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LLNL-TR-744856

Textile Resource Conservation Final Report CRADA No. TC-0699-93

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January 22, 2018

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Textile Resource Conservation

Final Report
CRADA No. TC-0699-93

Date: September 11, 2000

Revision: 3

A. Parties

This is an agreement between Lawrence Livermore National Laboratory (LLNL) and the Institute of Textile Technology (ITT).

Lawrence Livermore National Laboratory
7000 East Avenue
Livermore, CA 94551

Institute of Textile Technology
2551 Ivy Road
Charlottesville, VA 22903-4614

B. Project Scope

This project was undertaken to develop and demonstrate on a pilot scale the use of electro-osmotic transport to increase the efficiency of textiles wet processing operations. In particular, we sought to develop a means of rinsing textiles to remove material entrapped between the individual fibers that constitute a yarn. Material trapped within the yarn is slow to exchange with rinse water flowing primarily in the open weave are abetween the yarns. The application of an external field (strength, 5-50 kV/m) requires only a few volts for most fabric thicknesses. This field is sufficient to promote a rapid exchange of material to enhance rinsing and reduce the water required for rinsing from about 20 kg/kg-fabric to 3-6 kg/kg-fabric. We successfully developed technical and economic models of application of the process to the rinsing of many materials of industrial importance, including dyes, tints, chemicals, detergents and dye electrolytes. We demonstrated the process on a pilot plant scale using a translator designed in cooperation with Milliken and Company (Spartanburg, SC).

The project identified but could not pursue the testing of related processes whereby electro-osmosis is used to speed the penetration of chemicals and dyes into a fabric while minimizing the use of water. All technical developments and discoveries for both the rinsing and chemical-applications processes are covered by an issued patent evolved from this research.

This project used technology within the Department of Energy's (DOE) National Laboratories to speed the infusion of resource conservation technology into the American Textile Industry. Its purpose was to assist the American textile industry in regaining its economic competitiveness. The project involved several national laboratories and ITT, comprised of about fifty U.S. companies in the textile industry. This report focuses on the effort and contributions of LLNL and ITT.

At the time of the CRADA, The Textile Resource Conservation (TReC) Project was the only project in the Environmental Quality and Waste Minimization (EQ/WM) Technology Area of the American Textile Partnership (AMTEX™) between the DOE, the Integrated Textile Industry, and the National Laboratories. These technologies cut across all modes of textile manufacturing and included but were not limited to separation and recovery techniques, the quantification and reduction of air emissions, high efficiency chemical applications, and improved cleaning and washing. Applications of waste-minimization techniques as well as the most efficient methods of resource recovery and reuse technology would increase the competitiveness of the U.S. textile industry.

The objective of the TReC project was to provide technology and methods allowing full utilization of the resources required to produce high quality textile products.

C Technical Accomplishments

The technical accomplishments of this project (one of several within the TreC designation) were as follows:

- 1994: Invention and proof of principle of use of electro-osmotic rinsing in textiles
- 1995: Continued small scale tests of the electro-osmotic process.
- 1996: Operation of plane-parallel cells (600 cm²) for limited tests on moving fabric
- 1997-8: Design and fabrication of a pilot-scale system for testing the process

Multiple testing on tints, dyes, and caustic solutions.

The process was found successful for the removal of low conductivity materials (range of 100-1000 micro-mho/cm) but clearly inapplicable to the removal of very concentrated electrolytes, such as the 4-20% caustic solutions used in mercerization. The process is most applicable at this point to niche applications including the treatment of microfiber fabrics and yarns, and to the applications of chemicals under conditions of minimal water use.

There were nine tasks comprising this TreC Project:

1. Recovery of Colorants and Auxiliaries
2. Recovery and Reuse of Fibrous Solid Waste
3. Metals Speciation
4. Sensing and Reduction of Air Emissions
5. Low Waste Chemical Application Methods
6. Alternative Cleaning Technologies
7. Strategic Planning for Source Reduction through Process Development
8. Integrated Energy Conservation Strategic Planning
9. Communications and Coordination

LLNL supported tasks 5, 7, and 8. Task 5 focused on developing chemical application systems that generate low amounts of waste, that allow rapid process changeover, and that provide energy reduction opportunities in textile chemical applications. Task 7 focused on full utilization of material resources, with the ultimate goal of zero discharge, preservation of environmental quality while substantially improving the international competitiveness of the U.S. textile industry. The goal of Task 8 was to provide data and methods for improving energy research directions.

