3: Process Byproduct Testing and Evaluation-Evaluation of Performance

The byproduct testing indicated that the HydroFlex byproduct provides similar performance as technical grade sodium sulfate powder with regard to barium and strontium removal from produced water. However, the byproduct also demonstrated better iron removal, better calcium removal, and better settling characteristics. These results are promising as an outlet for the process byproducts is pursued.

Table 6 summarizes all analyses performed. The pre- and post-mixing test concentration in the produced water sample was evaluated for total recoverable metals (Fe, Al, Ba, Sr, Mg, and Ca), pH, sulfate and alkalinity (ICP-AES per EPA method 200.7 was performed by an outside lab). All precipitation reaction effluent samples were analyzed in triplicate for statistical analysis.

Table 6. Analysis plan for reaction effluent

Sample	Parameters	Analyses
Precipitation Reaction Product	pH, SO_4, alkalinity, turbidity, Ca, Mg, Ba, Sr, Fe	• 2 tests (byproduct and prepared $NaSO_4$); • x 3 concentrations (0.5, 1.0, 2.0 x stoichiometric value); • x 3 reactions (triplicate for statistical analysis) = **18 total samples**

Metals were analyzed by EPA method 200.7, and the results are summarized in Table 7. In general, both the process byproduct and standard sodium sulfate addition reduced all metals by the same order of magnitude. Reduction of all metals increased with increasing concentration of byproduct and sodium sulfate. Both the quantity of sulfate added to the reaction effluent and alkalinity increased with increasing concentration of HydroFlex byproduct.

Table 7. Summary of Precipitation Reactions with HydroFlex Byproduct and Sodium Sulfate Powder

Analyte	Raw Produced Water (mg/L)	Treatment with HydroFlex Byproduct (mg/L)			Treatment with Sodium Sulfate Powder (mg/L)		
		0.5	1	2	0.5	1	2
Ba	641	11.74 (+/-10.5)	2.92 (+/-0.58)	0.72 (+/-0.23)	4.11 (+/-5.3)	1.21 (+/-1.8)	0.37 (+/-0.18)
Ca	12,200	12,733 (+/-1507)	11,433 (+/-1051)	9,963 (+/-72)	13,167 (+/-570)	13,267 (+/-1099)	12,567 (+/-663)
Fe	133	2.11 (+/-0.99)	0.73 (+/-0.82)	0.171 (+/-0.34)	26.5 (+/-3.1)	24.5 (+/-1.1)	23.1 (+/-1.78)
Mg	1290	1,437 (+/-136)	1,340 (+/-128)	1,300 (+/-45)	1,450 (+/-88)	1,470 (+/-120)	1,413 (+/-124)
Sr	2120	1,823 (+/-52)	1,480 (+/-85)	1,197 (+/-103)	1,833 (+/-73)	1,483 (+/-137)	1,057 (+/-36)
Alkalinity	0	287 (+/-33)	347 (+/-13)	393 (+/-24)	123 (+/-68)	133 (+/-52)	77.5 (+/-20)
Sulfate	2.1	589 (+/-87)	1,553 (+/-6.5)	3,490 (+/-118)	544 (+/-15)	1,073 (+/-86)	1,863 (+/-288)
Turbidity	>2000	100 (+/-49)	50 (+/-0.98)	36 (+/-27)	>1,100	>1,100	>1,100

Note: 95% confidence intervals are shown in parentheses.

Table 8. Percent Removal of Precipitation Reaction Metals

Analyte	Raw Produced Water (mg/L)	Hydroflex Byproduct (mg/L)			Solid Sodium Sulfate (mg/L)			Byproduct Removal Capacity
		0.5	1	2	0.5	1	2	
Ba	641	98.2%	99.5%	99.9%	99.4%	99.8%	99.9%	Similar
Ca	12,200	N/A	6.3%	18.3%	N/A	N/A	N/A	Increased
Fe	133	98.4%	99.5%	99.9%	80.1%	81.6%	82.6%	Increased
Mg	1290	N/A	N/A	N/A	N/A	N/A	N/A	Similar
Sr	2120	14.0%	30.2%	43.6%	13.5%	30.0%	50.2%	Similar

The HydroFlex byproduct and sodium sulfate powder demonstrated similar ability to remove key metals Ba and Sr from the produced water sample, as seen in Figure 16 and Figure 17. Sufficient sample analysis was conducted such that confidence intervals for the analysis could be determined. Note that percent removal values are shown next to each data point in addition to error bars which represent the 95% confidence interval. These test results indicate that the HydroFlex process byproduct is able to achieve similar removal rates as existing chemicals used in softening operations. Calcium and iron removal may also be important to O&G end users of the processed water since calcium, iron, and other polyvalent metals can interfere with the performance of crosslinkers in gel frac fluids. The byproduct appeared to have better removal of iron at all doses as shown in Figure 19, and calcium at the higher doses as in Figure 18. This is expected since the byproduct is likely to have a higher pH which aids in iron precipitation, while the carbonate present in the byproduct results in precipitation of insoluble calcium carbonate.

