# **Oil & Natural Gas Technology**

### DOE Award No.: DE-FE0001466

# Quarterly Report 2 (1/1/10 - 3/31/10)

#### Zero Discharge Water Management for Horizontal Shale Gas Well Development

#### Submitted by:

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## **Executive Summary:**

Major effort this quarter was devoted to testing technologies needed to produce a water quality suitable for the next fracture treatment. Particulates greater than approximately 3 microns were completely removed from one Marcellus raw water sample by the FilterSure PDU now installed at the WVU high bay area. A second Marcellus sample was shipped to a provider of Electrical Coagulation (EC) technology for testing. The EC treated water was returned to WVU and, after filtering, produced a visually clear liquid. The initial results suggest that EC followed by filtration will adequately condition the water for recycle. Water chemistry tests now underway will determine if additional downstream treatment is needed to create a process that can be implemented commercially. Setup costs and the costs of two complete filtration tests have largely exhausted the funds made available for year 1 programs. FilterSure has therefore suspended further testing until the balance of the Phase I funds are made available. These funds are required to support a continuous process demonstration at the University that links the EC and the FilterSure technologies to verify performance and to establish process economics.

## Progress Report-Phase I

This progress report covers the progress of the Phase effort from 1/1/10 to 3/31/10. The report is organized by task as listed in the Statement of Project Objectives.

### PHASE I /Budget Period 1

#### Task 1.0 – Project Management and Planning

Work planned in the prior quarter was initiated. A chemical hygiene plan was developed for both the PDU filtration and the bench-scale work. Both have been approved by the NRCCE Facilities Manager, the NRCCE Chemical Hygiene Officer, and WVU Environmental Health and Safety. A Technology Status Report was compiled and submitted.

In lieu of an Industry Contact Group meeting, we developed and sent a detailed questionnaire to the members of our Industry Contact Group in order to gain more information in a shorter period of time. We have received responses from four producers, and anticipate receiving more. This has provided extremely useful information regarding water volumes and parameters necessary for recycling of frac water. In addition, we met with representatives from a company in our Industry Contact Group to show the PDU, and discuss plans for obtaining data and water samples from wells around the region, as well as possibilities for deployment of the Mobile Treatment Unit in Phase II of the project.

The 2-GPM FilterSure Process Development Unit (PDU) was prepared for shipment from its New Jersey location to WVU. The 1,600-pound PDU was delivered in February and testing was initiated. Test results were reviewed at a March project planning meeting held at the University. These results were sufficiently encouraging (discussed in Task 2.4) to warrant testing Electro Coagulation (EC) as a second component of a commercial process train. Members of the project team toured a Marcellus Shale gas well site, and obtained a large volume (130 gallon) frac water sample from a member of our Industry Contact Group. Arrangements were made to have the water sample shipped to an EC manufacturer for treatment. Plans were made to have the EC treated water returned to the University for processing through the FilterSure PDU.

#### Task 2.1 Develop Conceptual Process Train

A conceptual process train is being developed around the FilterSure technology and equipment needed to support this technology. With the currently available data (summarized in Task 2.4) it appears that the EC followed by the filter will produce water with virtually no solids. The need for subsequent processing to reduce Total Dissolved Solids (TDS) will depend on the effluent water chemistry. Treatment may not be necessary if the effluent TDS can be diluted to a point where it can be recycled for the next frac job.

Acceptable recycle water quality has been identified in industry responses to the questionnaire provided by our Industry Contact Group. Information provided by Group members contains water chemistry requirements for recycling flow back water. Members of the Industry Contact Group also supplied frac return-water samples from shale wells in the region, and more water samples have been promised. We are processing these samples to obtain water chemistry and suspended solids characteristics.

Collecting a representative frac water sample proved to be a key issue and was subject to some debate. Did we need samples of frac flow-back at different times (e.g., early, middle, *and* end of the flow-back)? Should samples be obtained directly from the flow-line or at some other point? After discussing this issue with industry personnel, we concluded that evaluating flow-back samples over time was impractical and unnecessary. The most practical approach is to sample only the water that would likely be treated; i.e., water from the frac return-water storage tanks or pits.

