Dynamic Binary Complexes (DBCs) as Super-Adjustable Viscosity Modifiers for Hydraulic Fracturing Fluids

DE-FE0031778

Texas A&M University
Department of Chemical Engineering
Texas A&M Energy Institute

U.S. Department of Energy
National Energy Technology Laboratory
Oil & Natural Gas
2020 Integrated Review Webinar
Program Overview

- **Project Funding**

<table>
<thead>
<tr>
<th></th>
<th>Federal</th>
<th>Cost Share</th>
<th>Total Costs</th>
<th>Cost Share %</th>
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<tbody>
<tr>
<td><strong>Budget Period 1</strong></td>
<td>$492,699</td>
<td>$124,683</td>
<td>$617,382</td>
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<td><strong>Budget Period 2</strong></td>
<td>$496,841</td>
<td>$127,098</td>
<td>$623,939</td>
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<td><strong>Budget Period 3</strong></td>
<td>$510,169</td>
<td>$123,209</td>
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<td><strong>Total</strong></td>
<td>$1,499,709</td>
<td>$374,990</td>
<td>$1,874,699</td>
<td>20.00%</td>
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- **Project Performance Dates**

<table>
<thead>
<tr>
<th>Task Name</th>
<th>Year 1</th>
<th>Year 2</th>
<th>Year 3</th>
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<tbody>
<tr>
<td>Task 1: Investigation of Flow and Rheological Characteristics of DBCs</td>
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<tr>
<td>Task 2: Determination of Proppant Dispersion Stability under Various Conditions</td>
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<tr>
<td>Task 3: Obtaining an Understanding of Extent of Reversibility and Reusability</td>
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<tr>
<td>Task 4: Investigation of Compatibility with Other Chemicals in Fracking Fluids</td>
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<tr>
<td>Task 5: Development of Models to Describe Proppant Transport and Fracture Propagation</td>
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<tr>
<td>Task 6: Construction of Models for Adsorption and Desorption of DBCs</td>
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<tr>
<td>Task 7: Development of Models for Estimating Wastewater Recovery and Gas Production Rates</td>
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<tr>
<td>Task 8: Selection and Optimization of DBC Formulations for Laboratory-Scale Tests</td>
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<tr>
<td>Task 9: Carrying out Laboratory Experiments to Evaluate Hydraulic Fracturing Performance</td>
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<tr>
<td>Task 10: Scale-up, Manufacturing, and Field Testing of DBCs</td>
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<tr>
<td>Task 11: Preparation of Cost-Benefit Analysis and Evaluation of Economic Impact</td>
<td></td>
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</table>
Program Overview

• Project Participants
  Texas A&M University (Research and Development)
  • Department of Chemical Engineering
  • Texas AM& Energy Institute
  Incendium Technologies (Commercialization)

• Project Personnel
  Mustafa Akbulut, Associate Professor, Texas A&M University
  Joseph Kwon, Assistant Professor, Texas A&M University
  Shuhao Liu, Graduate Student
  Silabrata Pahari, Graduate Student
  Yu-Ting Lin, Graduate Student
  Bhargavi Bhat, Graduate Student
  Spencer Doyle, Undergraduate Student
  Landry Ray, Undergraduate Student
  Ankit Anand, Undergraduate Student
  Sek Kai Leong, Project Technician
  Cengiz Yegin, Product Development Engineer, Incendium Technologies
Project Objectives