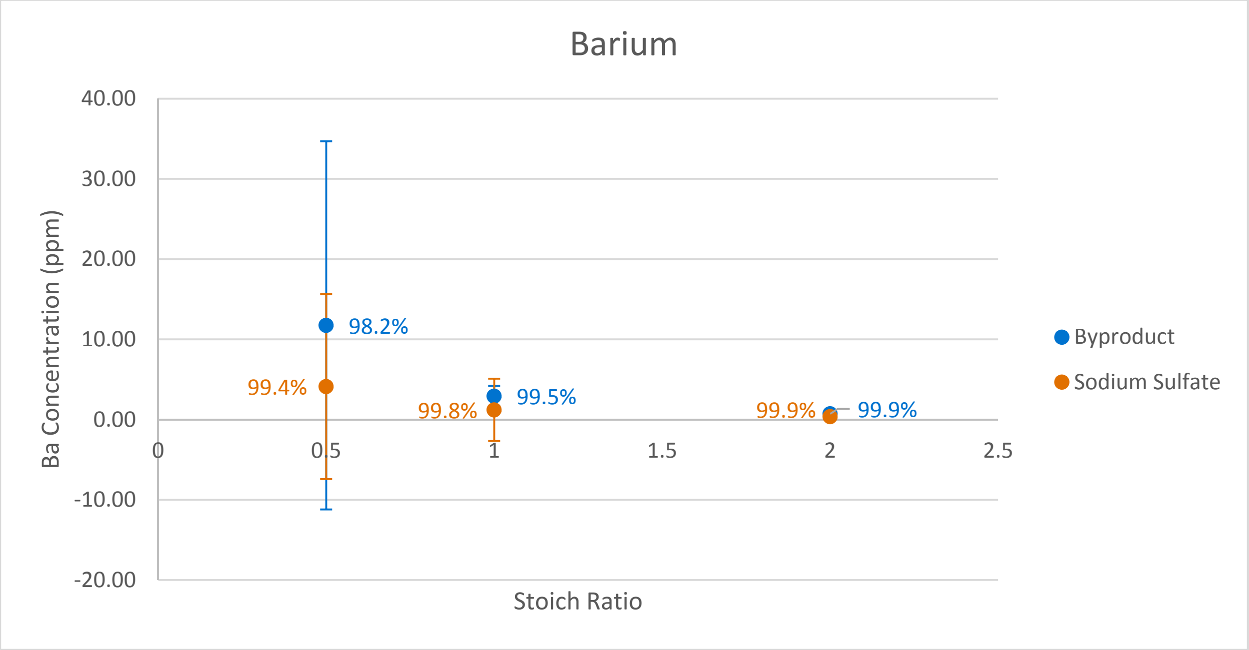

Figure 16. Barium Removal Results for Byproduct and Sodium Sulfate Powder

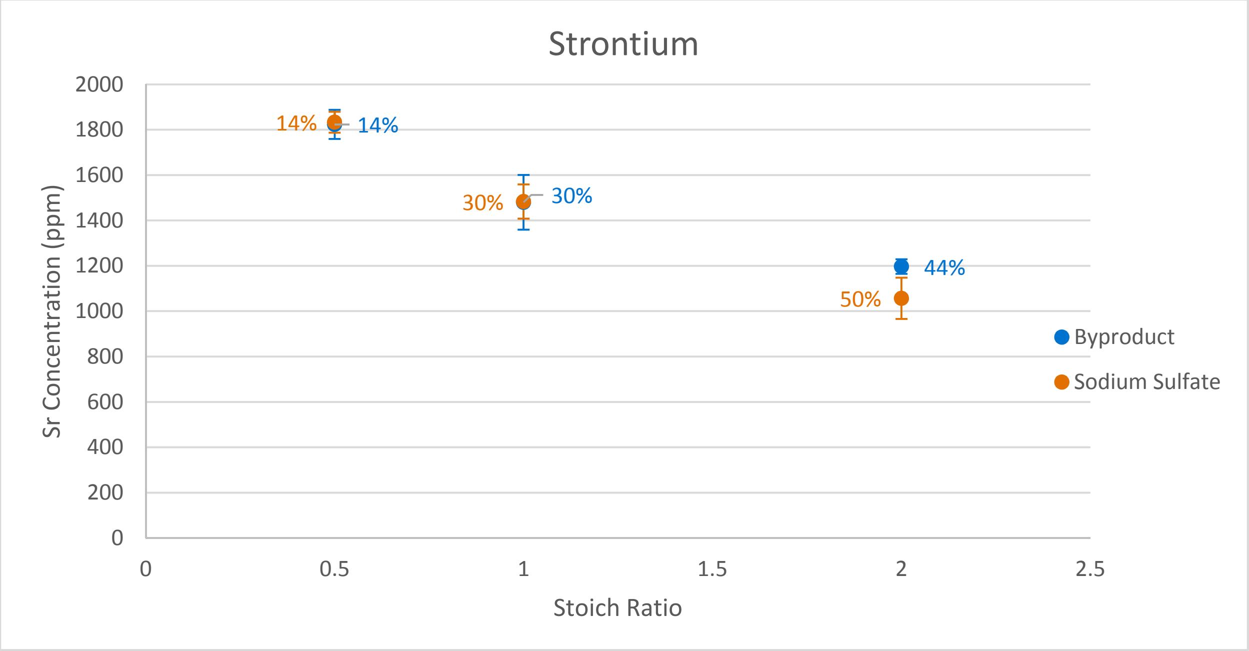

Figure 17. Strontium Removal Results for Byproduct and Sodium Sulfate Powder

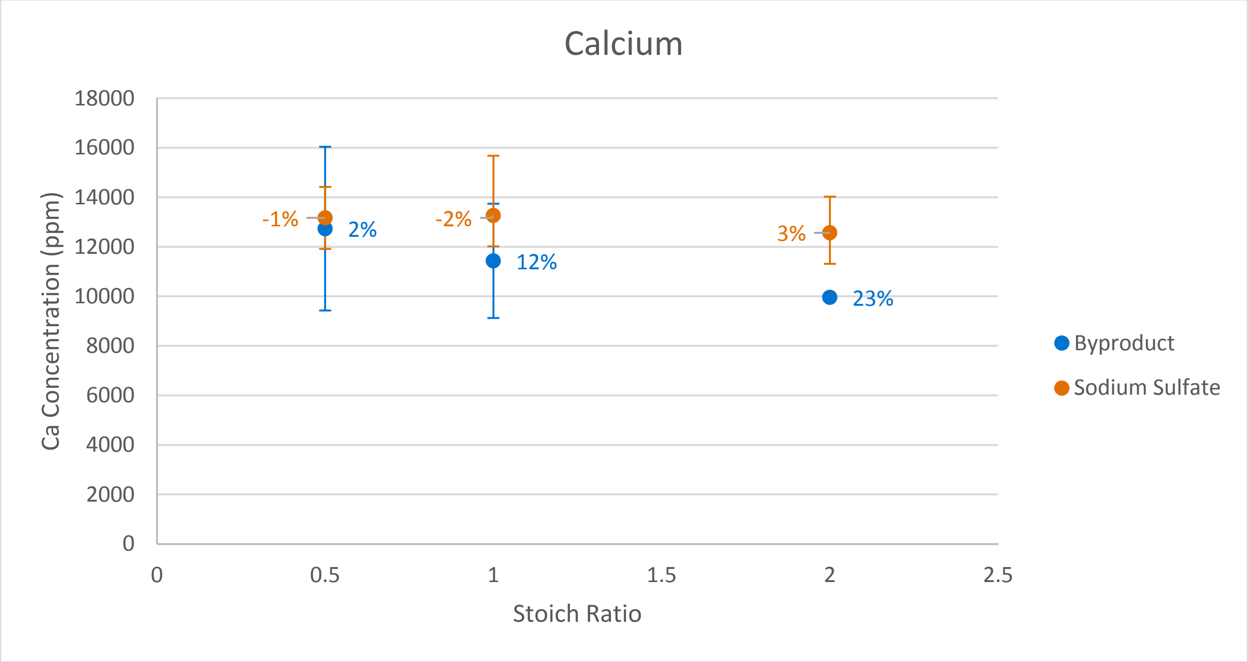

Figure 18. Calcium removal results for byproduct and sodium sulfate powder. Error bars represent the 95% confidence interval, while percentage labels show the average percentage removal.

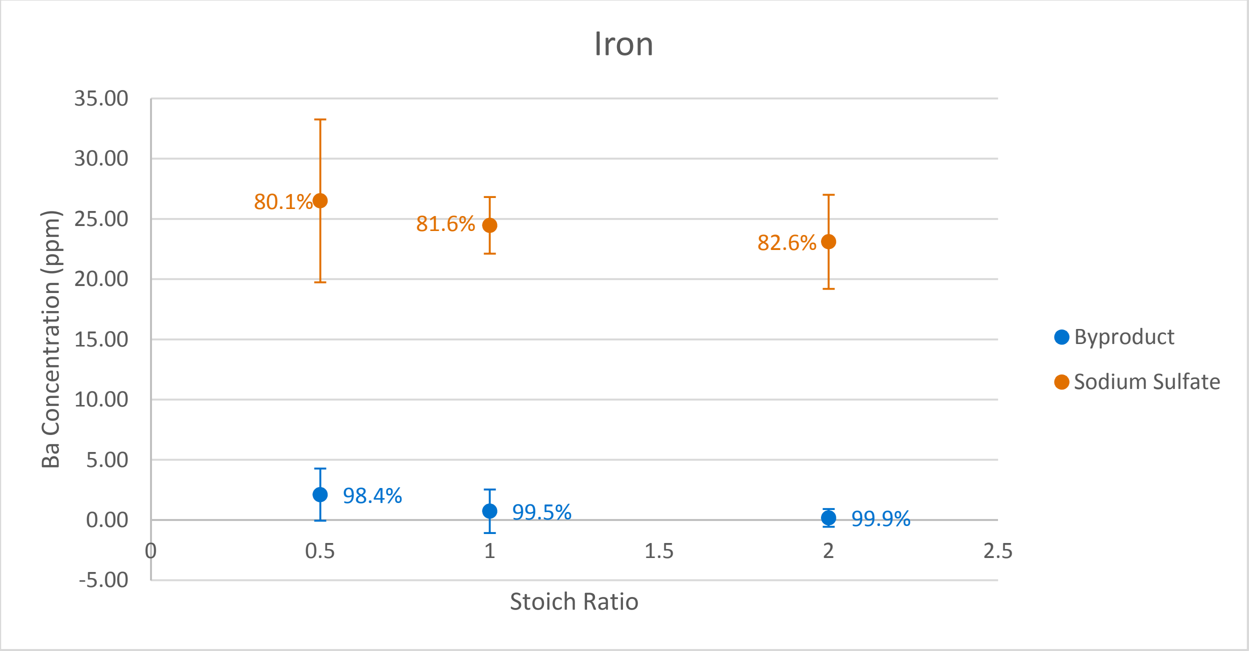

Figure 19. Iron removal results for the process byproduct and solid sodium sulfate

During the tests, it was observed that the samples treated with the HydroFlex byproduct settled much faster than the samples treated with the standard sodium sulfate powder. To quantify this observation, turbidity measurements on all samples were taken after 5 minutes of settling time. The results of this testing are shown in Table 9.

Table 9. Turbidity measurements (NTU) by stoichiometric dose rate for byproduct and sodium sulfate after 5 minutes of settling time.

	HydroFlex Byproduct			Na_2SO_4		
	0.5	1	2	0.5	1	2
Ave Turbidity (NTU)	100	49.5	36	>1100	>1100	>1100

As shown, the turbidity of the samples treated with the HydroFlex effluent is lower by at least an order of magnitude when compared to the standard sodium sulfate powder. This is believed to be advantageous for a treatment process which may use similar chemicals to achieve a softening reaction, as more rapid clarification of the sample typically results in decreased residence time, helping to reduce the footprint required for settling tanks and ponds and increasing the throughput of the process. Figure 20 shows representative samples of the treated effluent, illustrating the difference in clarity between samples treated with byproduct and with standard sodium sulfate. While specific analysis regarding why clarification is improved when using the HydroFlex process byproduct was not conducted, it is believed it is due to the presence of dilute constituents from the AMD source, including trace metals, which assist in flocculation and settling of particles.

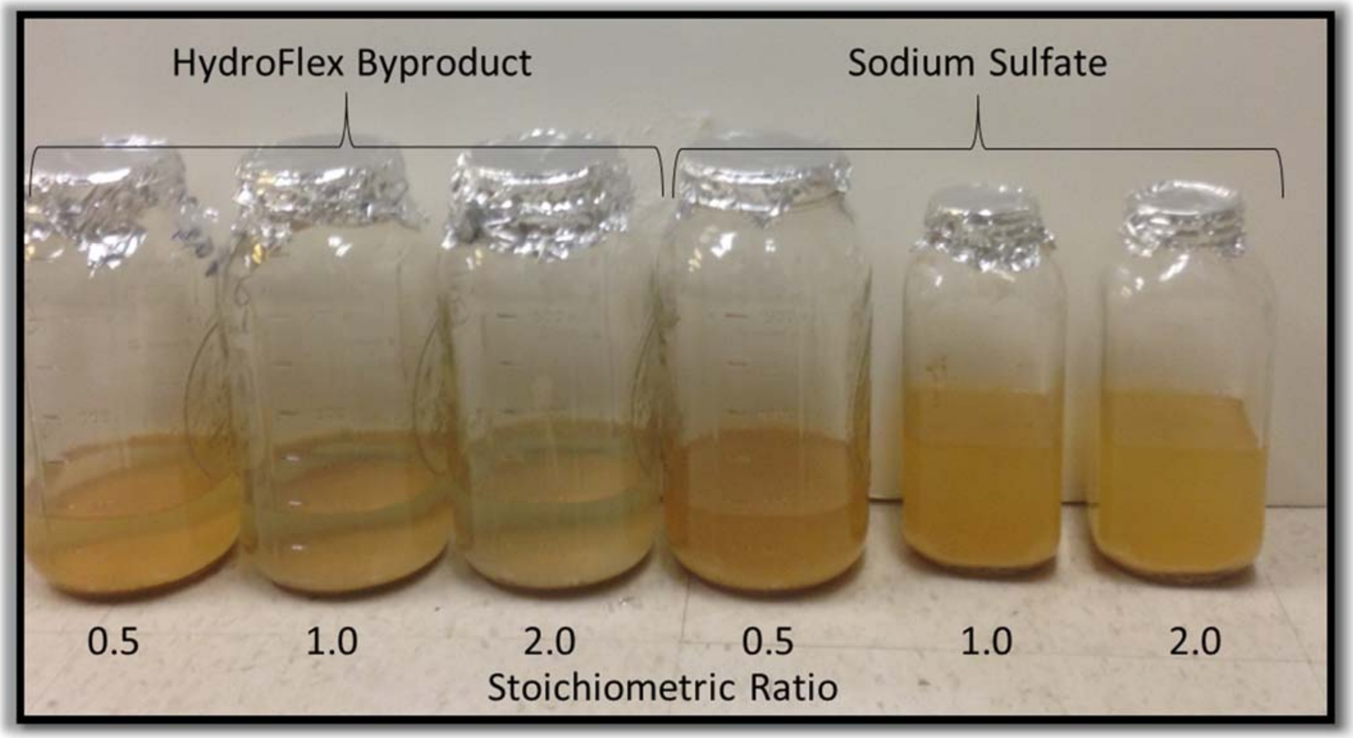

Figure 20. Samples of produced water treated at different stoichiometric ratios with byproduct and sodium sulfate solids. Note that byproduct treated samples appear clearer than those treated with sodium sulfate.

