

DOE/MT/92009--T2

**EVALUATION OF THE FREEZE-THAW/EVAPORATION PROCESS
FOR THE TREATMENT OF PRODUCED WATERS**

QUARTERLY TECHNICAL PROGRESS REPORT

Contract No. DE-AC22-92MT92009

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Executive Summary

The use of freeze-crystallization is being increasingly acknowledged as a low-cost, energy-efficient method for purifying contaminated water. Freeze-crystallization has been shown to be effective in removing a wide variety of contaminants from water. Water purification by using natural conditions to promote freezing appears to be an extremely attractive process for the treatment of contaminated water in many areas where natural climatic conditions will seasonally promote freezing. The natural freezing process can be coupled with natural evaporative processes to treat oil and gas produced waters year round in regions where sub-freezing temperatures seasonally occur. The objectives of this research are related to development of a commercially-economic natural freeze-thaw/evaporation (FTE) process for the treatment and purification of water produced in conjunction with oil and gas.

During the reporting period of 4/1/95 to 6/30/95, project research concentrated on Subtasks 2.0 (Task 2 Project Reporting) and 2.2 (Re-evaluation of Process Economics Based on Laboratory-scale Process Simulation Results). The objectives of Task 2 are to conduct laboratory- and bench-scale simulations for optimizing the design of the FTE process. Task 2 requires completion of six subtasks: Subtask 2.0 - Task 2 Project Reporting (initiated 3/1/93), Subtask 2.1 - Laboratory-scale FTE Simulations, Subtask 2.2 - Re-evaluation of Process Economics Based on Laboratory-scale Process Simulation Results, Subtask 2.3 - Bench-scale FTE Simulations, Subtask 2.4 - Economic Assessment of Bench-scale Simulations, and Subtask 2.5 - Technical Report of Task 2. The completion of Subtask 2.2 was planned for this quarter.

Research efforts this quarter were:

- to complete Subtask 2.2,
- to find an industrial partner to provide cost-share funding to conduct a commercial demonstration of the process this year,
- to continue work to finalize the draft of the "Task 1 and Task 2 Report" (RETEC has requested that the Task 1 and Task 2 reports be combined for publication), and
- to present the project status to the US DOE Bartelsville Project Office Contractor's Review at the Fountainhead Resort in southeastern Oklahoma.

Subtask 2.2 is completed at the end of the reporting period. The revised process economics resulting from Subtasks 2.1 and 2.2 are more favorable than the initial economics resulting from Task 1 efforts. The results of the laboratory-scale FTE process simulations conducted in Subtask 2.1 were more favorable than the assumed process performance in Task 1. The key process performance parameters determined from Subtask 2.1 results that improved the process economics were:

- the contaminant concentration of the treated water produced was lower in the laboratory-scale simulations than the value assumed in Task 1 calculations,
- the contaminant concentration of the brine produced was generally higher in the laboratory-scale simulations than the value assumed in Task 1 calculations, and
- the evaporation efficiency, expressed as % of PAN evaporation, for the produced water holding pond was higher in the laboratory-scale simulations than the value assumed in Task 1 calculations.

Negotiations with parties interested in supporting a field demonstration of the process have resulted in a funding commitment from a major oil and gas producing company with significant operations and water disposal requirements in the Rocky Mountain Region. Contacts with these personnel are being directed by Mr. John Harju of the University of North Dakota (UND) Energy and Environmental Research Center (EERC).

The draft of the "Task 1 and Task 2 Report" delineating all project research completed thus far is continuing at the end of the reporting period.

Plans for the next quarter are to:

- complete the final editing of the "Task 1 and Task 2 Report,"
- complete the required annual reports, and
- get funding in place to conduct a commercial demonstration of the process.