Recycle water quality criteria have been established and will be vetted with our Industry Contact Group. Solids removal efficiency has been established by laboratory testing of the Marcellus samples we have received. This information quantifies the volume of solid materials that will need to be managed during commercial operations, a critical variable in developing a conceptual process train.

#### Task 2.2 Develop Process Flow Model:

Initial results suggest that EC followed by filtration will adequately condition the water for recycle from a solids loading perspective. The ability to remove heavy metals and other targeted materials will be known from water chemistry tests currently underway. These tests will largely determine if additional downstream treatment is needed to model a process flow that can be implemented commercially.

#### Task 2.3 Identify Recycling Operational Requirements

Recycling operational requirements are being evaluated using the data from the industry questionnaire responses. These data and the water chemistry results will provide the technical data needed to address these requirements.

#### Task 2.4 Develop and Test Treatment Methods

#### Cost of Water Treatment

A program that calculates the unit costs of various treatment methods is being updated by the addition of some new methods (such as the FilterSure system and EC) and current prices. Table 1 shows costs per 1000 gallons for a number of treatments, including EC. The EC method appears to be competitively priced among the various options.

Figures 1 – 3 detail some initial investigations comparing nanofiltration, which preferentially separates divalent ions, and reverse osmosis. These are two proposed technologies for TDS removal. The graphs compare cost versus process efficiency, cost versus flow, and cost versus electricity cost. In all instances nanofiltration is cheaper because of its lower pressure requirements. The rise in cost at low efficiency for the first plot is due to increasing reject disposal costs. Low flow also increases the cost due to the fixed costs associated with the system. The increase in cost due to electricity rates is almost linear. Improved data are being added to the database.

Table 1. Costs of various treatment methods

Treatment Method	\$/1000 gallons
Surface disposal	\$0.07
Deep injection well - existing	\$0.66
Evap/infil pond w/ spray	\$0.99
Spray Irrigation	\$1.08
Microfiltration	\$1.36
Evaporative pond - Lined-Spray	\$1.97
Electrocoagulation	\$2.00
Shallow injection/aquifer renewal	\$2.85
Evaporative pond/infiltration	\$2.98
Water hauling	\$4.82
Deep injection well - new	\$5.64
Nanofiltration	\$6.15
Reverse Osmosis	\$6.94
Evaporative pond - Lined	\$27.56

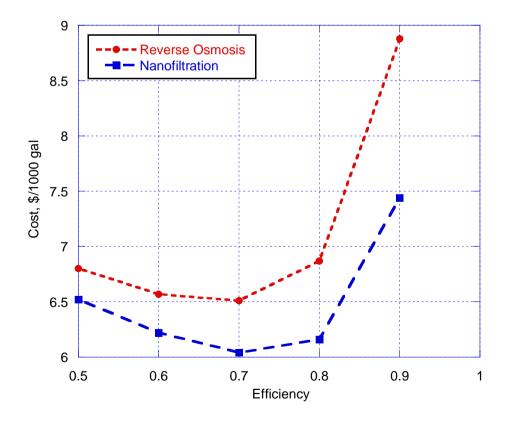


Figure 1. Comparison of cost and efficiency between nanofiltration and reverse osmosis