While initial tests of the process byproduct for produced water treatment were promising, additional testing is required to fully validate the process. Future testing should include processing of multiple samples of produced water from several different formations to ensure that the process chemistry is effective across all anticipated water chemistries. Additionally, scale up of the process should be considered, and future tests should be conducted on a continuous system. Finally, the dilute concentration of the process byproduct may pose challenges for treatment facilities, as its use may result in a larger volume of water resulting from the treatment process. This should also be considered in future testing.

5.0 Communication of Project Results

To address the program objective to support transition of the technology and project results into implementation by the oil and gas industry, the project results to date were presented in this budget period at two conferences by Battelle's subcontractor, Winner. These conferences were selected because of the opportunity to disseminate project information to industrial, regulatory, and academic parties of interest, as well as to receive feedback on the challenges encountered when unconventional water sources are used as a source water.

Table 10. Conference attendance information.

Event	Location	Date	Presenter	Presentation Title
Society of Petroleum Engineers, Annual Technical Conference and Exhibition	Houston, TX	9/28/15-9/30-15	Todd Beers, Winner Water Services	Paper No. SPE-174947-MS Treating Acid Mine Drainage for Use as Source Water: A Pilot Study
Ohio Mineland Partnership	Marietta, OH	10/13/15	Carolyn Kotsol, Winner Water Services	Development and Validation of an Acid Mine Drainage Treatment Process for Hydraulic Fracturing Source Water

6.0 Conclusions

The objectives and milestones set forth for BP2 have been accomplished, although continued optimization of the HydroFlex technology is required to meet the industry cost targets. Test campaigns were conducted using the 100 GPM HydroFlex unit to collect data pertaining to the sulfate removal abilities of the system as well as the associated operating costs. During TC1, it was determined that the system was consistently able to remove sulfate from the AMD to levels below 150 or 100 mg/L, depending on the number of extraction stages in operation. Removal of sulfate to this level is aligned with industry stakeholder feedback on threshold sulfate levels for source water.

System operational costs were estimated based on TC1 data. These data were used to identify discrete tests conducted as part of the CTC which demonstrated adjusted operating conditions capable of achieving similar sulfate removal performance at a reduced operating cost. Based on the results of the CTC, treatment costs of ~$0.44/bbl were estimated, however these costs do not include transportation or any mark-up for profit. With further modifications and long term operation of the HydroFlex system, it is projected that water can be treated for $0.16/bbl, leaving room for the addition of transportation and mark-up costs, while still meeting typical source water costs of $0.12-$0.50/bbl. However, to achieve this would require identification of an outlet for the process byproduct.

Laboratory testing conducted on the process byproduct stream against industrial water softening chemicals indicate that use of the byproduct sodium sulfate solution for the removal of barium from produced water may be advantageous. Testing indicates that both industrial sodium sulfate solutions and the HydroFlex process byproduct solution are capable of removing more than 99% of barium present and over 50% of strontium present. It was found that produced water samples treated with the HydroFlex process byproduct clarify more rapidly than samples treated with industrial sulfate solutions. Decreased turbidity in the treated produced water effluent indicates that smaller settling areas may be required to achieve similar removal efficiencies, potentially allowing for increased production from existing produced water treatment facilities.

This project allowed for the demonstration and validation of a novel technology to treat AMD and provides a means to convert an environmental liability to a benefit for the HF industry. The technology has evolved significantly over the course of the program, and process improvements were identified which allow the system to operate more efficiently. Future steps taken by Winner will include implementation of the process improvements identified as part of this study, as well as continued efforts to improve process efficiency. Additionally, Winner is working to translate the findings from this project into other industrial applications, further increasing the versatility of the HydroFlex technology.

7.0 Bibliography

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8.0 List of Acronyms and Abbreviations

Acronym	Definition
AMD	Acid Mine Drainage
BP1	Budget Period 1
BP2	Budget Period 2
CTC	Confirmatory Test Campaign
DOE	Department of Energy
E:A (or E/A)	Extractant to Aqueous phase ratio
EDP	Experimental Design Plan
HF	Hydraulic Fracturing
NCTE	No Cost Time Extension
PADEP	Pennsylvania Department of Environmental Protection
RPM	Revolutions per Minute
TC1	Test Campaign 1
TC2	Test Campaign 2
TOC	Total Organic Content
VOC	Volatile Organic Compound

9.0 Appendices

Appendix A: (Confidential – not included in this document)

Appendix B: Test Campaign 1 Test Plan

Appendix C: (Confidential – not included in this document)

Appendix D: Selected Stakeholder Meeting Minutes

Appendix E: Selected Stakeholder Project Update Communications

Appendix F: Cost Information Back-Up

Appendix G: Technology Introduction

Appendix A: (Confidential – not included in this document)

Appendix B: Test Campaign 1 Test Plan

DEVELOPMENT AND VALIDATION OF AN ACID MINE DRAINAGE TREATMENT PROCESS FOR SOURCE WATER

Field Demonstration Test Campaign One

July 6, 2015 through August 14, 2015

WORK PERFORMED UNDER AGREEMENT

DE-FE0014066
DUNS: 007901598

BY

Battelle Memorial Institute
505 King Ave
Columbus, OH 43201

July 6, 2015

PRINCIPAL INVESTIGATOR

Ann Lane, Battelle
614-424-3266
lanea@battelle.org

Test Campaign Background

A single test campaign (TC1) and an optional second test campaign (TC2) are planned under the DOE sponsored AMD treatment project. The objectives of these test campaigns, as stated in the Statement of Project Objections (SOPO) approved by the DOE, are:

1. Demonstrate AMD water remediation by HydroFlex for use as source water in HF.
2. (Optional) HydroFlex process chemical optimization, including concentration, feed methods, and usage minimization. The process chemicals to be examined may include sodium bisulfite, sulfuric acid, sodium carbonate, and extractant.

After completion of TC1, a "GO/NO GO" decision will be made by Battelle and the DOE regarding whether or not TC2 will be conducted. If a "GO" decision is reached, TC2 will be conducted and additional required funding will be awarded by the DOE to conduct the test campaign. Should a "NO GO" decision be reached, field testing will halt and the project will focus on completion of the remainder of the tasks in Task 3 and Task 4. Criteria for the "GO/NO GO" decision point include the ability of the system to achieve the sulfate removal targets set forth by the project stakeholder committee as well as to have line of sight to an economical process operation.

Battelle will develop a test plan for each of the two test campaigns to include the field system operating conditions, sampling and analysis procedures, and direction regarding the length of time the system shall be operated at each condition. Statistical analysis of the test results shall be conducted to evaluate the performance of the HydroFlex water treatment process and its ability to meet the project objectives. The test results shall be compared to industry requirements for source water. The results of this evaluation shall be shared with the stakeholder committee to confirm the applicability of the technology to their requirements. A field test report will be compiled detailing the field test results and initial stakeholder feedback.

Prior to the start of each campaign, the test plan will be finalized with Winner Water Services (Winner) such that the objectives and operational conditions of the test are understood. The test plan for TC2 will be drafted in full upon completion of TC1, such that all relevant information gathered during TC1 may be reflected and built upon in TC2.

During TC1, the primary objective will be to treat AMD to meet the requirements of the HF industry. The target which the stakeholders have communicated to Battelle is that sulfate levels will range between 50-100 ppm. This will be accomplished by screening several E:A ratios to confirm that the behavior observed during laboratory testing at various process conditions in the field can be correlated with the behaviors associated during BP1 laboratory testing. This will be followed by long term steady state testing at a single E:A ratio.

The objectives of TC2 will be to optimize the process economics given the results of TC1. The full test plan for TC2 will be developed after the completion of TC1, in order to incorporate the results of TC1 and re-evaluate the planned test execution to accomplish the TC2 objectives if necessary. The test approach anticipated for TC2 will be to adjust the carbonate dosing rates with the goal to a) reduce system chemical operating costs, and b) improve stripping performance. If required, additional stripping stages may be added to the system to improve stripping performance while lowering carbonate consumption. The impact of the changes in the carbonate dosing rate on the byproduct characteristics will also be evaluated.