D. Expected Economic Impact

The purpose of the program was to assist the American textile industry in regaining its economic competitiveness. The objective of the TReC project was to provide technology and methods allowing full utilization of the resources required to produce high quality textile products. As with many manufacturing industries, many textile processes are not 100% efficient, which results in unused raw materials that become waste. The TReC project was designed to develop and deploy technologies to assist in the production of textile products with minimized wasted raw materials, thereby reducing the cost of waste disposal.

D.1 Specific Benefits:

TReC provided significant long-term benefits to maintaining domestic employment, making the largest single segment of our Gross National Product competitive with international markets, and provided a significant benefit to reducing the negative environmental impacts resulting from textile manufacturing. Additionally, the industry-led technique employed in this program could serve as a significant demonstration to an effective coupling of our National Laboratory resources with commercially justifiable use.

The broad scope of this industry and the broad range of company sizes participating simultaneously provided major benefits to both large and small domestic firms.

The project represented a significant contribution by the DOE to the national Textile Industry, through retention of employment and to the long-term health and welfare of the U.S. environment.

E. Partner Contribution

Our primary industrial contacts were with Institute of Textile Technology and with Milliken and Company (Spartanburg SC), which provided very frequent technical review and consultations. In addition Milliken supplied samples of fabrics and chemicals for use in the program, and provided insights into important economic and industrial constraints. In addition, ITT convened a steering committee which regularly reviewed and advised the project.

The Industrial Research Partners (IRPs) worked with National Laboratory researchers in the Energy Conservation Strategic Planning task as an integral part of the Task Team and made significant contributions to the effort in several areas.

In the initial phase of the task effort, the IRPs helped the National Laboratories locate the information needed to compile energy data on textile manufacturing. The IRPs worked with the National Laboratories in selecting representative facilities in which to conduct a field review of textile manufacturing operations -- including cotton and synthetic fiber production, textile manufacturing (e.g., yarn spinning, weaving, dyeing and finishing), product fabricators (e.g., domestic and apparel), distribution and retail, as well as hosting visits to the selected facilities.

IRPs participated in the data collection and analyses with the process models and techniques employed by the national laboratories. A review of the energy conservation ranking system developed under this task was necessary by the industry participants and was one of the tasks for them to give guidance and direction to the National Laboratories.

Finally, the IRPs participated in the prioritizations, evaluations and recommendations.

F. Documents/Reference List

"Applications of Electro-Osmosis to Rinsing of Fabrics," Final Report: Amtex TreC-V: 1995-1998 by John F. Cooper and Roger Krueger, LLNL

Subject Inventions:

CRADA Article I defines Subject Invention as any invention of LLNL or ITT or ITT's agent conceived or first actually reduced to practice as defined under the United States patent laws in the performance of work under this CRADA.

Under CRADA Article XIV the Parties agree to disclose to each other each and every Subject Invention, which may be patentable or otherwise protectable under the Patent Act. The disclosures should be in such detail as to be capable of enabling one skilled in the art to make and use the invention under 35 USC 112.

LLNL Subject Inventions:

IL-9379, "Electro-Osmotic Transport in Wet Processing of Textiles" John F. Cooper. Disclosure date 10/19/93. U.S. patent application filed 1/17/96. USP #5,810,996 issued 9/22/98.

ITT and ITT Agent Subject Inventions:

None

Joint Subject Inventions:

None

Copyrightable Computer Software:

CRADA Article XV provides that each Party shall own the title in Intellectual Property, defined as patents, copyrights, trademarks, and mask works, created solely by its employees or agents, and that title in jointly made intellectual property shall be jointly owned.

Under CRADA Article XIII, the Party owning the copyright in any copyrighted computer software produced in the performance of this CRADA must provide the source code, an expanded abstract, the object code, and the minimum support documentation needed by a competent user to understand and use the software to DOE's Energy Science and Technology Software Center.