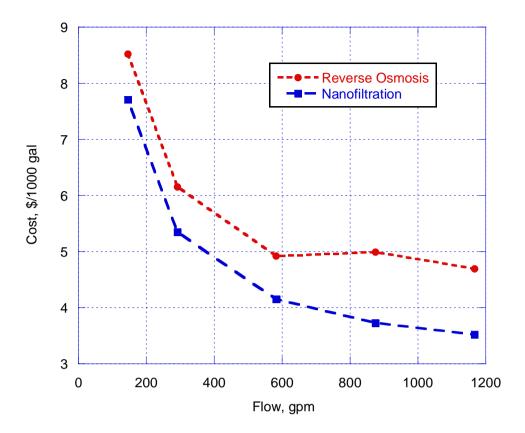


Figure 2. Comparison of cost and flow between nanofiltration and reverse osmosis

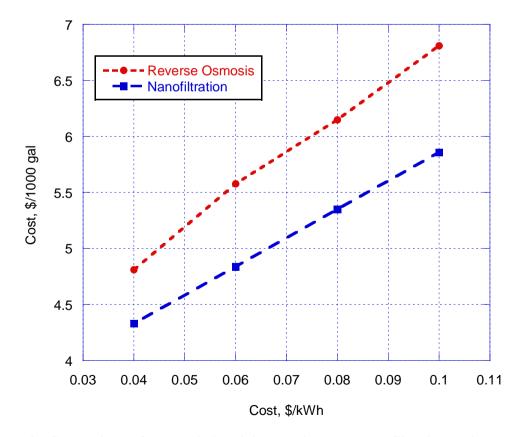


Figure 3. Comparison of cost and electricity cost between nanofiltration and reverse osmosis

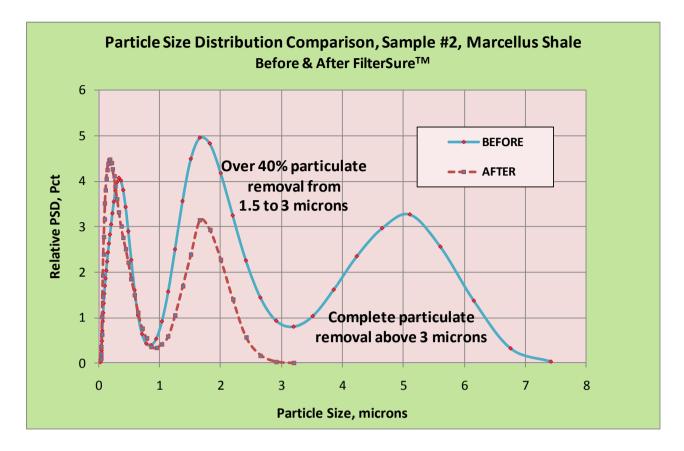


Figure 4. Particle size distribution following FilterSure<sup>TM</sup> PDU test on Marcellus water

#### **Upstream Technologies**

Major effort this quarter was devoted to testing both the FilterSure and the EC technology as key upstream process technologies. Raw water from one Marcellus sample was filtered by the FilterSure 2-GPM PDU. Figure 4 plots the Particle Size Distribution (PSD) as a percent of the total particles vs. the particle size in microns. Results show that particles greater than approximately 3 microns were completely removed from the water sample by the FilterSure technology. Additionally, particles larger than about 1 micron were reduced by an estimated 40%.

A second Marcellus sample provided by an industry partner was shipped to a provider of Electrical Coagulation (EC) technology for testing as a potential pre-treatment option. The EC treated water was returned to WVU for evaluation. The results (presented in Figure 5) show that the EC technology had a major impact on the distribution of the solids.

Specifically, the EC technology caused the solids to shift from a few microns in size to larger solids having a normal bell shaped distribution. Heavy metals and/or radioactive compounds are expected to be contained in the EC mobilized solids that will be removed by filtration and managed as a part of a commercial process.

Using the particle size distribution results, FilterSure selected filter media needed to remove the larger EC produced solids. The EC treated water was processed through the FilterSure PDU with the tests witnessed by DOE and other representatives. Effluent from the PDU was visually clear without particulates. Water chemistry of the effluent is currently underway and will be reported in the next quarterly report.

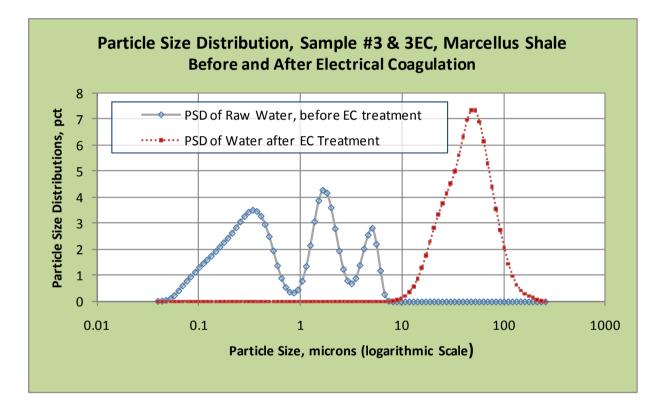


Figure 5. Particle size in raw and EC treated Marcellus water

#### Removal of Salt Using Solvents

Methods for measuring the effect solvent addition are being developed. Pure solubilities are being determined. The solubility determined for KCl (mean of 3 trials) was 34.5g/100g water. The literature value is 34.7 g/100g. The experimental value is less than 1% (0.58%) different than the literature value, indicating the method to be sound. Subsequent studies will determine solubilities in mixed solvent systems.

