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**OAK RIDGE
NATIONAL
LABORATORY**

MARTIN MARIETTA

**CRADA Final Report
for
CRADA Number ORNL92-0106**

**USE OF ELECTRIC FIELDS TO ENHANCE INDUSTRIAL
CHEMICAL PROCESSING**

**T. C. Scott
Oak Ridge National Laboratory**

**M. D. Bowe
Rohm & Haas Company**

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DEPARTMENT OF ENERGY**

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C/ORNL-92-0106

MARTIN MARIETTA ENERGY SYSTEMS, INC.

POST OFFICE BOX 2009
OAK RIDGE, TENNESSEE 37831

December 8, 1994

Mr. Peter D. Dayton
Director, Procurement and Contracts
Department of Energy, Oak Ridge Operations
Post Office Box 2001
Oak Ridge, Tennessee 37831-2001

Dear Mr. Dayton:

Final Report for CRADA No. ORNL92-0106 with Rohm & Haas

The subject CRADA has been completed and enclosed is the Final Report for this project.

This report does not contain proprietary information or Protected CRADA Information. Neither Energy Systems nor the participant object to public distribution of this report.

If you have any questions, please feel free to contact me.

Very truly yours,

A handwritten signature in cursive script, appearing to read "C. A. Veltman".

for Brian Bovee
Business Manager
Office of Technology Transfer

BBB:cav

Enclosure - As Stated.

cc: File - RC

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Internal Correspondence

I 18830

MARTIN MARIETTA ENERGY SYSTEMS, INC.

Date: October 19, 1994

To: W. P. Painter

From: T. C. Scott, 4505, MS-6226 (4-5962)

Subject: Final Summary Report for CRADA No. ORNL 92-0106

Enclosed is a final summary report of progress made in CRADA No. ORNL 92-0106 entitled "Use of Electric Fields to Enhance Industrial Chemical Processing" between ORNL and the Rohm & Haas (R&H) Company. Neither Martin Marietta Energy Systems, Inc. (MMES) nor R&H asserts any claim to information contained in the final report as qualifying as "Protected CRADA Information." In addition, MMES and R&H have no objection to public distribution of the final report. The report contains no Proprietary Information.

Timothy C. Scott, Ph.D.
Oak Ridge National Laboratory

Date

Michael D. Bowe, Ph.D.
Rohm & Haas Company

Date

TCS:jst

Enclosure

FINAL REPORT
CRADA No. ORNL 92-0106
BETWEEN
OAK RIDGE NATIONAL LABORATORY
AND
ROHM & HAAS COMPANY

**USE OF ELECTRIC FIELDS TO ENHANCE
INDUSTRIAL CHEMICAL PROCESSING**

Timothy C. Scott, David W. DePaoli, and Michael D. Bowe

ABSTRACT

The Rohm and Haas Company (R&H) and Martin Marietta Energy Systems, Inc. (MMES) agreed in 1992 to enter a two-year Cooperative Research and Development Agreement (CRADA). This research effort called upon the unique capabilities that exist in the Energy Research Section of the Chemical Technology Division of Oak Ridge National Laboratory (ORNL) to investigate the use of electric fields to enhance R&H process operations involving multiphase chemical separations and synthesis of materials in multiphase reactive systems. The goal of the effort was to carry out research that would yield greater than order-of-magnitude improvements in processing efficiency. Efforts were made to apply novel electrically enhanced chemical processing technology to improve an existing R&H process. R&H identified a target system for removal of undesired organic chemicals from aqueous polymer suspension products by solvent extraction. Several improvements in the EPC were made to address problems posed by this viscous aqueous feed with high solids content. These developments will have a positive impact in other applications of EPC technology; however, the physical properties of the polymer suspensions make them poorly suited to this approach.

FINAL REPORT
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Timothy C. Scott, David W. DePaoli, and Michael D. Bowe

The Rohm and Haas Company (R&H) and Martin Marietta Energy Systems, Inc. (MMES) agreed in 1992 to enter a two-year Cooperative Research and Development Agreement (CRADA). This research effort called upon the unique capabilities that exist in the Energy Research Section of the Chemical Technology Division of Oak Ridge National Laboratory (ORNL) to investigate the use of electric fields to enhance R&H process operations involving multiphase chemical separations and synthesis of materials in multiphase reactive systems. The goal of the effort was to carry out research that would yield greater than order-of-magnitude improvements in processing efficiency. The development of advanced separations/reactive systems is both a stated goal of the ORNL program with the U. S. Department of Energy and a necessity for R&H to gain competitive advantages over other industrial entities. The desired result of this program was successful operation of advanced ORNL technologies utilizing actual R&H process fluids. Potential technologies included: (1) electrified solvent extraction for aqueous-phase cleanup, (2) electrified coalescence for breaking water-in-oil emulsions, (3) dielectric filtration of submicron particles from organic media, and (4) manufacture of single and multicomponent ceramic particles.

Upon discussion with R&H, priority was placed upon development of advanced methods for removal of undesired organic compounds from aqueous polymer dispersions. Successful development of such a process would represent a breakthrough in product quality, providing R&H a significant competitive advantage in a segment of their products. It would also provide an opportunity for testing of the emulsion phase contactor (EPC), an electrified solvent extraction device, with a viscous aqueous feed of high solids content.