Test Campaign One Overview

During TC1, the primary objective will be to treat AMD to meet the requirements of the HF industry. The target which the stakeholders have communicated to Battelle is that sulfate levels will range between 50-100 ppm. This will be accomplished by screening several E:A ratios to confirm that the behavior observed during laboratory testing at various process conditions in the field can be correlated with the behaviors associated during BP1 laboratory testing. Excess carbonate flow[1] will be maintained throughout this phase of testing.

It is anticipated that the impact of varying E:A ratio on sulfate removal will be observed within several hours, as the aqueous phase should turnover in the system a minimum of once every hour (only E-1 volume incorporated). The variability of sulfate removal observed in Phase 1 testing during steady state runs will be used to estimate the number of samples needed at each E:A to ensure confidence in the results. With the results, a correlation will be drawn for sulfate removal with E:A, such that projections can be made for the system performance in future runs. Additionally, the results will be compared to lab testing results to draw correlations between the lab data and field data. At least 3 E:A ratios will be tested to enable detection of curvature in the correlation.

After the initial screening of the E:A ratios are complete, long term steady state testing will be conducted at the E:A ratio which was projected to best achieve the sulfate removal targets. This long term, steady state testing will allow the performance of the sulfate stripping stages to be evaluated, which in turn will impact the performance of the extraction stages. While the aqueous turnover rate for the system (including T1 volume) is less than three hours, the turnover time for the sulfate stripping solution is expected to be a minimum of 40 hours. Therefore, the pilot system will need to be run for several weeks at the identified E:A ratio to validate steady state performance. As before, variability data from the phase 1 run will be used to estimate the required number of samples to ensure confidence in the system performance at this condition.

A summary table of the run conditions to be conducted in the first test campaign is shown below.

Parameter	E:A Ratio Testing	Steady State Testing
Blended AMD Flow Rate (GPM)*	60	60
Blended AMD Sulfate Concentration (ppm)	550-700	550-700
Estimated Total Run Time (hours)	16	200
Carbonate Flow Rate (GPM)	1.5	1.5

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[1] Determined from current sulfate levels in blended stream on site.

Effluent Sulfate Target Concentration (ppm)	50-100	50-100
Effluent Sulfate Maximum Concentration (ppm)	300	300

*AMD flow rate to be adjusted as required to maintain target sulfate concentrations

Testing Operations

System Startup

At the start of the test campaign, the system will first be operated for several hours at a baseline condition to ensure that there is no transient behavior as the test campaign begins. Startup and operation of the system will be conducted per Winner's system operating procedures. The system will be set to a flow rate of 60[2] gpm and an E:A ratio of 1:10. To compensate for the low sulfate levels in the existing site ponds, the existing water will be blended with another stream which is higher in sulfate concentration prior to feeding the HydroFlex system. Throughout the test campaign, the blended AMD stream is required to maintain a sulfate level between 550 and 700 ppm. The overall AMD flow rate may be adjusted as required to maintain influent sulfate levels within the required window. If sulfate levels drop below 550 ppm for a prolonged length of time, testing will be suspended until sulfate levels can recover.

Operation under these conditions for 8 hours will ensure that the system is under steady state conditions and ready to begin the test campaign, confirmed by sulfate samples taken at the 6 and 8 hour mark. Total Organic Carbon (TOC) samples will be pulled from the E-1 aqueous sample port when sulfate samples are pulled and may be analyzed to help determine system performance.

E:A Screening

During the E:A screening stage of TC1, three additional E:A ratios beyond the E:A ratio explored during system startup will be evaluated. The system will be transitioned through the E:A ratios specified below, but in a random order and with repeats as necessary to ensure confidence in the results:

1. 1:10 (startup E:A)
2. 1:6
3. 1:4
4. 1:2 (as allowable by system design)

During testing at any one E:A ratio, it is acceptable for a deviation of +/- 10% to occur in the E:A ratio without invalidating the test. Sufficient run time to allow for a turnover of the aqueous volume in E-1, estimated not to exceed 4 hours, will be conducted at each ratio (excluding the initial 1:10 E:A), with samples being pulled during the final hour of run time, as per the sampling plan (for example: five samples over a one hour period). After testing at each E:A ratio for the prescribed amount of time, the testing will be paused until the E:A ratio which provides the best performance has been identified.

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[2] Subject to modification based on ability to achieve required sulfate concentrations.

The best sulfate removal will be identified as the lowest average sulfate level as measured by HACH kit during the four hour run. Prior to moving along to the steady state run portion of TC1, sulfate levels on the discharge water must be consistently less than 300 ppm. Should sulfate levels exceed 300ppm, testing will be paused as the need for incorporation of a second extraction stage is evaluated.

During this phase in testing, carbonate flow to the stripping stages will be maintained at 1.5 gpm based on an influent flow of 60 gpm of AMD and a blended sulfate level of 600 ppm. Should the influent flow rate or blended sulfate level change, the carbonate flow will need to be adjusted accordingly.

Steady State Run

After the E:A ratio which provides the greatest sulfate removal has been identified, the system will be operated until steady state conditions are reached, not to exceed 300 hours of run time. During this run the system will be set to a flow rate of 60[3] gpm. This will allow for the stripping stages to turn over several times, and provide valuable data as to the extraction performance long term. Sulfate levels of the process influent will remain the same concentration which was used in during the initial E:A testing. Sampling will be conducted throughout this run as stated in the sampling plan.

Sampling and Data Management Plan

Samples will be taken during TC1 per the sampling plan set forth in this test plan. Table 1 shows the samples to be collected, the sampling location, and the sample frequency for each stage of TC1. Note the different sampling frequencies for the first stage of TC1 (E:A screening), and the second stage (steady state run).

Table 1: Sampling plan for TC1

Sample	Location	E:A Run Screening Frequency	Steady State Run Frequency[4]
Sulfate	E-1 discharge	5 per E:A	6 hours of run time
Sulfate	S1-S4	n/a	24 hours of run time
Sulfate	SO4 product tank	n/a	Prior to emptying of tank
TOC	E-1 discharge	Triplicate per E:A	24 hours
TOC	SO4 product tank	n/a	Prior to emptying of tank
Extractant strength	S4	n/a	Once per extractant turnover (~40 hours)

Winner will complete all required sample analyses and will provide results to Battelle in an excel file sent on a weekly basis. Winner will utilize a sample numbering/tracking system, approved by Battelle, to ensure accurate tracking and analysis of samples. This tracking system is outlined below:

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[3] Subject to modification based on ability to achieve required sulfate concentrations.

[4] Subject to modification based on system variability.

Logbook Describing Sample- Logbook Page Describing Sample - Sample Number on that page – Stage Sampled – Phase (E or A)

For example, if an aqueous sample from S4 is described in logbook one, page 30, and is the fifth sample on that page, it would be labeled as 1-30-5-S4A.

Additional samples may be requested by Battelle at any point in the test campaign. Should a process upset occur, an additional round of samples will be taken to help diagnose the upset condition. Analysis of these samples will not be conducted until a possible explanation of the process upset is made.

An operators log will be maintained for the duration of TC1, tracking events such as major setpoint adjustments, process upsets, sampling times/locations, system operators, and other events which may be relevant to the final results analysis. This log will be made available to Battelle on a weekly basis.

The control system database history will be recorded in the event of a process upset, and may be requested by Battelle at any time given 24 hours' notice.

Appendix C: (Confidential – not included in this document)

Appendix D: Selected Stakeholder Meeting Minutes

Project Update:
Development and Validation of an AMD Treatment Process for Source Water Generation

Provided to Stakeholder Group Members
May 27, 2015

Purpose of Update Meeting

A stakeholder group update meeting was held on April 29, 2015 to update the project stakeholders on the results achieved during the first phase of the project as well as to present the plans for the pilot testing planned in the second phase. Revised preliminary results of the water quality produced by the HydroFlex platform were shared.

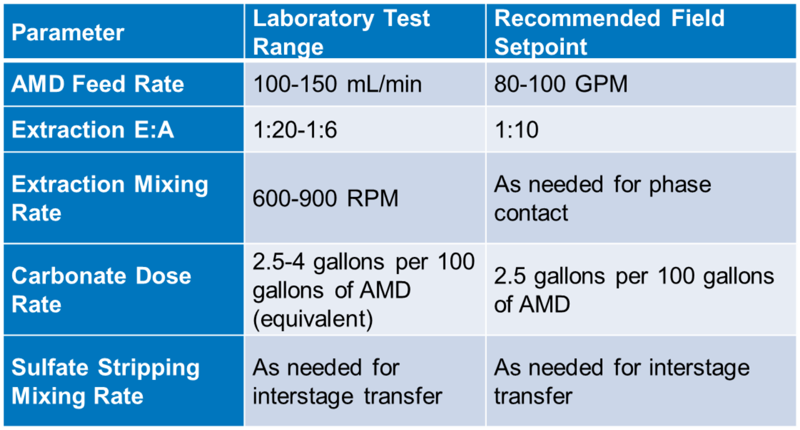
Meeting Summary

The attendees available to participate in the stakeholder kickoff meeting are listed in the table below.

Name	Organization
Mike Heinrichs	Battelle
Annie Lane	Battelle
Kurt Blohm	Battelle
Nan Zhang	Statoil
Dave Leiford	PADEP Conservation and Restoration
Joe Adams	PADEP O&G Program
Brent Means	Office of Surface Mines
David Cercone	DOE NETL Program Manager

At the meeting, Battelle presented the laboratory efforts conducted during Phase 1. During this phase, Battelle performed continuous testing with its bench scale system to determine the optimal operating parameters for steady state operation of the process. Samples were collected during this testing to evaluate the metal and sulfate removal, as well as the byproduct quality and extractant regeneration efficiency. Laboratory results indicated that sulfate removal within the range of that acceptable to industry for use as source water can be achieved. Additionally, the laboratory results helped to generate process variable setpoints used in the field. The primary process variables tested in the lab, along with their translation to the field unit, are shown in Table 1.