Licensing Status:

Under CRADA Appendix C LLNL grants ITT an option to enter into a License Agreement containing provisions granting ITT and its member entities an exclusive, worldwide, paid-up license to practice, including the right to sublicense, in the Field of Use specified in Appendix C, Intellectual Property owned solely by LLNL which is developed under this CRADA. ITT and its member entities grant to LLNL an option to enter into a License Agreement containing provisions granting LLNL an exclusive, worldwide, paid-up license to practice, including the right to sublicense, outside the Field of Use specified in Appendix C, Intellectual Property owned solely by ITT which is developed under this CRADA. Appendix C also provides for cross-licensing of jointly-developed Intellectual Property. The term of the options

granted in Appendix C is the term of the CRADA plus 6 months after the conclusion of work under the CRADA.

G. Acknowledgement

Participant's signature of the final report indicates the following:

- 1) The Participant has reviewed the final report and concurs with the statements made therein.
- 2) The Participant agrees that any modifications or changes from the initial proposal were discussed and agreed to during the term of the project.
- 3) The Participant certifies that all reports either completed or in process are listed and all subject inventions and the associated intellectual property protection measures generated by his/her respective company and attributable to the project have been disclosed and included in Section E or are included on a list attached to this report.
- 4) The Participant certifies that if tangible personal property was exchanged during the agreement, all has either been returned to the initial custodian or transferred permanently.
- 5) The Participant certifies that proprietary information has been returned or destroyed by LLNL.



Dan J. McCreight
Institute of Textile Technology

10-27-2000

Date



John Cooper
Lawrence Livermore National Laboratory

11/22/00

Date

Attachment I – Final Abstract
Attachment II – Project Accomplishments Summary

Textile Resource Conservation

Final Abstract
Attachment I
CRADA No. TC-0699-93

Date: September 11, 2000

Revision: 2

This project was undertaken to develop and demonstrate on a pilot scale the use of electro-osmotic transport to increase the efficiency of textiles wet processing operations. In particular, we sought to develop a means of rinsing textiles to remove material entrapped between the individual fibers that constitute a yarn. Material trapped within the yarn is slow to exchange with rinse water flowing primarily in the open weave are between the yarns. The application of an external field (strength, 5-50 kV/m) requires only a few volts for most fabric thicknesses. This field is sufficient to promote a rapid exchange of material to enhance rinsing and reduce the water required for rinsing from about 20 kg/kg-fabric to 3-6 kg/kg-fabric. We successfully developed technical and economic models of application of the process to the rinsing of many materials of industrial importance, including dyes, tints, chemicals, detergents and dye electrolytes. We demonstrated the process on a pilot plant scale using a translator designed in cooperation with Milliken and Company (Spartanburg, SC).

The project identified but could not pursue the testing of related processes whereby electro-osmosis is used to speed the penetration of chemicals and dyes into a fabric while minimizing the use of water. All technical developments are covered by an issued patent arising with this research.

This project used technology within the Department of Energy's (DOE) National Laboratories to speed the infusion of resource conservation technology into the American Textile Industry. Its purpose was to assist the American textile industry in regaining its economic competitiveness.

At the time of the CRADA, The Textile Resource Conservation (TReC) Project was the only project in the Environmental Quality and Waste Minimization (EQ/WM) Technology Area of the American Textile Partnership (AMTEX™) between the DOE, the Integrated Textile Industry, and the National Laboratories. These technologies cut across all modes of textile manufacturing and included limited to separation and recovery techniques, the quantification and reduction of air emissions, high efficiency chemical applications, and improved cleaning and washing. Applications of waste-minimization techniques as well as the most efficient methods of resource recovery and reuse technology would increase the competitiveness of the U.S. textile industry.

The objective of the TReC project was to provide technology and methods allowing full utilization of the resources required to produce high quality textile products.