- To develop novel dynamic binary complexes to achieve super-adjustable, reversible viscosities and the implementation and wide-spread utilization of these novel viscosifiers in hydraulic fracturing fluids.
- To mature the Technology Readiness Level (TRL) of this concept from TRL 2-3 to TRL 5-6.
- To investigate and optimize rheological properties of aqueous solutions containing DBCs with respect to shear rate, concentration, temperature, salinity, and pressure.
- To evaluate and optimize the compatibility of DBCs with other chemicals used in fracking fluids such as clay stabilizers, corrosion inhibitors, scale inhibitors, friction reducers.
- To develop computational models and frameworks for investigating the effect of DBC on proppant transport, fracture propagation, bank formation, and fluid leak-off during hydraulic fracturing.
- To develop a 3D, three-phase black oil model for estimating the production rates of formation water, recovered DBC, and gas from the fractured wells.
- To assess the efficiency of proppant transport into fissures and fractures and permeability enhancements using the selected optimum DBC formulations and to compare the performance of developed DBCs with that of currently available fracking fluids.
- To outline comprehensive manufacturing design and strategy for the large-scale synthesis of the most optimum DBC formulation.
- To carry out a comprehensive cost-benefit analysis considering the cost of raw materials, labor, capital investment of manufacturing equipment, operational costs, and percent improvements in shale gas recovery.
## Technology Background

<table>
<thead>
<tr>
<th>Fluid Type</th>
<th>Viscosity (cP)</th>
<th>Advantages</th>
<th>Disadvantages/Limitations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water frac</td>
<td>2–5</td>
<td>• Inexpensive</td>
<td>• Requires high pump rates</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Insensitive to salinity</td>
<td>• Poor proppant transport</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• Narrow fracture width</td>
</tr>
<tr>
<td>Linear aqueous gels</td>
<td>10-30</td>
<td>• Environmentally friendly</td>
<td>• Not re usable</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Support transport of medium-sized proppants</td>
<td>• Somehow narrow fracture width</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• Some residue leftover in fractures</td>
</tr>
<tr>
<td>Cross-linked aqueous gels</td>
<td>100-1000</td>
<td>• Wide fracture width</td>
<td>• Not re usable</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Reduced fluid loss</td>
<td>• Corrosive/toxic breakers</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Enhanced proppant transport</td>
<td>• Fracture damage by residues</td>
</tr>
<tr>
<td>Aqueous viscoelastic surfactant (VES)-based fluids</td>
<td>100-1000 cP</td>
<td>• Wide fracture width</td>
<td>• High-cost</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Enhanced proppant transport</td>
<td>• Poor temperature/salt tolerance</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• No residue leftover in fractures</td>
<td>• High volume of fluid leak-off</td>
</tr>
<tr>
<td>Foam fluids</td>
<td>10-100</td>
<td>• Very low fluid loss</td>
<td>• High cost of gas</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Mediocre proppant transport</td>
<td>• Gas availability</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Reduced environmental impact</td>
<td>• Depressurization damage in fractures</td>
</tr>
<tr>
<td>Gelled oil-based fluids</td>
<td>50-1000</td>
<td>• Compatible with all formations</td>
<td>• Gelling and clogging problems</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Lower formation damage</td>
<td>• Higher cost</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• More toxic than water-based systems</td>
</tr>
</tbody>
</table>
Technology Background

**pH-Responsive**

![Graph showing pH-Responsive behavior with viscosity data](image)

- **Supramolecular Formulation (2 wt%)**
- Viscosity: $\sim 25$, $\sim 330$

**Thermo-Responsive**

![Graph showing Thermo-Responsive behavior with viscosity data](image)

- **Supramolecular Formulation (2 wt%)**
- Viscosity: $\sim 10^{-2}$, $\sim 10^6$
### Technology Background

#### Shale Gas Recovery

<table>
<thead>
<tr>
<th>Region</th>
<th>Shale Depth (ft)</th>
<th>Temperature (°F)</th>
<th>Salinity (mg/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anadarko</td>
<td>4,000 – 11,000</td>
<td>140° to 280° F</td>
<td>&lt;50</td>
</tr>
<tr>
<td>Appalachia</td>
<td>5,000 – 9,000</td>
<td>160° to 240° F</td>
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<td>Niobrara</td>
<td>7,000 – 8,000</td>
<td>200° to 220° F</td>
<td>50-100</td>
</tr>
<tr>
<td>Permian</td>
<td>6,700 – 11,300</td>
<td>190° to 280° F</td>
<td>100-150</td>
</tr>
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</table>