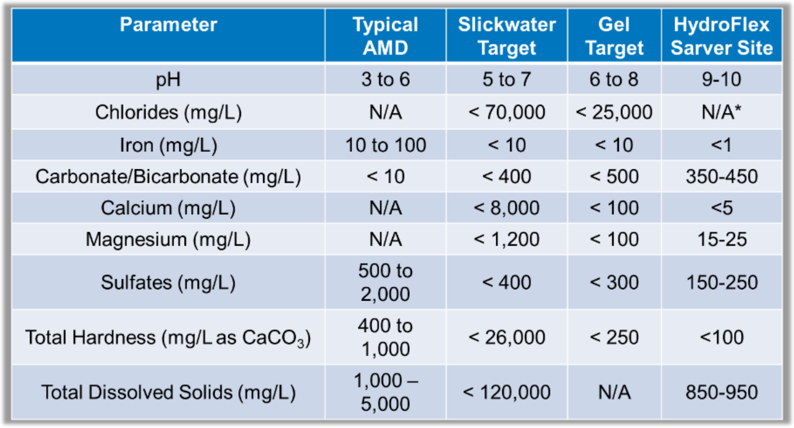
Table 1: Selection of process variables tested in the laboratory and their scaled field counterparts



Parameter	Laboratory Test Range	Recommended Field Setpoint
AMD Feed Rate	100-150 mL/min	80-100 GPM
Extraction E:A	1:20-1:6	1:10
Extraction Mixing Rate	600-900 RPM	As needed for phase contact
Carbonate Dose Rate	2.5-4 gallons per 100 gallons of AMD (equivalent)	2.5 gallons per 100 gallons of AMD
Sulfate Stripping Mixing Rate	As needed for interstage transfer	As needed for interstage transfer

Battelle presented data from continuous runs of its field shakedown testing conducted in Fall 2014. At flows of 40-50 gpm, the field demonstration achieved sulfate removal similar to that observed in laboratory testing. These data were used to update the projected water quality characteristics expected of the fully optimized HydroFlex platform which will be tested during the second phase of the project. These updated water quality characteristics are shown in Table 2 below.

Table 2: Revised preliminary water quality characteristics.



Parameter	Typical AMD	Slickwater Target	Gel Target	HydroFlex Sarver Site
pH	3 to 6	5 to 7	6 to 8	9-10
Chlorides (mg/L)	N/A	< 70,000	< 25,000	N/A*
Iron (mg/L)	10 to 100	< 10	< 10	<1
Carbonate/Bicarbonate (mg/L)	< 10	< 400	< 500	350-450
Calcium (mg/L)	N/A	< 8,000	< 100	<5
Magnesium (mg/L)	N/A	< 1,200	< 100	15-25
Sulfates (mg/L)	500 to 2,000	< 400	< 300	150-250
Total Hardness (mg/L as $CaCO_3$)	400 to 1,000	< 26,000	< 250	<100
Total Dissolved Solids (mg/L)	1,000 – 5,000	< 120,000	N/A	850-950

. **Note-chlorides are not present in AMD influent stream.*

Battelle then discussed the schedule for Phase 2 of the project which includes field demonstration of the system and evaluation of the process byproducts. Two test campaigns are planned: Test Campaign 1, which will test the effect of operating parameters on sulfate removal, and Test Campaign 2 to optimize the process to reduce operating expenses while meeting water quality requirements.

Questions and Answers

Several questions were raised and discussed with the attendees during the stakeholder group kickoff meeting:

- Brent Means of the Office of Surface Mines requested that Battelle provide the cost and amount of the sodium carbonate solution used in the process:
    - The cost of the sodium carbonate solution delivered to the field demonstration is approximately **$0.30/lb** at a concentration of 98% wt/wt. Note that this price reflects delivery of the carbonate in the bagged form used for convenience during the pilot demonstration.

- Nan Zhang of Stat-Oil requested that Battelle provide the composition of the process byproduct stream, which is shown in Table 3 below:

Parameter	Sodium Sulfate Byproduct (mg/L)
Appearance	Clear aqueous solution
Odor	Mild solvent odor
Total Solids	8-10 wt%
Carbonate, as Na2CO3	5-6 wt%
Sulfate, as Na2SO4	4-5 wt%
pH	10.5-11.5 wt%

Table 3: Sodium sulfate byproduct composition. Note that composition is expected to change during the optimization runs conducted during the test campaigns.

Next Steps

We anticipate beginning the first of the test campaigns in mid June. The system will be operated on a continuous basis to gather long term process data. The next stakeholder update meeting will be held July 29th, 2015, at which point we plan on presenting data gathered during the test campaigns executed to date.

Attachments: (1) Byproduct Spec Sheet, (2) Stakeholder update meeting presentation.

Appendix E: Selected Stakeholder Project Update Communications

Project Update:
Development and Validation of an AMD Treatment Process for Source Water Generation

Provided to Stakeholder Group Members
October 6, 2015

Background

Battelle is currently executing the second budget period of a $1,125,000 project (DE-FE-0014066) sponsored by the U.S. Department of Energy (DOE) and Pennsylvania Department of Community and Economic Development (PA DCED). The project objectives include demonstration of Acid Mine Drainage (AMD) remediation using the HydroFlex™ technology to meet water quality requirements for reuse in hydraulic fracturing and evaluation of the process byproducts for use in flowback water treatment. Accordingly, the Budget Period 2 activities include field testing operations to validate the ability of the technology to achieve the desired water quality and comparative testing of the process byproducts with commercially available water treatment chemicals.

Field Test Campaigns

The original field testing schedule was delayed by several weeks due to low sulfate concentrations in the acid mine drainage source at the demonstration site. Testing started on August 3, 2015, once an influent stream with sulfate concentration between 500 and 600 ppm was achieved.

The first test campaign was comprised of two steps. During the first week of operation, the Extractant to Aqueous (E:A or E/A) ratio of the system was varied to determine its impact on sulfate removal. Once an optimum E:A ratio was identified, resulting in the targeted sulfate removal performance, the system was operated for three weeks under steady state conditions.

E:A Ratio Evaluation Discussion

The system was operated at a single E:A ratio each day for the first week of operation. Sulfate samples were pulled after several hours of run time at each E:A ratio. Figure 1 shows the sulfate extraction performance observed at each E:A ratio. As expected, at higher E:A ratios, where more extractant is present relative to the water, more sulfate was extracted.

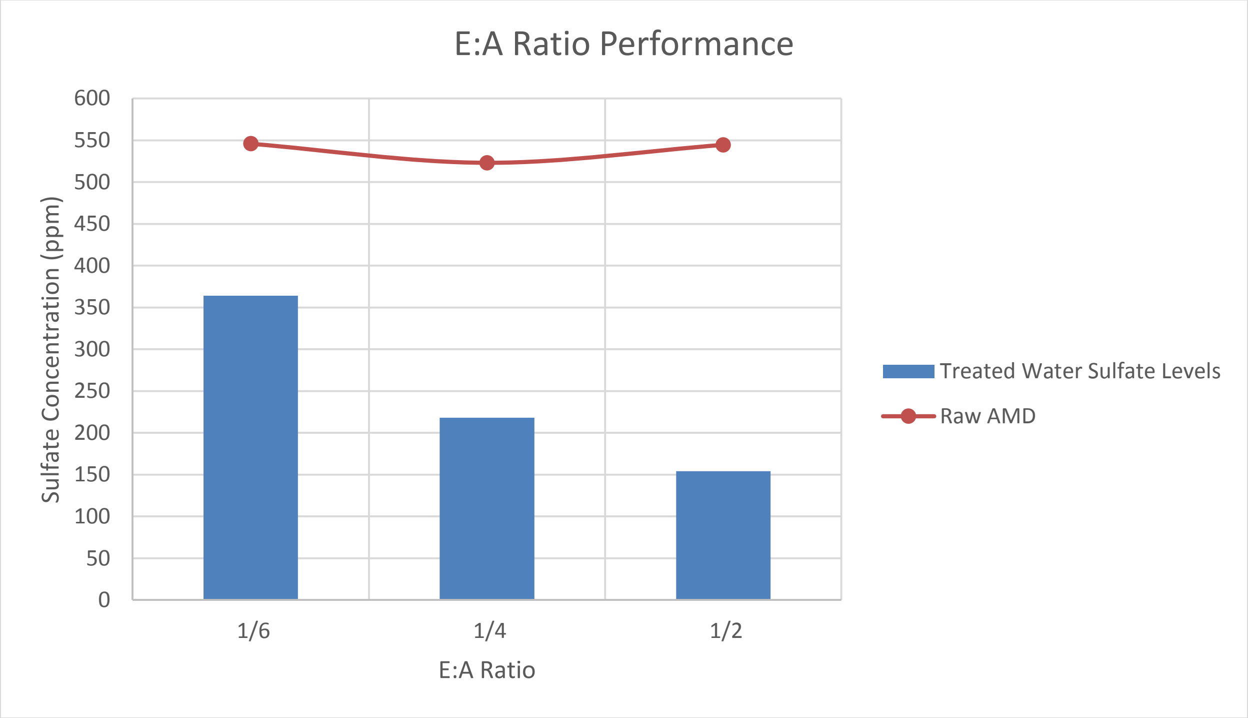


Figure 1: Comparison of E:A ratios evaluated during the first week of the test campaign.