1.0 Introduction

1.1 Background

The cost of treating the water produced in association with oil and natural gas has prevented the completion of wells in economically marginal formations and has caused low-productivity wells to be prematurely shut-in. An economical method for treatment, disposal, and/or reuse of these waters on a commercial-scale would assist the oil and natural gas industries in continuing to provide reasonably priced fuels to the consumer by allowing for economic production from marginal, unconventional, and depleted reserves. A treatment process that could produce water of suitable quality for reuse would also be advantageous for municipal, industrial, and agricultural development in the arid western United States where there is significant oil and natural gas production.

The natural processes of freezing and evaporation can be coupled to effectively and inexpensively treat waters produced in association with natural gas. This document delineates research conducted, during the time period from 4/1/95 to 6/30/95, for evaluating the technical and economic feasibility of this water treatment process. The research required for development of this process can be completed in two tasks:

- 1) Task 1: Literature Survey and Preliminary Economic Analysis
- 2) Task 2: Laboratory-Scale Process Evaluation and Field Demonstration of the Process

These two tasks as described above represent a change from previous plans which contained three tasks: 1) Literature Survey and Preliminary Economic Analysis, 2) Laboratory-and Bench-Scale Process Evaluation, and 3) Field Demonstration of the Process. The current contract (US DOE contract No. **DE-AC22-92MT92009**) is for completion of research to be conducted in the original Tasks 1 and 2; and if successful, funding for Task 3 will be solicited from other sources. Task 1 research has been completed. Laboratory-scale research in Task 2 is only near completion due to a delay in obtaining the required co-funding. However, results of Task 1 and the laboratory-scale research already completed in Task 2 strongly confirm the technical and economic viability of the process. Based upon these results, conversations with personnel of the Gas Research Institute (GRI), and conversations with independent oil and gas producers in the Rocky Mountain Region, a field demonstration of the FTE process is recommended in-place of the bench-scale process simulations previously planned. The field demonstration could be conducted in the same time frame and for the same budget as the original bench-scale simulations. Conducting the field demonstration at this time would greatly reduce the amount of time required for commercial-scale application of the process by: 1) demonstrating the technical and economic feasibility of the process and 2) demonstrating the environmental acceptability of the process. Demonstration of the technical and economic feasibility of the process is needed to obtain investment capital for commercialization and demonstration of the environmental acceptability of the process is needed to obtain the required permits for a commercial facility. If modification of the research plans are approved and the required co-funding obtained, the FTE process can be proven ready for commercialization within the next eighteen months. The recommended modification to the original research plan is consistent with the project objectives and is cost-effective.

1.2 Research for the Current Reporting Period

Research conducted during this time period was related to Task 2. The objectives of Task 2 are to conduct laboratory- and bench-scale simulations for optimizing the design of the FTE process. Task 2 requires completion of six subtasks: 2.0 - Task 2 Reporting, 2.1 - Laboratory-scale FTE Simulations, 2.2 - Re-evaluation of Process Economics Based on Laboratory-scale Process Simulation Results, 2.3 - Bench-scale FTE Simulation, 2.4 - Economic Assessment of Bench-scale Simulation, and 2.5 - Task 2 Technical Report. Subtasks 2.0, 2.1, and 2.2 were scheduled during the reporting period.

Subtask 2.0 - Task 2 Reporting - Required project reports were submitted.

Subtask 2.1 - Laboratory-scale FTE Simulations - All of the twenty-one planned laboratory-scale FTE process simulations have been completed. In these simulation series, the coal bed methane (CBM), O&G, and natural gas (NG) produced waters were each tested using atmospheric conditions similar to northeastern Colorado (Simulation Series #2), northwestern New Mexico (Simulation Series #3), and central Wyoming (Simulation Series #4). The objective of the fifth series of simulations was to duplicate the most favorable climatic conditions of the previous series of simulations and then conduct more detailed chemical analyses of the samples from the series. The central Wyoming climate yielded the most favorable results in the previous simulations and was simulated in Series #5. Samples from the fifth simulation series were subjected to detailed inorganic, organic, and radionuclide analyses. The enhanced evaporation pond design with bubblers and the water column freezing pad design were used in all three simulators in these series. The duration of each simulation in each of the series was nominally twenty four days.

