# V.O.10 Fuel Cell Coolant Optimization and Scale Up*


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## Objectives

The overall objective of this project is to optimize and scale up the process to make Dynalene FC fuel cell coolant with a great deal of reproducibility. The following are some specific objectives that would help to reach the overall goal of the project:

- Demonstrate the production of one key ingredient of the coolant (a nanoparticle) in 10 L, 20 L and 100 L batches in a pilot-scale operation and study the effect of various process parameters on the size and charge density of the particles.
- Produce the nanoparticles necessary for the fuel cell coolant, Dynalene FC, in a very consistent manner (i.e., particle size, charge density and yield).
- Optimize the filtration process for the nanoparticles to minimize the cleaning time for different scales of operation.

## Technical Barriers

This project addresses the following technical barriers from the Fuel Cells section (3.4.4) of the Fuel Cell Technologies Program Multi-Year Research, Development and Demonstration Plan:

(A) Durability

(B) Cost

## Technical Targets

Dynalene FC is expected to help the fuel cell industry achieve their durability and cost targets to some degree. First of all, the coolant itself is being designed to have a life of 5,000 hrs. It is also expected to have excellent compatibility with the system materials and inhibit corrosion in the coolant loop. This will help in extending the durability of the fuel cell system components such as the pump, the radiator, valves, seals/gaskets and any other components coming in contact with the coolant. The coolant is also designed to work at -40°C, which will assist both transportation and stationary fuel cells to quickly warm up during cold-starts.

The cost target for the coolant (in plant-scale production) is about $10/gallon, which is very close to the retail price of current automotive coolants. This coolant will also eliminate the deionizing filter and other hardware associated with it (i.e. fittings, valves). It is also being designed to work with cheaper, lighter and thermally efficient components such as aluminum radiators (instead of stainless steel) and brass heat exchangers.

## Accomplishments

Dynalene FC has been demonstrated by field testing to maintain a very low electrical conductivity and stay stable over several years. This was possible due to the addition of a nanoparticle into the coolant. This project addresses the optimization and scale up of this nanoparticle ingredient. The main accomplishments in the first few months of the project are the design of the reactor systems, procurement of the reactors and the components and commissioning.

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## Introduction

This project addresses the goals of the Fuel Cell Technologies Program of the DOE to have a better thermal management system for fuel cells. Proper thermal management is crucial to the reliable and safe operation of fuel cells. A coolant with excellent thermophysical properties, non-toxicity, and low electrical conductivity is desired for this application.

Dynalene Inc. has developed and patented a fuel cell coolant with the help of DOE Small Business Innovation Research Phase I and Phase II funding (Project # DE-FG02-04ER83884). However, this coolant can only be produced in lab-scale (500 ml to

2 L) due to problems in optimization and scale up of a nanoparticle ingredient. This project will optimize the nanoparticle production process in 10 L, 20 L and 100 L reactors, optimize the filtration process, and develop a high throughput production method for the final coolant formulation.

## Approach

Dynalene researchers have been producing the nanoparticles ingredients used in the fuel cell coolant in 100 ml, 500 ml and 2 L reactors. The main parameters that contribute to the quality of the nanoparticles are size and surface charge density. Another important factor needs to be optimized is the yield from the production as well as cleaning/filtration. At 100 ml scale, the nanoparticles were produced using magnetic stirring and the heating was provided by a constant temperature bath. At the 500 ml scale, the stirring mechanism was changed to a mechanical stirrer with impellers, while the heating mechanism was still through a constant temperature bath. At the 2 L scale, the stirring was through a mechanical stirrer whereas the heating was accomplished by pumping hot water through the jacket of the reactor. In this project (for 10 L and 100 L reactors), mechanical mixers with multiple impellers and heating systems using the jacket of the reactor will be used. Mixing/stirring mechanism as well as the heating method impacts the particle size, charge density and the yield of the reaction. Therefore, an understanding of the influence of these parameters is very essential to obtain the nanoparticles with reproducible properties. Dynalene and Lehigh University have partnered to develop the scale up criteria needed to go from a 2 L scale to a 10, 20 or 100 L scale.