In general, the lowest E:A ratio capable of providing the sulfate removal required is optimal, as a smaller overall volume of extractant is required to treat a given water source, resulting in lower capital and operating expenses. From the run data collected, it was determined that a 1:2 E:A ratio would be required to reduce sulfate to the required levels. This E:A ratio differs from the 1:10 E:A ratio previously used during field test runs conducted during the first budget period. It is believed that the increased E:A ratio is required due to degradation of the extractant over a prolonged period of system operation. During this temporary pilot demonstration, steps to maintain the extractant formulation were not incorporated into the process. Costs associated with the maintenance of the extractant will be included in operating cost projections.

Steady State Run Discussion

The system was operated for approximately 150 hours, processing nearly 500,000 gallons of AMD, to gather information about the system during steady state operation at the 1:2 E:A ratio. During the three weeks of run time, the feed to the HydroFlex process was maintained, on average, between 500 and 600 mg/L sulfate. For the majority of the test campaign (approximately 120 operational hours), operation of a single extraction stage was able to achieve a sulfate level of below 150 mg/L. This equates to a sulfate removal of an average of 403 mg/L or a 74% reduction in sulfate content. Figure 2 shows the sulfate concentration of influent AMD stream and the process discharge stream.

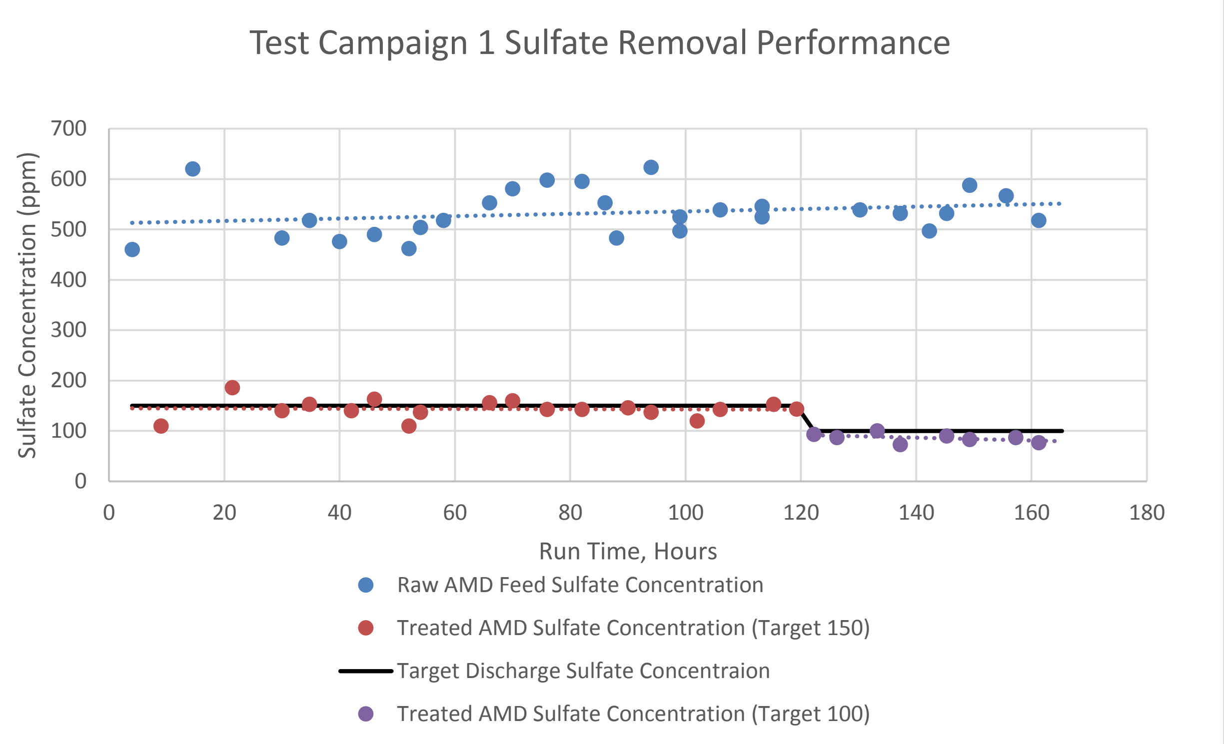


Figure 2: AMD feed sulfate concentration and treated water sulfate concentration for both 150 mg/L and 100 mg/L targets.

After approximately 120 hours of operation, the process conditions was altered to achieve sulfate levels below 100 mg/L per the guidance of our stakeholder committee. A second extraction stage (E2) was brought online to provide additional sulfate removal capability. The impact of the second extraction stage is illustrated by the step change in the sulfate removal targets shown in Figure 2 **Error! Reference source not found.**. Under these conditions, sulfate was consistently reduced to levels below 100 mg/L, removing an average of 448 mg/L of sulfate or a reduction of sulfate by 85%.

Test Campaign 1 Conclusions

Data from the first test campaign indicates that sulfate levels are effectively reduced to target concentrations with the HydroFlex platform. An initial screening of the E:A ratio effect on sulfate removal performance indicated that an E:A approaching 1:2 is necessary to achieve the required sulfate levels under the Sarver, PA site conditions . A lower E:A ratio is anticipated to be sufficient to achieve similar sulfate removal when the extractant has been continuously maintained, as evidenced by the results from the demonstration runs conducted in BP1 with fresh extractant.

Operation with a second extraction stage achieves additional sulfate removal, resulting in the reduction of the sulfate levels to below 100 mg/L during the test campaign. Test Campaign 1 demonstrated long term, steady state system behavior. Over TC1, sulfate removal consistently improved throughout the entire test campaign, indicating that improved sulfate numbers may be realized by operation of the system over a longer period of time. Table 1 summarizes the water quality information for water treated during the field test campaign.

Table 1: HydroFlex process water quality compared to projected water requirements for source water.

Parameter	Sarver Site AMD	Slickwater Target	Gel Target	HydroFlex Sarver Site
pH	8-9	5 to 7	6 to 8	9-10
Chlorides (mg/L)	N/A	< 70,000	< 25,000	N/A*
Iron (mg/L)	<1	< 10	< 10	<1
Carbonate/Bicarbonate (mg/L)	< 10	< 400	< 500	350-450
Calcium (mg/L)	30-60	< 8,000	< 100	<5
Magnesium (mg/L)	20	< 1,200	< 100	20
Sulfates (mg/L)	550-650	< 400	< 300	<100
Total Hardness (mg/L as $CaCO_3$	<100	< 26,000	< 250	<50
Total Dissolved Solids (mg/L)	600-800	< 120,000	N/A	500-700

Preliminary Process Economics

Preliminary process economics were estimated at the conclusion of TC1. The process operating costs are driven by chemical consumables, waste disposal, electrical utility costs, and labor costs. For the process economic analysis, we examined two separate scenarios: operational costs derived from TC1 data, and projected operational costs which include anticipated operational conditions of a production scale environment. For example, in a commercial process the cost of carbonate may be much lower when purchased in bulk. Inputs to the operational cost model for both TC1 and projected operation are shown in Table 2. Table 3 shows the treatment cost per thousand gallons associated with each operational parameter. It should be noted that the prices presented do not contain any potential income associated with sale of the water or process byproducts.

Table 2: Inputs for TC1 and projected operating cost model.

Cost Analysis Inputs			
Parameter	**TC1 Rate**	**Projected Rate**	**Basis of Estimate**
Carbonate Cost	$0.285/lb	$0.20/lb	Bulk chemical vendors
Carbonate Flow	1.7 GPM	1 GPM	TC1 and Laboratory Results
Extractant Cost	$7.75/lb	$7.75/lb	Provided by WWS
Extractant Makeup	0.021 lb/min	0.010 lb/min	TC1 and Laboratory Results
Filter Replacement Rate	36 kgal/filter	36 kgal/filter	Provided by WWS
Waste Disposal Cost	$0.30/gal	$0.00/gal	Provided by WWS
Waste Gen. Rate	1.7 GPM	1 GPM	TC1 and Laboratory Results
Electricity Cost	$0.1019/kW-hr	$0.0714/kW-hr	EIA Electrical Cost Estimates
Labor Cost	n/a	$25/hr	*Plant Design and Economics for Chemical Engineers,* Peters et.al.

Table 3: Operating cost per thousand gallons for TC1 and projected operating conditions.

Projected Production Economics		
Parameter	**TC1 Cost/barrel**	**Projected Cost/barrel**
Carbonate Cost	**$0.22**	**$0.12**
Extractant Cost	**$0.14**	**$0.06**
Filtration Cost	**$0.03**	**$0.03**
Waste Disposal*	**$ -**	**$ -**
Electricity Cost	**$0.01**	**$0.01**
Labor Cost**	**$0.03**	**$0.03**
Total/barrel	**$0.43**	**$0.25**

* Note that in these cases wastes were assumed to be dispositioned at no cost for processing of produced water.
**Additionally, since the research level staff loading in TC1 is not expected to reasonably represent a commercial installation, the projected commercial labor costs were inserted for TC1 costs.

The primary cost driver associated with the cost to treat AMD in the first test campaign is the waste disposal cost. This is the cost associated with disposing of the process byproduct, a sodium sulfate solution. One of the remaining tasks of this project is dedicated to determining avenues of use for this byproduct stream, converting it from a revenue-depleting waste stream to a positive revenue source for the process. Produced water treatment companies have shown interest in learning more about how the HydroFlex process byproduct stream can be used to treat produced water. Therefore, in the projected process economics model, we have conservatively assumed that the byproduct can be dispositioned at no cost.

Another large cost driver is the minor losses of extractant in the effluent water. Despite being a parts per million level loss, the replacement cost is significant, representing an additional $0.14 to the price per barrel of treated water. In a production system, it is anticipated that the process will include enhancements to 1) reduce the losses of extractant, and to 2) provide a constant, low volume makeup stream of the extractant to the process to ensure that it is continually regenerated and able to extract sulfate as efficiently as possible. Planned improvements to the process to optimize the extractant use while minimizing these losses including further optimization system design and potential pretreatment steps to enhance phase disengagement after sulfate removal steps.