**Target Conditions:**
- **Temperature:** 140°F to 280°F
- **Salinity:** 50 mg/mL to 200 mg/mL
- **Depth:** 4000 ft to 10,000 ft
Technology Background

(a) pH-Responsive DBC

(b) Thermo-Responsive DBC

Multilayer, Flexible Nanowires

Layers

Reversible

Twisted Nanoribbon
Technology Background

Current Viscosity Modifiers

**Cross-Linked Aqueous Gel**
- Guar Gum
- Cross-Linked Polymer Chains

**Viscoelastic-Surfactant (VES) Gel**
- Above Critical Concentration
- Wormlike Micelle
- Entangled Wormlike Micelles

Developed DBCs

Dynamic Binary Complex (DBC) Gel
- A
- B
- A-B Complex
- Reversible Complexation
- Above Critical Concentration
- Acidic pH
- Basic pH

Entangled Multilayer Nanowires
- Multilayer Nanowires
- Nanoscale Rolling
- Lamellar Assembly
Technology Background

- **Advantages of DBC Technology**
  - *Viscosity Adjustability*: Viscosity can reversibly be strongly controlled by adding acid or base.
  - *Reusability*: DBC does not rely on permanent breakers and can be reused multiple times.
  - *Environmentally Benign*: Building blocks are opted from nontoxic and biological-origin materials.
  - *Superior Proppant Carrying Ability*: DBC can actively and passively interact with proppants.
  - *High Durability*: DBCs can be used at elevated temperatures and salinity for prolonged periods.
  - *Possibility of Eliminating Permeation Damage*: DBCs can be assembled and disassembled dynamically.

- **Challenges of DBC Technology**
  - *Current Economic Difficulties of Fracturing Industry*
  - *Market Adaptation*
  - *Material Cost*
Project Scope

- **Flow and Rheological Characteristics of Dynamic Binary Complexes (DBCs)**
  - Viscosity of a fracking fluid is critical to ensure carrying the proppant from the wellbore to the fracture tip, forcing proppant entrance into the fracture, and generating a desired net pressure to control proppant bank height growth.
  - Investigate viscosity as a function of DBC nanoarchitecture, DBC concentration, temperature, and salinity

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- **Proppant Dispersion Stability**
  - Study the settling time of sand (proppant) in DBCs with respect to DBC nanoarchitecture and concentration, temperature, and salinity and maximize it
Project Scope

- **Extent of Reversibility and Reusability**
  - Evaluate the reversibility of DBCs against repetitive temperature and pH cycles

- **Compatibility with Other Compounds in Fracking Fluids**
  - Fracturing Fluid Components: Biocide, Breaker, Clay Stabilizer, Corrosion Inhibitor, Friction Reducer, Iron Control, Emulsion Preventer, Scale Inhibitor
  - Investigate the compatibility of DBCs with other components of fracking fluids and identify the optimum compound with the highest compatibility for each function

- **Computational Models to Describe Proppant Transport and Fracture Propagation**
Project Scope

- **Computational Studies of Estimating Wastewater Recovery and Gas Production Rates after Fracking Fluid clean-up**
  - Develop a computational framework and sub-routines to enable the implementation of DBCs in the 3D, three-phase black oil model
  - Estimate gas production, water production, and DBC recovery rates
- **Selection and Optimization of DBC Formulations for Laboratory-Scale Fracturing Tests**
- **Laboratory Experiments to Evaluate Hydraulic Fracturing Performance**

- **Scale-up of DBC Production and Construction of Pilot Plant**
- **Field Tests**
- **Cost-Benefit Analysis**
Milestones and Success Criteria