### **Issues & Challenges:**

The excellent cooperation of our Industry Contact Group in providing frac water samples has resulted in a high burn rate on year-one funding for FilterSure. As a result, available funds in the FilterSure subcontract will be depleted earlier than expected and some tests will have to be deferred until year-two funds are available. This affects the testing of higher-salinity (e.g., 80-100,000 mg/liter and up) water samples by the PDU, and especially affects further work on the EC complementary water treatment technologies in conjunction with the PDU.

### **Milestone Report:**

No milestones were scheduled to be accomplished in the second quarter. We expect to be able to accomplish all milestones on schedule.

### **Cost Status Report:**

Overall project costs are detailed in Appendix A. A new fiscal task was created in order to pay a graduate research assistant within the WVU Department of Civil and Environmental Engineering to complete the particle size distribution analysis and provide experimental support for the removal of salts using solvents.

FilterSure installed its 1,600 pound PDU in the WVU high bay research area. Setup costs and the costs of two complete filtration tests have largely exhausted FilterSure subcontract funds planned for year 1. FilterSure is suspending further testing until the balance of the Phase I funds is made available. These funds will support a continuous process demonstration that links the EC and the FilterSure technology. This test is needed to establish process economic feasibility.

### **Summary of Accomplishments:**

The project is currently ahead of schedule, but with corresponding costs higher than originally scheduled. Significant accomplishments this quarter are listed below:

- An Industry Contact Group was created to gain information on operating parameters and to obtain representative water flowback samples.
- Responses to a questionnaire developed for this project are providing engineering information on volumes of flow back water and water chemistry requirements for recycling of flow back water.
- Industry Contact Group members have provided three flowback frac water samples this quarter. The WVU Radiation Safety Department tested all samples for radioactivity and found all to be at or below background values. A faculty member and his graduate research assistant in the WVU Department of Civil and Environmental Engineering is determining particle size distribution measurements for each sample received and tested under this program. The WVU analytical lab is measuring water chemistry.
- Tests of one Marcellus water sample show that the FilterSure PDU will remove 100 % of the frac water suspended solids greater than three microns.
- EC shifted the distribution of the suspended particles creating larger size particles as compared with the raw water sample.
- Suspended solids in the EC treated water were easily removed with the FilterSure technology resulting in an effluent that was visually clear without particulates.

## Appendix A: Cost Status Report

## **Total Project Funds:**

Source	Federal \$ Received	Federal Expended	Remaining	Cost Share	Cost Share Expended	Remaining	Total Project Value	Total Expended	Total Remaining
SALARIES:	65,015.86	2,154.36	62,861.50	21,381.32	297.12	21,084.20	86,397.18	2,451.48	83,945.70
BENEFITS:	14,196.17	253.24	13,942.93	5,772.96	77.25	5,695.71	19,969.13	330.49	19,638.64
SUPPLIES:	5,504.00	1827.46	3,676.54				5,504.00	1,827.46	3,676.54
TRAVEL:	3,000.00	60.76	2,939.24				3,000.00	60.76	2,939.24
SUBK:	435,890.00	46,509.18	389,380.82	350,600.00		350,600.00	786,490.00	46,509.18	739,980.82
OTHER DIRECT COSTS:	15,000.00	201	14,799.00				15,000.00	201.00	14,799.00
TOTAL DIRECT COSTS:	538,606.03	51,006.00	487,600.03	377,754.28	374.37	377,379.91	916,360.31	51,380.37	864,979.94
INDIRECT COSTS: F&A @ 46.5	71,012.94	6,537.11	64,475.83	12,626.75		12,626.75	83,639.69	6,537.11	77,102.58
TOTAL:	609,618.97	57,543.11	552,075.86	390,381.03	374.37	390,006.66	1,000,000.00	57,917.48	942,082.52