Subtask 2.2 - Re-evaluation of Process Economics Based on Laboratory-scale Process Simulation Results - This subtask has been completed at the end of the reporting period.

Subtask 2.3 - Field Demonstration of the FTE Process - This subtask is delayed until the required cost-share funding can be obtained.

Subtask 2.4 - Re-evaluation of Process Economics based upon Field Demonstration Results - This subtask is delayed until the required cost-share funding can be obtained.

Subtask 2.5 - Final Project Report - This subtask is delayed until the required cost-share funding can be obtained.

No other subtasks were scheduled for this reporting period.

2.0 Project Description

2.1 Project Research Tasks and Subtasks

Following is a brief description of the project tasks and subtasks. The research required to complete each task/subtask is also summarized:

2.1.1 Task 1: Literature Survey and Preliminary Economic Analyses

A literature survey and preliminary economic feasibility and sensitivity analyses will be conducted to evaluate the technical feasibility and commercial viability of the FTE process. Specific subtasks to be performed are:

Subtask 1.1 - Literature Survey of FTE Research: 1) identify economically important FTE process parameters, 2) summarize the response of organics, metals and salts in contaminated waters to the FTE process, and 3) estimate potential interactions between constituents that may impact the process. Subtask 1.1 objectives have been achieved with one exception: a literature survey to provide data depicting the behavior of organics and heavy metals in a natural freezing water purification process. Natural freezing process data found in the literature was related to salts only. However, data in the literature related to artificial freezing processes confirm organic and heavy metals compounds can be successfully and efficiently removed from contaminated water by freezing processes.

Subtask 1.2 - Characterization of NG Production Waters and Conventional Treatment Costs: 1) review of literature and data bases to characterize typical waters that are generated in association with production from natural gas reservoirs, oil and gas reservoirs, and methane drainage from coal seams, 2) survey meteorological data to establish an expected range of atmospheric conditions at selected production sites where the FTE process is applicable (survey will include daily wind velocity and temperature cycles), and 3) survey local producers to determine their current treatment/disposal methods, costs, and willingness to participate in a field demonstration of the process. All objectives of Subtask 1.2 have been achieved.

Subtask 1.3 - Evaluation of Process and Environmental Constraints: 1) estimate FTE discharges and evaluate regulatory requirements for field and commercial-scale demonstration, 2) assess process discharges, regulatory

requirements, and costs of conventional methods of disposal/treatment of production waters, and 3) compare the environmental acceptability, regulatory requirements and costs of the FTE process to conventional methods. All objectives of Subtask 1.3 have been achieved.

Subtask 1.4 - Conceptual Process Design: 1) design a preliminary FTE process based on the results of work elements 1.1 through 1.3 to address environmental, regulatory and process issues for various types of produce waters. All objectives of Subtask 1.4 have been achieved.

Subtask 1.5 - Preliminary Economic Feasibility and Sensitivity Analyses: 1) develop a numerical discounted cash flow /rate of return economic model for the preliminary FTE process design resulting from Subtask 1.4; 2) evaluate the economics of a probable, base case operating scenario which assumes reasonable fixed values for: a) facility size and location, b) concentrations of salts, organics and heavy metals in the production water, c) atmospheric conditions, d) capital equipment costs, e) annual operating expenses, f) debt to equity ratio, g) bond interest, and h) return on investment after taxes; and 3) determine the economic sensitivity of the FTE process by evaluating the projected water treatment costs for a minimum of 33 differing operating scenarios. All objectives of Subtask 1.5 have been achieved.

Subtask 1.6 - Task 1 Summary Report: 1) provide a comprehensive analysis of the results of Tasks 1.1 through 1.5 and 2) determine if the FTE process is technically feasible, economically viable and economically stable. All objectives of Subtask 1.6 have been achieved.