- Achieve a viscosity of 50–1000 cP with 0.1–2% of DBC solutions at shear rates of 40–100 s⁻¹ [Year 1]
- Obtain 50% improvements in proppant carrying capacity compared to three commercial fracturing fluids for a given concentration of DBC [Year 1]
- Accomplish a reversible re-adjustability of 20-fold in viscosity of DBCs at typical reservoir pressures, temperatures, and salinities via pH stimulus [Year 1]
- Achieve a maximum of 20% reduction in viscosity and proppant carrying capacity after 5 stimulus cycles [Year 2]
- Successful development of dynamic models for 3D, simultaneously growing multiple fractures with at least 2D proppant transport [Year 2]
- Determine all kinetic parameters of DBC formulation with the best laboratory-scale performance, which are the main scale-up parameters [Year 2]
- Obtain 50% enhancements in fracture permeability and conductivity using DBCs compared to four commercial fracturing fluids [Year 2]
- Realize 50 pounds/day production rate at a minimum yield of 85% in a pilot plant specifically designed manufacturing of DBC [Year 3]
- Achieve at least 50% improvements in the hydrocarbon recovery on the field tests compared to the current-state-of-art [Year 3]
- Prepare a comprehensive cost-benefit analysis considering raw material cost, production cost, deployment costs, durability, life-time, and potential benefits [Year 3]
49 new formulations have been developed!
6 formulations with exceptional flow properties and proppant carrying ability have been identified.
Progress and Current Status of Project

pH-Adjustability of DBC Viscosifiers

- pH Adjustability for A7/B10: 228-fold
- pH Adjustability for A5/B5: 1624-fold
- pH Adjustability for A8/B1: 284-fold
- pH Adjustability for CF1: ~1-3 fold
- pH Adjustability for CF2: ~1-3 fold
Progress and Current Status of Project

Effect of Salt on Viscosity of DBCs

A7/B10

A5/B5
Progress and Current Status of Project

Salt-Induced Viscosification and Gelling of DBC A8/B1!

DBC A8/B1

0 ppm salt

N,N,N-trimethyl-4-(octadecylamino-4-oxobutan-1-aminium

Maleic acid

B1
Progress and Current Status of Project

Effect of Temperature on Viscosity of DBCs

A7/B10

A5/B5
Progress and Current Status of Project

Characteristics of “a Dream” Fracking Fluid:
- large proppant carrying capacity at elevated temperatures
- large proppant carrying capacity at high salinity
- high adjustability to precisely control proppant transport and deposition

Formulation | Proppant Settling Time
--- | ---
CF1 @25 °C | 8 min
CF2 @25 °C | < 30 sec
CF3 @25 °C | ~ 1 min
DBC A8/B1 @25 °C | ~ 3 days
DBC A5/B5 @25 °C | ~ 2 days
DBC A7/B10 @25 °C | ~ 1 day
CF1 @90 °C | 3 min
CF2 @90 °C | < 30 sec
CF3 @90 °C | < 30 sec
DBC A8/B1 @90 °C | ~ 8 hr
DBC A5/B5 @90 °C | 90 sec
DBC A7/B10 @90 °C | ~ 15 min
• A thermodynamic model has been developed by considering all the components of the free energy in a cylindrical DBCs.
• The model is able to predict packing fraction of the micelles in response to varying pH.
• Equilibrium dimensions are used to predict the length of DBCs. This length is used as an input for the rheology model discussed in the next slide.

The altering packing fraction in DBC in response to different pH.

The change of free energy with varying Rc.

The change of free energy with varying Rs.
Novel coarse-grained Brownian dynamics (BD)/kinetic Monte Carlo (kMC) model developed for predicting rheology of DBCs

- In this model, DBC chains are represented by springs and beads while the entanglements are represented by slip-springs.
- Simulation of stress relaxation is modeled through mechanisms like reptation, contour length fluctuations, constraint release, and dynamic union and scission, which are executed by the kMC method using the standard metropolis algorithm.
- The kMC algorithm simulates the reptation mechanism by the process of slip spring hopping.