The emphasis at R&H was placed upon identifying solvents which are compatible with aqueous polymer suspensions, while the emphasis at ORNL was upon improving the design of the bench-scale emulsion-phase contactor (EPC) system to be used in extraction tests with R&H fluids. R&H determined a suitable organic solvent for extraction of the aqueous suspensions; initial shake-out tests by R&H with two generic polymer suspensions, labelled JR1501A and JR1507, indicated that a significant amount of the target compounds are removed from the aqueous polymer solution and the solid polymer particles with a single one-to-one contact. The extraction appears to take place without significant detrimental effects to the polymer particles. However, in low-surface-area tests, the removal is time-dependent on the scale of hours; hence,

a high-surface-area system like the EPC is required for efficient operations.

The two primary issues which impeded the application of the EPC at the initiation of this project involved the high wall-area-to-volume ratio characteristic of all small-scale experiments and the need to provide for significant system hold-up to assess mass transfer performance in the absence of end effects. Design changes addressing the first of these two problems involved the use of a hydrophobic film on the front and rear walls of the experimental EPC. Machined pieces of the film were inserted on the inside of the front and rear walls of the contactor. The film serves both as a hydrophobic screen to prevent the build-up of aqueous phase and as a seal to the front and rear panels of the column. The system was tested on an aqueous/organic fluid pair comprised of distilled water and kerosene. The film was able to prevent build-up of aqueous phase on the front and rear walls of the EPC and perform as a seal without any detectable leakage. This approach provides a definite improvement over the previous system.

M. D. Bowe of R&H travelled to ORNL on June 10-11, 1993 to observe operation of the bench-scale EPC devices with these fluids. The smaller experimental EPC was operated with the JR1501A feed. Droplet breakup and coalescence were achieved readily; however, conducting films were formed which caused continuous arcing and loss of field strength. After repeated attempts at operation, it was determined that this experimental design will not work with the fluids.

A larger EPC device, designed to reduce edge effects, was completed. This device was constructed with a two-foot electrode length, or approximately one-fourth of that envisioned for full-scale industrial use. This EPC was operated with both R&H feeds. The JR1507 feed exhibited good dispersion and coalescence performance. Thin, mobile films were formed along the walls and the electrodes. Film formation on electrode supports eventually led to arcing and loss of dispersing capability due to the lower field strength. These films disengaged from the surfaces upon deactivation of the electric field. The JR1501A material was fed to the EPC at full strength and diluted by 20 volume percent with water. Although the diluted system performed slightly better, in both cases the performance was undesirable due to coagulated product.

Additional partitioning studies were conducted by R&H to determine the extent and rate of transfer of target compounds from the model fluids to the solvent and to detect any incompatibilities with the solvent. Tests were conducted by contacting 5 g samples of process fluid with 25 g of solvent for up to an hour. Transfer of a target compound from JR1507 was completed in 15 minutes, a reasonable residence time in a full-scale process vessel. The partitioning from JR1501A was proven by 90 percent or greater removal of two target compounds in 60 minutes, with no intermediate samples taken. Particle size was maintained in both fluids, indicating that the particles are stable with contact with the solvent. The stability in these tests indicate that the coagulation problems noted in the EPC runs is not due to the EPC operations; rather, it is likely shear-induced from the feed pump and/or the electrical dispersion process.

EPC design modifications aimed at reducing arcing at the electrode supports were completed. Operational testing conducted using water as the aqueous feed indicated that the design modifications were successful; however, further development of the contactor is necessary to eliminate buildup of aqueous feed along the walls of the device. Due to this difficulty, the design is not well-suited to viscous polymer suspensions. Operation of this modified EPC system resulted in the discovery of a phenomenon which may have significant impact on the DOE-supported ORNL basic research — very long and slender electrically stabilized liquid

bridges were repeatably formed. This is a novel finding and one which will provide opportunity for further fundamental research.

Testing was conducted using a single-stage electrically-driven extraction process based upon the modular electrospray nozzle developed at ORNL for the electric dispersion reactor. The process would have less electrified surface area than the EPC devices used, and thus would be less susceptible to problems caused by viscous film formation. It would also be practical in the test systems considered, in which a single equilibrium stage resulted in approximately 90% removal of target compounds. Tests focused on feasibility of the electrical dispersion. Dispersion of the R&H feed designated JR1507 in organic solvent at a volume ratio of 0.2% was performed successfully, but phase separation was not satisfactory. The JR1501A feed formed fine solid particles that coagulated, much like the behavior in the EPC. The JR1507 feed was dispersed successfully at a 1.0% volume ratio, resulting in a suspension with good phase separation. However, long-term operation of the device was hampered by film formation and electrical breakdown along the insulation near the electrodes.

SUMMARY

Efforts were made to apply novel electrically enhanced chemical processing technology to improve an existing R&H process. R&H identified a target system for removal of undesired organic chemicals from aqueous polymer suspension products by solvent extraction. Several improvements in the EPC were made to address problems posed by this viscous aqueous feed with high solids content. These developments will have a positive impact in other applications of EPC technology; however, the physical properties of the polymer suspensions make them poorly suited to this approach.