2.1.2 Task 2: Laboratory- and Bench-Scale Process Simulation

Task 2 is the laboratory and bench-scale evaluation of the FTE process. The following subtasks are required for completion of Task 2:

Subtask 2.1 - Laboratory-scale Process Simulations: 1) design and construct a laboratory-scale simulator to test the FTE process; 2) conduct an initial series of nine process simulations to optimize the FTE process design by evaluating the effectiveness of the three different freezing design options: wetted column freezing, conventional water sprays, and atomizing sprays and three different evaporation techniques: conventional evaporation ponds, solar evaporation ponds, and solar distillation ponds; 3) conduct an additional series of eight process simulations, using the optimum process design for treating three different produced waters under three differing sets of atmospheric conditions, to determine the effectiveness of the FTE process in removing organic, metal, and salt constituents from mixtures; 4) conduct a duplicate simulation for each of the produced waters tested to verify experimental results. This subtask has been completed and laboratory-scale simulation results confirm the feasibility and commercial potential of the process.

Subtask 2.2 - Re-evaluation of Process Economics Based Upon Laboratory-scale Simulation Results: 1) re-evaluate FTE process economics using the numerical model developed in Subtask 1.5 based upon Subtask 2.1 simulations results.

Subtask 2.3 - Bench-Scale FTE Simulations: 1) design and construct three bench-scale simulations to verify the process effectiveness under actual climatic conditions, 2) conduct the simulations for one year, 3) confirm laboratory-scale simulation results under atmospheric conditions in Laramie, WY, 4) demonstrate the effectiveness of the process, and 5) acquire data for process scale-up.

Subtask 2.4 - Re-evaluation of Process Economics Based Upon Bench-scale Simulation Results: 1) re-evaluate FTE process economics using the numerical model and the Subtask 2.3 simulation results, and 2) refine the process design, equipment selection, construction procedures, and plant operating procedures for field demonstration using an FTE process.

Subtask 2.5 - Final Technical Report of the Simulation Results, Revised Process Economics, and Final Demonstration Plant Design and Economic Requirements: 1) write a technical report summarizing the results of the FTE process simulations, providing a commercial-scale process economic projection and the finalized technical and economic requirements of an FTE process demonstration plant for the treatment of natural gas production waters. This report will also provide detailed requirements for completion of Task 3.

2.1.3 Task 3: Field Demonstration of the FTE Process

Task 3 will be a field demonstration of the FTE process conducted at an operating production site. Task 3 will be initiated if results of Task 2 show FTE to be a technically and economically viable process. The field demonstration will confirm the process' commercial viability. It will incorporate all technical innovations and process improvements resulting from previous research efforts. The details relating to the work required to complete Task 3 will be determined in the research conducted in Tasks 1 and 2 of the current contract.

2.1.4 Proposed Modification of Contract Tasks and Subtasks

Limited laboratory data already acquired and results of previous research, conducted by others, strongly confirm the technical feasibility of the process. In addition, the results of Task 1 of this research strongly suggest economic viability of the process. For these reasons, a field demonstration of the process is recommended to be conducted in-place of the bench-scale process simulations originally proposed. The field demonstration of the process would better meet the project objective of developing and demonstrating a cost-effective economically viable commercial technology utilizing the FTE process to treat water produced in conjunction with oil and natural gas. The field demonstration would meet the two key process development needs required for commercial application of the process: 1) demonstration of the technical and economic viability of the process, and 2) obtaining regulatory acceptance of this novel process. The recommended field demonstration can be conducted in a fashion such that the total contract budget and duration do not change. However, completion of the project as modified will depend upon the ability to obtain the required cost share for project Subtasks 2.3, 2.4, and 2.5. RTC and the UND EERC have been unsuccessful in obtaining the required cost share for these subtasks as originally proposed or as modified in this plan.