Progress and Current Status of Project

The Coarse grained BD/kMC rheology model simulations are compatible with the experimental data, which demonstrates that reptation, constraint release, contour length fluctuations, and dynamic union and scission are the major relaxation mechanisms in the WLMS.
A three-dimensional, multiphase production simulator is developed to predict the production from a reservoir, hydraulic fractured with VES fluids, by considering the impact of formation damage, fracture geometry, and fluid flowback.

The production simulator is used to carry out the sensitivity analysis for determining the optimal viscosity of fracturing fluids, for a specific reservoir.

A closed-loop optimization problem is formulated and solved to obtain the optimum pumping schedule necessary for obtaining the final fracture geometry with uniform proppant concentration inside the hydraulic fractures.

Impact of using VES fluids
Determine optimal viscosity
Obtaining a pumping schedule

The 2D view of the production simulator
Sensitivity analysis to determine optimal viscosity prediction
Optimal pumping schedule to attain target fracture geometry
Plans for Future Testing/Development/Commercialization

Future Plans/Development

- Compatibility studies with other components of fracturing fluids
- Investigation of permeation improvements
- Laboratory scale fracturing tests
- Large-scale fracturing tests
- Development of models for estimating fracturing performance of DBCs
- Aging studies for storage over prolonged periods of time
- Scale-up studies
Plan for Future Testing/Development/Commercialization

- **Scale-up and Commercialization**
  - As a first step in scale-up, **Incendium Technologies** are obtaining kinetical parameters for reactions of the most promising three formulations.
  - Pilot reactor is being designed and will be built based on the kinetical parameters obtained.
  - The sourcing of raw materials for pilot scale production will be established.
  - The energy consumption and operational costs will be monitored to be able to estimate the cost of novel viscosifiers.
  - After confirming enhanced fracturing efficiency and pilot scale production, Incendium Technologies and **Eastman Chemicals** will establish agreements for large scale production.
Summary

- Novel gelling agents with super-adjustable viscosity have been developed.
- Nanoarchitecture of building blocks of DBCs can be tuned to alter the target pH for stimuli-responsiveness.
- DBCs have demonstrated high-tolerance against temperature.
- Salinity has a weak influence on the viscosity of DBCs.
- One particular formulation has been discovered to have increased viscosity with increasing salinity.
- DBCs have demonstrated exceptional ability to suspend proppants.
- Synergistic influence of viscosity and intermolecular interactions (DBC mesh adhering to proppant particles) are responsible for enhanced proppant stability.
Appendix

Organizational Chart

Texas A&M University

Akbulut Group
- Materials Synthesis
- Formulation Development
- Fluid Characterization
- Proppant Stability Analysis

Kwon Group
- Proppant Transport Model
- Fracture Propagation Model
- Bank Formation Model
- Gas Production Model

Incendium Technologies
- Kinetic parameters
- Scale-up
- Process Development
- Cost-Benefit Analysis
- Commercialization
## Gantt Chart

