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Texas Commission on Environmental Quality
New Technology Research & Development (NTRD) Program
Monthly Project Status Report

Contract Number: 582-5-65591-0002

Grantee: The University of Texas at Austin

Date Submitted: April 6, 2005

Report for the **Monthly** period:

Starting Date: March 1, 2005 Ending Date: March 31, 2005

Section I. Accomplishments *(Please provide a bulleted list of project accomplishments as well as a description of their importance to the project.)*

PROJECT OBJECTIVES

The overall objective of this project is to develop cost-effective, high-temperature gas separation membranes for producing inexpensive, high-purity hydrogen (H_2) from synthesis gas generated by steam reforming or gasification of fossil fuels. The two specific technical objectives of the project are:

(i) To develop polymeric and/or polymer-based nanocomposite membranes with high H_2 permeability, high H_2/carbon monoxide (CO) and H_2/carbon dioxide (CO_2) selectivities, and high thermal stability up to 250-300 °C.

(ii) To demonstrate the technical and economic feasibility of using such membranes for producing low-cost fuel-cell-quality H_2 from synthesis gas streams.

PROJECT ACTIVITIES AND STATUS

The Grant Activities for the project consist of seven (7) tasks. The project accomplishments during this report period are summarized in the bulleted list below.

UT

- An additional film of MgO filled Ultem® 1000 has been prepared. **(Task 1)**

RTI

- Negotiations on the Agreement for PBI Polymer or Solution Use were completed between RTI and Celanese Advanced Materials, Inc. (CAMI), and the Agreement was signed by both parties. PBI (polybenzimidazole) polymer was then ordered from CAMI for the project and should be received next month. The acquisition of PBI is important because PBI is one of the key thermally stable polymers with attractive H_2 separation properties to be developed into useful membranes in this project. PBI membranes, with and without nanoparticles, will be the focus of membrane development work at RTI. ***(Project Task 1)***

- At the end of this period, the high-pressure, stainless-steel, tubular membrane module housing and one AccuSep-supported, thin-film PBI membrane tube ordered last month from Pall Corporation were received. RTI plans to begin evaluating the gas separation properties of this tubular PBI composite membrane module next month. Determination of the H_2 separation properties of this PBI composite membrane is an important first step for the project because it will provide a separation-performance reference point based on a current state-of-the-art, supported, thin-film PBI membrane that is in a form readily packageable and scaleable into a useful industrial membrane module. Membrane performance data obtained on this first PBI composite membrane tube will be used to guide our next development steps on how thin to make the selective PBI layer on microporous supports so that the optimal combination of high H_2 selectivity and high H_2 productivity (flux) of this membrane can be obtained for the application of interest. ***(Project Task 1)***

- The high-temperature, high-pressure mixed-gas membrane permeation system at RTI was set up and leak-tested to ready it for the tests in this project. Mass flow controllers on the system and the online gas chromatograph to be used were checked for proper operation and calibrated. These system setup and calibration steps are important to the project because they ensure that the permeation system will operate properly and generate accurate gas permeation data on the membranes to be tested. ***(Project Task 2)***

Specific results and details of this period's project activities are discussed on a task-by-task basis below.

Indicate which part of the Grant Activities as defined in the grant agreement, the above accomplishments are related to:

TASK 1: Prepare High-Temperature Membranes

UT

During this period, UT prepared a film of Ultem® 1000 filled with 7 volume percent MgO.

RTI

After signing the Agreement for PBI Polymer or Solution Use that was negotiated with Celanese Advanced Materials, Inc. (CAMI) this month, RTI, our subcontract partner, purchased ~1 pound of PBI (polybenzimidazole) polymer for the project. When this polymer is received next period, PBI membrane films with and without nanoparticles dispersed in this polymer matrix will be prepared.

Additionally, the high-pressure, tubular membrane module housing plus one AccuSep-supported, thin-film PBI membrane tube ordered from Pall Corporation were received. Testing of this PBI membrane module with a three-component syngas mixture is scheduled for next month. These AccuSep-supported PBI membrane tubes will be a good initial step toward demonstrating the proposed thin-film composite (multilayer) membrane approach as one method to increase the flux of high-temperature polymer membrane materials into a range suitable for commercial deployment.

TASK 2: Evaluate Membrane Permeation Properties

UT

The film from Task 1 is currently being tested for pure gas transport properties.

RTI

The membrane permeation system at RTI was reconfigured to better adapt it for the tests in this project. All connections on the modified permeation system were leak-tested under pressure with helium, and any leaks were eliminated. Helium was also used to calibrate the downstream sweep mass flow controller because it will be used as the sweep gas in permeation tests. The online gas chromatograph (GC) to be used was checked for proper operation and calibrated with two- and five-component gas mixture standards containing such key species as H_2, CO, CO_2, and CH_4. Proper functioning and operation of the permeation apparatus was then validated by characterizing a polymer membrane standard with several pure gases and comparing the data obtained to its known permeation properties.

TASK 3: Evaluate Membrane Reactor Properties

RTI

This task is not yet scheduled to begin at RTI.

TASK 4: Characterize Thermal and Morphological Properties of Membranes

UT

No significant activities occurred for this task at UT during this report period.

RTI

No significant activities occurred for this task at RTI during this report period.

TASK 5: Prepare Integrated System Process Design

RTI

This task is not yet scheduled to begin at RTI.

TASK 6: Perform Technical and Economic Analysis/Develop Commercialization Strategy

RTI

This task is not yet scheduled to begin at RTI.

TASK 7: Manage Project/Prepare Reports

UT/RTI

The second monthly project report was prepared.

Section II: Problems/Solutions

Problem(s) Identified *(Please report anticipated or unanticipated problem(s) encountered and its effect on the progress of the project)*	UT No problems were encountered this period. RTI No problems were encountered this period.
Proposed Solution(s) *(Please report any possible solution(s) to the problem(s) that were considered/encountered)*	UT N/A this period. RTI N/A this period.
Action(s) Conducted and Results *(Please describe the action(s) taken to resolve the problem(s) and its effect)*	UT N/A this period. RTI N/A this period.

Section III. **Goals and Issues for Succeeding Period**: (*Please provide a brief description of the goal(s) you hope to realize in the coming period and identify any notable challenges that can be foreseen)*

UT

During the next period, we plan to prepare a range of nanocomposite samples based on Ultem® and MgO nanoparticles. We will also extend our studies to Matrimid polyimide, in the hopes of identifying a second polymeric matrix that we could use for composite materials preparation. We will use MgO as well as other particles, from the family of particles described in the proposal, at various loadings and sizes to understand the influence of these variables on nanocomposite permeability and selectivity. We also plan to begin thermal and related characterization of the structure of the nanocomposites during this period.

RTI

Next period, the AccuSep-supported PBI composite membrane module will be tested with a ternary syngas mixture of H_2, CO_2, and CO as a function of feed pressure and temperature (up to 250-300 °C). Additionally, when the raw PBI polymer is received, neat (nanoparticle-free) and nanoparticle-doped PBI membrane films will be prepared via solution-casting and characterized as they are ready. Thermal stability of these PBI-based films will also be determined using thermogravimetric analysis (TGA). To determine the effect of nanoparticle addition on the thermal properties of the base polymer, the TGA data for PBI nanocomposites will be compared to that measured for neat PBI.

Benny Freeman

Authorized Project Representative's Signature

Date: 4/6/05

NOTE: *Please attach any additional information that you feel should be a part of your report or that may be required to meet the deliverable requirements for tasks completed during this reporting period.*