2.2 Project Objectives

The general objective of the research is to develop and demonstrate a cost-effective economically viable commercial technology that utilizes the natural FTE process to treat water produced in conjunction with oil and natural gas. The specific objectives of the research are to:

- develop an economic model for determining the commercial viability, economically significant parameters, and research issues of the FTE process,
- conduct laboratory- and bench-scale process simulations to optimize the design of the FTE process, and
- to conduct on-location treatment of water from a producing well to demonstrate the technical and economic viability of the FTE process.

3.0 Project Status

3.1 Work Performed during the Reporting Period

3.1.1 Subtask 2.0 Task 2 Project Reporting

During the reporting period, monthly project reports required for the months of April, May, and June 1995 were completed and submitted to the US DOE Document Control Center at PETC and to RETEC. The Quarterly project report for the time period of 1/1/95 - 3/31/95 was also completed and submitted to the US DOE Document Control Center at PETC and to RETEC. John Boysen presented the project status June 28, 1995 to the US DOE Bartelsville Project Office Contractor's Review at the Fountainhead Resort in southeastern Oklahoma. No budget or schedule problems exist for this subtask.

3.1.2 Subtask 2.2 - Re-evaluation of Process Economics Based on Laboratory-scale Process Simulation Results

Subtask 2.2 is completed at the end of the reporting period. The revised process economics resulting from Subtasks 2.1 and 2.2 are more favorable than the initial economics resulting from Task 1 efforts. The results of the laboratory-scale FTE process simulations conducted in Subtask 2.1 were more favorable than

the assumed process performance in Task 1. The key process performance parameters determined from Subtask 2.1 results that improved the process economics were:

- the contaminant concentration of the treated water produced was lower in the laboratory-scale simulations than the value assumed in Task 1 calculations,
- the contaminant concentration of the brine produced was generally higher in the laboratory-scale simulations than the value assumed in Task 1 calculations, and
- the evaporation efficiency, expressed as % of PAN evaporation, for the produced water holding pond was higher in the laboratory-scale simulations than the value assumed in Task 1 calculations.

Negotiations with parties interested in supporting a field demonstration of the process have continued. During the quarter, discussions were held with personnel of a major oil and gas producing company with significant operations and water disposal requirements in the Rocky Mountain Region. These discussions resulted in a commitment for a portion of the required cost-share funding. Contacts with these personnel are being directed by Mr. John Harju of the University of North Dakota (UND) Energy and Environmental Research Center (EERC).

The draft of the "Task 1 and Task 2 Report" delineating all project research completed thus far is continuing at the end of the reporting period.

No other research was in progress during this reporting period. Subtasks 2.3, 2.4, and 2.5 were scheduled to be in progress during this reporting; however, initiation of these subtasks are delayed until the required cost-share funding is obtained.

3.2 Summary of Achievements

Project achievements for the time period of 4/1/95 to 6/30/95 are:

- Subtask 2.2 is completed at the end of the reporting period. The revised process economics resulting from Subtasks 2.1 and 2.2 are more favorable than the initial economics resulting from Task 1 efforts. The results of the laboratory-scale FTE process simulations conducted in Subtask 2.1 were more favorable than the assumed process performance in Task 1. The key process performance parameters determined from Subtask 2.1 results that improved the process economics were: the contaminant concentration of the treated water produced was lower in the laboratory-scale simulations than the value assumed in Task 1 calculations, the contaminant concentration of the brine produced was generally higher in the laboratory-scale simulations than the value assumed in Task 1 calculations, and the evaporation efficiency, expressed as % of PAN evaporation, for the produced water holding pond was higher in the laboratory-scale simulations than the value assumed in Task 1 calculations.
- Negotiations with parties interested in supporting a field demonstration of the process have continued. During the quarter, discussions were held with personnel of a major oil and gas producing company with significant operations and water disposal requirements in the Rocky Mountain Region. These discussions resulted in a commitment for a portion of the required cost-share funding. Contacts with these personnel are being directed by Mr. John Harju of the University of North Dakota (UND) Energy and Environmental Research Center (EERC).
- The draft of the "Task 1 and Task 2 Report" delineating all project research completed thus far is continuing at the end of the reporting period. (RETEC has requested that the Task 1 and Task 2 reports be combined for publication).
- A project status presentation was given by John Boysen June 28, 1995 to the US DOE Bartelsville Project Office Contractor's Review at the Fountainhead Resort in southeastern

Oklahoma.