## Year 1 Project Funds:

Source	Federal \$ Received	Federal Expended	Remaining	Cost Share	Cost Share Expended	Remaining	Total Project Value	Total Expended	Total Remaining
SALARIES:	25,619.72	2,154.36	23,465.36	5,581.20	297.12	5,284.08	31,200.92	2,451.48	28,749.44
BENEFITS:	5,051.28	253.24	4,798.04	1,506.92	77.25	1,429.67	6,558.20	330.49	6,227.71
SUPPLIES:	998.00	1827.46	-829.46				998.00	1,827.46	-829.46
TRAVEL:	500.00	60.76	439.24				500.00	60.76	439.24
SUBK:	118,215.00	46,509.18	71,705.82	29,600.00	362.5	29,237.50	147,815.00	46,871.68	100,943.32
FilterSure	71,815.00	45,059.18	26,755.82	18,000.00	0.00	18,000.00	89,815.00	45,059.18	44,755.82
ShipShaper	46,400.00	1,450.00	44,950.00	11,600.00	362.50	11,237.50	58,000.00	1,812.50	56,187.50
OTHER DIRECT COSTS:	38,700.00	201	38,499.00				38,700.00	201.00	38,499.00
TOTAL DIRECT COSTS:	189,084.00	51,006.00	138,078.00	36,688.12	374.37	36,313.75	225,772.12	51,380.37	174,391.75
INDIRECT COSTS: F&A @ 46.5	56,205.00	6,537.11	49,667.89			0.00	56,205.00	6,537.11	49,667.89
TOTAL:	245,289.00	57,543.11	187,745.89	36,688.12	374.37	36,313.75	281,977.12	57,917.48	224,059.64

## Appendix B: Milestone Status Report

No milestones were scheduled to be accomplished in the second quarter. Progress is being made on future milestones, and these will be accomplished on schedule. There is currently no slip in our timeline. Milestones are shown in the Gantt chart below, indicated with diamonds.

Task Name	Start	Finish													
	- Chair				2010				2011					2012	
				Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3		Q4	Q1	Q2
			S	O N D	JF	MAM	JJA	SONE	) J F M	MAM	JJ	A S	OND	JFN	IAMJ
Task 1.0 Project Management Plan	10/1/09	2/1/10	) 🧹												
Task 2.0 Phase I Process Development	2/1/10	8/1/11			┝╤═						-	7			
Subtask 2.1 Develop Conceptual Process Train	2/1/10	7/30/10	) -					/30							
Subtask 2.2 Develop Process Flow Model	2/1/10	8/31/10	D -		┼┝═										
Subtask 2.3 Identify Recycling Operational Requirements	2/1/10	9/30/10	D -					9/30							
Subtask 2.4 Develop and Test Treatment Methods	2/1/10	7/1/11	ηΨ		+						<u>₩</u> 7	7/1			
Treatment Method Development Final Recommendations	7/1/11	7/1/11	1								₩7	7/1			
Subtask 2.5 Preliminary Cost Estimate/Development of Decision Criteria	3/14/11	7/1/11	1							(	<u>וייי</u> קא	7/1			
Subtask 2.6 Decision to Proceed	7/4/11	8/1/11	1								Č	<u>ኪ</u>			
Task 3.0 Phase II Mobile Unit Design, Test, and Evaluate	8/2/11	5/31/12	2								1	<u> </u>	1	1	
Subtask 3.1 Finalize System Design and Costs	8/2/11	8/31/11	1									Č.			
Subtask 3.2 Fabricate Mobile Treatment Unit (MTU)	9/1/11	9/28/11	1										9/28		
Subtask 3.3 Install the MTU	9/29/11	10/31/11	1										10/	31	
Subtask 3.4 MTU Testing and Startup	11/1/11	11/14/11	1										🏹 1	1/14	
Subtask 3.5 MTU Operations, Monitoring, and Maintenance	11/15/11	3/5/12	2												3/5
Subtask 3.6 MTU Decommissioning	3/6/12	4/6/12	2												
Subtask 3.7 MTU Demonstration Report	4/9/12	5/31/12	2											1 7	

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