Filtration costs to remove carbonate solids to preserve pump performance are also included in this application. It is possible that selection of a different pump design could avoid this cost; however, filtration is included in both the TC1 results and the projected costs to be conservative. Electricity cost projections were also lower than observed in TC1 since the current operation incurred peak demand charges that elevated the price of electricity above the industrial average, which is what was used for the projection.

From previous conversations with stakeholders who utilize source water for HF purposes, water is typically purchased for $0.12-0.50 per barrel. This indicates that under the assumptions used in this economic exercise economic operation of the HydroFlex process is possible. However, based on the data collected during TC1, several process optimizations will need to be made to achieve this end price point.

Requested Feedback

In order to ensure that data reported to the DOE is relevant to the current industry status, **Battelle is requesting feedback from its project stakeholders regarding the quality of water produced by the HydroFlex process, its ability to be used in HF operations, and the projected process economics.** Please send any feedback questions on this report via email or phone to Mike Heinrichs, (heinrichsm@battelle.org; 614-424-5732). The deadline to provide feedback is October 23rd, 2015.

Next Steps

As field testing associated with this DOE project concludes, Battelle is finalizing its analysis of the results and preparing the final project report. The report is scheduled to be delivered to the DOE by December 31st, 2015, and is expected to be made public after its acceptance. Winner Water Services, the owner and operator of the HydroFlex technology, is continuing its efforts to commercialize this technology in several water treatment applications.

Appendix F: Cost Information Back-Up

Appendix F: Cost Information Back-Up

Costs associated with HydroFlex process inputs were determined through measurement of key process input variables, including chemical costs, material consumables, and electrical requirements. Once consumption rates were determined, contributions to the overall process operating cost were determined for each of the process inputs. This appendix presents the calculations used to determine individual components of the overall system operating cost.

Carbonate Costs

The costs for carbonate fed to the process are described below. During steady-state operation during TC1 and the CTC, carbonate feed rates and solution concentrations were measured. Carbonate costs for TC1 and the CTC reflect the cost of bagged carbonate solid used during this demonstration. The ideal carbonate case uses flow rates identified using stoichiometric relationships associated with the extraction chemistry, and assumes discounted carbonate solid costs associated with procurement of carbonate in bulk form.

Carbonate TC1		
Sodium Carbonate Concentration, wt%		4.8%
Sodium Carbonate Cost, Dry, $/lb		0.285
Sodium Carbonate Flowrate, gpm		2.0
5% Sodium Carbonate SG		1.065
Dry Sodium Carbonate Flow, lb/min		0.844
Dry Sodium Carbonate Cost, $/min	$	0.24
Sodium Carbonate Cost per kgal	$	5.34

Carbonate CTC		
Sodium Carbonate Concentration, wt%		7.0%
Sodium Carbonate Cost, Dry, $/lb		0.285
Carbonate Flowrate, gpm		0.73
7.5% Sodium Carbonate SG		1.075
Dry Sodium Carbonate Flow, lb/min		0.457
Dry Sodium Carbonate Cost, $/min	$	0.13
Sodium Carbonate Cost per kgal	$	2.89

Carbonate (Projected Ideal)		
Sodium Carbonate Concentration, wt%		8.0%
Sodium Carbonate Cost, Dry, $/lb		0.245
Carbonate Flowrate, gpm		0.63
8% Sodium Carbonate SG		1.0816
Dry Sodium Carbonate Flow, lb/min		0.455
Dry Sodium Carbonate Cost, $/min	$	0.11
Sodium Carbonate Cost per kgal	$	2.48

Extractant Costs

A portion of the costs associated with the treatment of the AMD are associated with providing a makeup stream of extractant to the process to ensure extractant strength is maintained. During TC1, GC analysis identified that extractant makeup may be able to be accomplished by dosing only the active ingredient to the process, as it appeared to be preferentially lost. The cost associated with this active ingredient is $7.75/lb. During the CTC, a second round of analysis indicated that the extractant blend appeared to be lost equally. Therefore, for CTC and production system cost estimates, the cost of bulk extractant was used in the cost estimates ($27/gal). Additionally, bulk extractant is easier to dose to the process than just the active ingredient due to physical properties of the materials.

Extractant Makeup, TC1		
Makeup Rate (lb/min)		0.031
Extractant Cost, $/lb	$	7.75
Extractant Cost, $/min	$	0.24
Extractant Cost per kgal	$	5.34

Extractant Makeup, CTC		
Makeup Rate (gal/min)		0.00325
Extractant Cost, $/gal	$ 27.00	
Extractant Cost, $/min	$ 0.09	
Extractant Cost per kgal	$ 1.95	

Extractant Makeup, Production		
Makeup Rate (gal/min)		0.00069
Extractant Cost, $/gal	$ 27.00	
Extractant Cost, $/min	$ 0.02	
Extractant Cost per kgal	$ 0.41	

Filtration Costs

To protect pumps and process equipment from calcium carbonate solids which precipitated in the high pH environment of the extraction vessels, a bag filter skid was installed for TC1. During the CTC, existing centrifugal pumps were replaced with diaphragm pumps which were able to withstand solids. Therefore, the filter skid was removed during the CTC, and associated bag replacement costs were removed from the cost estimates. Consumption rates were determined through measurements in the field, and were determined to be 4 bags per 12.5 hours of operation.

Filters, TC1	
Cost per bag	7.2
Consumption, bags/hr	0.32
@ Flow Rate gpm	45
gph	2700
$/kgal	$ 0.85

Byproduct Disposal Costs

Byproduct disposal costs are shown in the tables below for TC1 and the CTC. Representative flow rates were used for TC1 and the CTC. During the CTC, the flow rate of byproduct was reduced due to the reduction in carbonate solution fed and better utilization of carbonate on the system. Updated waste hauler costs were incorporated into the CTC costs. Byproduct disposal costs are assumed to be negligible in a production system, with the sodium sulfate solution either sold at a profit for other purposes, or disposed of on-site or in a WWTP at minimal cost.

Waste Disposal, TC1		
Waste Flow Rate, gpm		1.53
Waste Disposal Cost, $/gal	$	0.30
Waste Disposal Cost, $/min	$	0.46
Waste Cost per kgal	$	10.20

Waste Disposal, CTC		
Waste Flow Rate, gpm		0.80
Waste Disposal Cost, $/gal	$	0.27
Waste Disposal Cost, $/min	$	0.21
Waste Cost per kgal	$	4.70

Electricity Costs

Electrical costs were determined through measurement of the energy consumed by the plant during operation. The electrical rate used at the Sarver demonstration site was $0.1017 kW-hr, while the rate assumed for a production system is a typical industrial sector rate ($0.0714 kW-hr).

Power, TC1		
Power Rate ($/kW-hr)	$	0.10
Power Used (kW)		7.8
Water Treated (gal)		592860
Averaging Duration (hrs)		205.8
Electrical Op Cost ($/1000 gal)	$	0.28

Power, CTC		
Power Rate ($/kW-hr)	$	0.10
Power Used (kW)		7
Water Treated (gal)		36000
Averaging Duration (hrs)		12
Electrical Op Cost ($/1000 gal)	$	0.24

Power, Projected		
Power Rate ($/kW-hr)	$	0.07
Power Used (kW)		7
Water Treated (gal)		36000
Averaging Duration (hrs)		12
Electrical Op Cost ($/1000 gal)	$	0.17

Labor Costs

Labor costs for the system were assumed to be $25/hr, referenced from Peters and Timmerhaus *Plant Design and Economics for Chemical Engineers.* It is assumed that a single operator would be on duty for 2 hours per day to observe the system operation and take required samples. A production system would be more automated than the demonstration system operated in Sarver, PA. The labor cost per thousand gallons was determined by dividing the total operator cost per day ($25 x 2 = $50) and dividing it across the amount of water processed in that time frame (100 GPM x 60 min/hr x 12 hr/day).

Appendix G: Technology Introduction

Appendix G: Technology Introduction

The HydroFlex technology is based on well understood and widely applied solvent extraction processes. Solvent extraction has been practiced in the mining industry for decades to recover copper, uranium, and other metals from leach solutions. In the process, an organic extractant phase is used to pull metals or other charged compounds from the aqueous solution into an organic phase by mixing. The organic phase then splits from the aqueous phase in a settler, and is drawn, along with the metals, into a separate stripping section. Typically in this section, the organic phase is contacted with an aqueous stream that has higher or lower pH than the leach stream, which causes the metals to be stripped back into an aqueous solution. High recycle rates in the stripping section allow for metals or other contaminants to be concentrated many times over to a recoverable level. The stripping section also regenerates the organic phase such that it can be recycled in the process, reducing cost and waste.

Process Description

Figure 1 represents the treatment steps employed by the technology—water purification and sulfate recovery. In addition to the treated AMD water, a process byproduct is produced by the process; sodium sulfate. The byproducts will either be used in water treatment processing or disposed of according to applicable waste disposal requirements.