4.0 Planned Activities for the Next Quarter

During the upcoming quarter (July 1 - September 30, 1995), plans are to:

- complete the final editing of the "Task 1 and Task 2 Report,"
- complete the required annual reports, and
- get funding in place to conduct a commercial demonstration of the process.

5.0 Summary

Task 1, and Subtasks 2.1 and 2.2 have been completed. A literature survey, environmental regulatory assessment, survey of current disposal practices and economics, and numerical process and economic modeling have been completed (Task 1). Twenty one laboratory-scale process simulations have been also been completed (Subtask 2.1). Previous research and laboratory simulation results both confirm the process' potential to produce a useable quality treated water by significant and simultaneous removal of salts, organics, and heavy metals (including NORM). Results of twenty-one simulations completed all show significant concentration reductions of these species in the treated water. In the simulations, a natural gas produced water, an oil and gas produced water, and a coal bed methane produced water were treated with varied climatic conditions. Treated waters generated from simulations in which only 182 hours of sub-freezing conditions existed had TDS concentrations ranging from 200 to 430 ppm. The feed water TDS concentrations in these simulations ranged from 2640 to 10900 and the estimated TDS concentration in the evaporation pond prior to freezing ranged from approximately 4500 to 18,800 ppm. Detailed analyses of all of the treated waters produced in Simulation Series #5 indicate virtually all detectable inorganic, organic, and radionuclide components were significantly reduced compared to either the produced water feed or the water in the evaporation pond when the freezing treatment was initiated. The masses of brine produced in these simulations ranged from 5 to 28% of the feed input indicating a 72 to 95% reduction in disposal volume is achievable using the FTE process. The simulations were designed to simulate climatic conditions of various regions but results analysis indicates the age of the ice pile (hours of freezing) was the key parameter affecting results. Since the simulations ran a year of climatic conditions in twenty four days, we expect a field demonstration to yield more favorable results. Even in a relatively mild climate such as the Farmington, New Mexico region, 1100 hours with sub-freezing temperatures typically occur annually. The maximum hours with sub-freezing temperatures in the simulations was 182 hours.

Economic results indicate the FTE process could reduce water disposal costs by 5 to 70% compared to conventional evaporation alone. The reduction depends upon the climate and feed water quality. Water disposal costs for an FTE facility in the San Juan that is fed with more than 500 bbl/day of 12,000 ppm TDS water range from \$0.05 to \$0.50/bbl. Treatment costs are dependent upon the facility size, state and federal regulatory requirements for pit construction, facility operation, and water discharge/use.

Results of all research completed continue to indicate the process has significant commercial economic potential and is an environmentally acceptable option to produced water disposal by deep well injection. Contacts have been and will continue to be made with oil and gas producers in the area. The objectives of these contacts are to obtain cost-share funding to continue research and to demonstrate the process. The acquisition of co-funding for the remainder of the contract research is an open item causing concern at RTC, although encouraging developments have occurred recently. Currently, the project is behind schedule but with no budget problems.

6.0 Report Distribution

The quarterly progress report distribution specified by the current contract is three copies of quarterly reports to:

Document Control Center
United States Department of Energy
Pittsburgh Energy Technology Center
P.O. Box 10940, MS 921-118
Pittsburgh, PA 15236 - 0940

7.0 References

None

8.0 Publications

None