## Results

Three glass reactors (one 10 L, one 20 L, and one 100 L) were purchased by Dynalene (Figures 1 and 2). During the course of the last quarter all reactors have been received and assembled.

A great deal of time was spent modifying the 20 L reactor that was purchased in an auction and not pre-customized to the specifications necessary to complete the scope of this project. Those modifications are completed and mixing studies have begun to optimize impeller placement and mixing speed. Reactors of this size require two impellers on the stir shaft and proper placement is crucial. The mixing experiments were done using an oil containing dye along with water to simulate the monomer/water mixture that is the basis of the nanoparticle synthesis. The studies are progressing well but more thorough experiments are necessary.

The heating and cooling system for the three reactors has been completed and installed. Heating and cooling experiments on the 20 L reactor have been run with water as the medium to simulate the reaction conditions. Some minor modifications were necessary to correct some initial problems with the system.

The 10 L and 100 L reactors have been assembled and similar testing on these will be performed in the future.

**FIGURE 1.** 20 L Glass Reactor for Nanoparticle Synthesis

**FIGURE 2.** 100 L Glass Reactor for Nanoparticle Synthesis

Optimization experiments on the 500 ml and 2 L scale of the anionic and cationic nanoparticles have led to narrowing some of the independent and dependent variables of scale. These small-scale reactions on the cationic nanoparticles have produced two possible recipes for further scaling. Both of these recipes will be studied at the 10 L scale to determine the best candidate for further work. The variables that need to be considered for scaling are listed below and need to be monitored at each scale up step.

Independent Variables:

- Mixer type
- Angle
- Speed
- Reactor type
- Baffled/non-baffled
- Recipe
- Temperature
- Heat transfer mechanism

Dependent Variables:

- Size
- Surface charge density
- Yield
- Time of reaction

More than 20 batches of anionic nanoparticles were produced at the 2 L scale. All batches showed yield of greater than 20% solids. Purification of the nanoparticles will be carried out at this level. Purification by tangential flow filtration (TFF) requires that the solids concentration be a maximum of 8%. A large dilution was necessary to achieve the proper flow through the membrane. Purification of 500 ml scale reactions can typically take two to three days after dilution so this larger purification would definitely lead to a bottleneck in the process.

The TFF system was set up with two membranes instead of one to double the surface area for cleaning. A dilution of 2 L to approximately 16 L was necessary for purification. A system using a large reservoir and the vacuum caused by the filtering was set up to constantly replenish the water lost during the process. This allowed for continuous filtering at a constant volume and decreased the time needed for cleaning from two or three days down to one day.

Following is a list of parameters for optimization of filtration:

- Flow rate
- Pressure
- Concentration (solids percentage)
- Membrane pore size
- Membrane construction (polyether sulfone or ceramic)
- Additives
- Mixing in reservoir

Dynalene is also studying a new ceramic TFF filter system, which may speed up the filtering process. This system may also have another benefit of allowing purification of both anionic and cationic particles in the same filter. The current process requires two separate systems for filtering. An initial study showed proof of concept in a short-term test with a membrane manufacturer. A longer, more substantial trial is being scheduled at this time.

## Conclusions and Future Directions

Dynalene has set up all the reactors for the nanoparticle production. After some preliminary testing, the 10 L and the 20 L reactors will be used for the synthesis of the nanoparticles. The scale up criteria developed in collaboration with Lehigh University will be verified.

The immediate tasks to be performed are as follows:

- Further testing of the heating and cooling components of the reactor system will be carried out to make sure that they are operating.
- Mixing parameters for the 10 L and 20 L reactors will be optimized.
- Scale up experiments for the anionic and cationic nanoparticles will be started.

## FY 2010 Publications/Presentations

**1.** Mohapatra, S and P. McMullen, "Fuel Cell Coolant Optimization and Scale-up", Poster presented at the Annual DOE Hydrogen Program Review Meeting, June 2010, Washington, D.C.