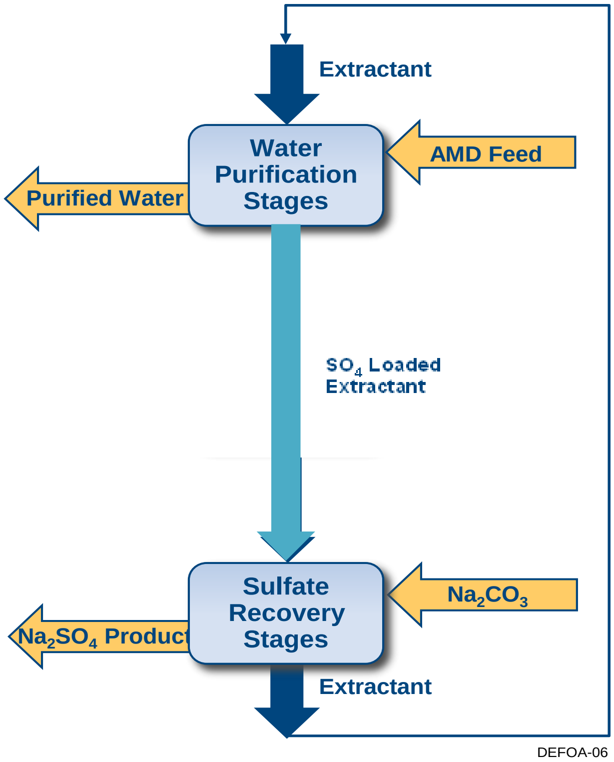


Figure 1: HydroFlex process overview, as configured for Sarver demonstration.

Water Purification Stages

The extractant and AMD water are combined in the mixers of the water purification stages, where the extraction process takes place. The mixture is transferred to an oil-water separator, where the organic extractant rises to the top, is collected in an oil overflow well, and is sent to the next stage. The AMD water then flows to the next stage if multi-stage operation is selected (four water purification stages are available total) and then to a collection tank. Note that in the testing conducted at Sarver, only one or two of the extraction stages were used due to the low concentration of sulfate in the AMD stream. Skid E3 was unused, and skid E4 was used as a polishing tank providing additional phase break time. Figure 3 shows the extraction mixer and separator stages.

The AMD water flows counter-current with extractant in the following manner:

1. AMD (from the outfall of the existing treatment system, see Figure 2) is pumped to mixer #1 (skid E-1), and combined with extractant from separator #2 (skid E-2, when two E-stages are in operation)
    a. Mixture flows from mixer #1 to separator #1 (skid E-1) for separation
    b. Extractant separates from the aqueous phase and is pumped to sulfate recovery equipment (see next section)
    c. AMD is then pumped to mixer #2 (skid E-2)
2. AMD combined in mixer #2 with extractant (regenerated extractant from sulfate recovery step – see section below)
    a. Mixture flows to separator #2 for separation
    b. Extractant is sent to mixer #1
    c. AMD is then pumped to mixer #4 (skid E-4)
    d. Treated AMD flows to E-4 stage for additional phase break time, then to transfer tank; pumped through granulated activated carbon filter (see Figure 4) and on to storage tanks for beneficial use, or reintroduced to the infall of the existing treatment system when no user is available or excess exists.

Figure 2: Inlet pump for AMD from outfall of existing treatment pond.

E-1 effluent:

AMD to GAC or E-2, extractant to E-2 or S-1, depending on configuration.

Separator (skid E-1)

Mixer (skid E-1):

AMD from influent,

Extractant from S4 or E2, depending on configuration.

Figure 3: Extraction skids, showing mixer tank and separator.

Figure 4: Granular Activated Carbon (GAC) beds used to polish effluent water. Beds were operated in series.

Sulfate Recovery Stages

After the extraction process, the spent extractant is treated in a sulfate recovery system. The sulfate recovery system is a four stage process that involves adding potassium or sodium carbonate to extractant that has gone through the metal recovery process in order to recover the sulfate as potassium or sodium sulfate. The carbonate is added to the extractant (in a volume contingent on the pH of the stages) and the mixture is pumped to a separator for separation. The extractant rises to the top of the separator, and is pumped to the next reaction tank (four stages total). Figure 5 shows the flow path through the sulfate stripping stages.

The spent extractant flows counter-current with carbonate in the following manner (see design drawings):

1. Spent extractant is pumped to mixer #1 (skid S-1), and combined with carbonate from separator #2 (skid S-2)
    a. Mixture flows from mixer #1 to separator #1 (skid S-1) for separation
    b. Extractant is skimmed off and sent to the next stage
    c. The resulting sulfate rich aqueous phase is collected in storage tanks for beneficial use
2. Spent extractant is pumped to mixer #2 (skid S-2), and combined with carbonate from separator #3 (skid S-3)
    a. Mixture flows from mixer #2 to separator #2 for separation
    b. Extractant is skimmed off and sent to the next stage
    c. The resulting aqueous phase containing carbonate and sulfate is sent to mixer #1
3. Spent extractant is pumped to mixer #3 (skid S-3), and combined with carbonate from separator #4
    a. Mixture flows from mixer #3 to separator #3 for separation
    b. Extractant is skimmed off and sent to the next stage
    c. Resulting aqueous phase containing carbonate and sulfate is sent to mixer #2
4. Spent extractant mixture is pumped to mixer #4 (skid S-4), and combined with fresh carbonate
    a. Mixture flows from mixer #4 to separator #4 (skid S-4) for separation
    b. Extractant is skimmed off and flows to a collection tank for reuse in AMD extractant contactor/separator process
    c. Resulting aqueous phase containing carbonate and sulfate is sent to mixer #3

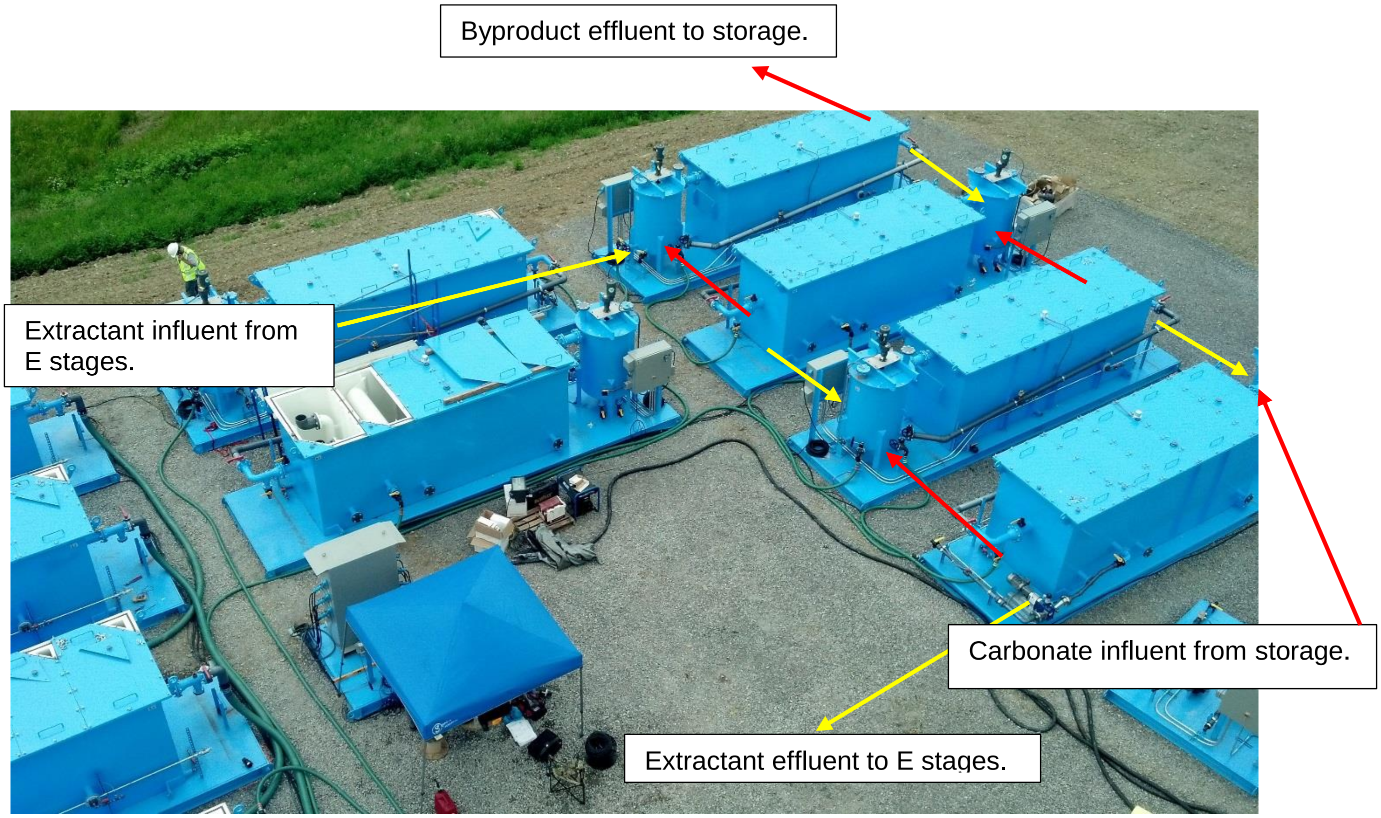


Figure 5: Sulfate stripping stages (right) showing flow path. Yellow arrows show extractant flow, Red arrows indicate carbonate flow.

Process Support Equipment

The on-site inventory of raw material chemicals and the expected byproducts from the process will be stored in polypropylene tanks.

Feed chemical storage includes:

- Two 7,000 gallon poly tanks for storing 8% sodium carbonate solution. This equates to approximately 4 days of storage at design operating conditions (see Figure 6).

The by-product quantities that will be stored on site are based on 3 days of storage for 16 hr/day operation. By-product storage includes:

- Two interconnected 7,000 gallon poly tank, manufactured per ASTM 1998-06 to comply with Pennsylvania residual waste requirements, for storing sodium sulfate solution (<20% solution) from the process (see Figure 7).

Two tanks will be used to hold polished AMD and excess extractant volume (see Figure 8).

Figure 6: Storage for carbonate solution fed to stripping stages.

Figure 7: Storage tanks for process byproducts.

Figure 8: Polished AMD tank (T1, left), and extractant overflow tank (T2, right).