<table>
<thead>
<tr>
<th>Task Description</th>
<th>Milestone</th>
<th>Milestone Description</th>
<th>Milestone Verification Process</th>
<th>Expected Quarter</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow and Viscosity Properties of DBCs</td>
<td>A1</td>
<td>Achieve a viscosity of 50–1000 cP with 0.1–2% of DBC solutions at shear rates of 40–100 s⁻¹</td>
<td>Rotational and oscillatory rheometry measurements with three repeats for each sample</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>A2</td>
<td>Accomplish a reversible re-adjustability of 100-fold in viscosity of DBCs at typical reservoir pressures, temperatures, and salinities via pH stimulation</td>
<td>High-pressure rheometry studies with three replicate measurements for each sample</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>Proppant Dispersion Stability</td>
<td>B1</td>
<td>Obtain 100% improvements in proppant carrying capacity compared to four commercial fracturing fluids for a given concentration of DBC</td>
<td>Static and dynamics sand settling experiments</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B2</td>
<td>Realize 3 orders of magnitude reversible control over the proppant settling velocity in situ</td>
<td>Static and dynamics sand settling experiments</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td><strong>Go/No-Go Decision Point 1</strong></td>
<td></td>
<td>Go if Milestones 1.1, 1.2, 2.1, and 2.2 are successfully completed.</td>
<td>No-Go if there are challenges associated with these Milestones. We will modify nanoarchitecture of DBCs</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>Extent of Reversibility and Reusability</td>
<td>C1</td>
<td>Achieve a maximum of 20% reduction in viscosity and proppant carrying capacity after 5 stimulus cycles</td>
<td>High-pressure rheometry and sand settling measurements verified with three different samples</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>C2</td>
<td>Achieve a maximum of 10% reduction in viscosity and proppant carrying capacity after 10 stimulus cycles</td>
<td>High-pressure rheometry and sand settling measurements verified with three different samples</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>Compatibility with Other Chemicals in Fracking Fluids</td>
<td>D1</td>
<td>Identify at least one compound that is compatible with DBCs for all functions of clay stabilization, friction reduction, corrosion inhibition, scale inhibition, and iron control suitable for hydraulic fracturing</td>
<td>Aging and phase behavior experiments, visual inspection, and chemical spectroscopy</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td>Computational Models to Describe Proppant Transport and Fracture Propagation</td>
<td>E1</td>
<td>Successful development of dynamic models for 3D, simultaneously growing multiple fractures with at least 2D proppant transport</td>
<td>Experimental validation within 20% accuracy</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>Adsorption and Desorption of DBCs on/from Proppant and Fracture Surfaces</td>
<td>F1</td>
<td>Successful development of dynamic models describing adsorption and desorption of DBCs on proppant surfaces</td>
<td>Laboratory-scale studies to verify the models with a criterion of 20% accuracy</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>Laboratory Experiments to Evaluate Hydraulic Fracturing Performance</td>
<td>G1</td>
<td>Obtain 50% enhancements in fracture permeability and conductivity using DBCs compared to four commercial fracturing fluids</td>
<td>Tests using specimens obtained unconventional reservoirs from Texas</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td><strong>Go/No-Go Decision Point 2</strong></td>
<td></td>
<td>Go if Milestones 3.1–7.1 are successfully completed.</td>
<td>No-Go if there are challenges associated with these Milestones.</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>Computational studies of estimating wastewater recovery and gas production rates after fracturing fluid clean-up</td>
<td>H1</td>
<td>Develop a subroutine for non-Newtonian fluid to compute the modified viscosity due to DBCs and use this subroutine for calculating gas production and water recovery rates</td>
<td>Experimental studies to confirm the validity of models with a criterion of 20% accuracy</td>
<td>9</td>
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<tr>
<td>Scale-up, Large-Scale Manufacturing, and Field Testing of DBCs</td>
<td>I1</td>
<td>Determine all kinetic parameters of DBC formulation with the best laboratory-scale performance, which are the main scale-up parameters</td>
<td>Time-resolved spectroscopic analysis</td>
<td>9</td>
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<tr>
<td><strong>Go/No-Go Decision Point 3</strong></td>
<td></td>
<td>Realize 100 pounds/day production rate at a minimum yield of 85% in a pilot plant specifically designed manufacturing of DBC</td>
<td>Spectroscopic analysis for determining the reaction yield and digital balances for measuring the production rates</td>
<td>10</td>
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<tr>
<td>Cost-Benefit Analysis</td>
<td></td>
<td>Achieve at least 50% improvements in the hydrocarbon recovery on the field tests compared to the current-state-of-art</td>
<td>Coriolis flow meter measurements of natural gas production in the selected well over 10 days</td>
<td>11</td>
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<tr>
<td><strong>Go/No-Go Decision Point 4</strong></td>
<td></td>
<td>Prepare a comprehensive cost-benefit analysis considering raw material cost, production cost, deployment costs, durability, life-time, and potential benefits</td>
<td>No-Go if there are challenges associated with these Milestones.</td>
<td>12</td>
<td></td>
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</tbody>
</table>