Textile Resource Conservation

Project Accomplishments Summary (Attachment II)

CRADA No. TC-0699-93

September 11, 2000

Revision: 3

A. Parties

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Institute of Textile Technology
2551 Ivy Road
Charlottesville, VA 22903-4614

B. Background

In the US alone, about 10 billion pounds of cotton fabric are processed each year use wet chemicals that require subsequent rinsing. In some unit rinsing steps, about 20 lbs of water are required to rinse each pound of fabric. The waste water must be reprocessed (prior to reuse or discharge) at a high dollar and energy cost. We have invented, patented and developed a process which reduces water requirements for many rinsing operations to as low as 3-5 kg-water per kg of fabric, using electro-osmosis to accelerate the exchange of liquids between the tightly packed interior of yarns and the more open spaces between the yarns. While not applicable to all materials being rinsed from fabrics, this process is under consideration for commercial applications in a variety of rinsing and chemicals-applications processes. The process is most applicable to rinsing materials with low electrolytic conductivity, such as some dyes, tints, chemicals and detergents. It is marginally applicable or inapplicable to rinsing materials of very high electrolytic conductivity, such as concentrated caustic solutions.

The U.S. Integrated Textile Industry is a major contributor to the national economy. The Textile Industry is a leader in implementing world-class manufacturing concepts and systems. It is the single largest industrial sector and contributes more to the gross domestic product than the automotive, petroleum, and primary metals industries. It is also the largest employer of women and minorities. However, in addition to enhanced sensitivity to energy and environmental concerns within the U.S., the domestic textile industry contains inefficiencies that reduce productivity and that make it vulnerable to foreign competition.

In 1991, apparel-related imports accounted for about 60% of U.S. clothing sales, more than double the 1980 level. That decade saw a loss of more than 400,000 jobs to foreign competition. Subsequent losses to the industry are upwards of \$25 billion annually.

Finding effective ways to turn this tide of rising imported goods and movement of jobs outside the U.S. borders was critical to our national economy. Revolutionary thinking and action was required to restore the industry to a competitive position in the global economy. The U.S. Textile Industry must drive the global standard in being responsive to the needs and wishes of the American consumer while competing in a worldwide market. This exceptional response to the consumer would overcome the economic advantage of non-domestic, low-cost labor.

The National Laboratories support the industry in three ways: (1) they offer an appropriate mechanism to focus the government investment in development of a whole industry view of the changes needed to rebuild this industry; (2) they have expertise in developing technology and rapidly demonstrating its possibilities; and (3) they are nonpartisan and can successfully address the cross-cutting problems difficult for single enterprises.

C. Description

The purpose of the project was to develop electro-osmotic transport as a means of increasing the efficiency of the rinsing process. While not developed by us, the same process may be used in reverse: i.e., to speed the transport of chemicals such as fire-retardants, wet-proofing or inks and dyes into the small spaces between fibers comprising the individual yarns. We derived relations between field strength (to 60 kV/m), fabric translation rate (to 30 m/minute), water flow rate and concentration reduction. The industry provided direct review and consultation services, provided samples of fabric materials, dyes and tints, and general guidelines as to where this process fits into existing industrial practice. In particular, Milliken and Company and the Institute of Textiles Technology (Virginia) were particularly helpful and supportive of this effort.

The objective of the TReC project was to provide technology and methods allowing full utilization of the resources required to produce high quality textile products. As with many manufacturing industries, many textile processes were not 100% efficient, which resulted in unused raw materials that became waste. The TReC project was designed to develop and deploy technologies to assist in the production of textile products with minimized wasted raw materials, thereby reducing the cost of waste disposal. The material to be conserved for this project was simply clean, low-conductivity water flowing in the streams of the Atlantic south east and now used in fabrics processing. The project involved several national laboratories and ITT, comprised of about fifty U.S. companies in the textile industry. This report focuses on the effort and contributions of LLNL and ITT.

D. Expected Economic Impact

The purpose of the program was to assist the American textile industry in regaining its economic competitiveness. The objective of the TReC project was to provide technology and methods allowing full utilization of the resources required to produce high quality textile products.

At this writing, the process is under consideration for the rinsing of micro-fiber textiles, and for injection of lubricants and water-proofing into heavy textiles used in rugs and automobile interiors. Longer range applications are also under consideration in general rinsing of fabrics following treatments with dyes and chemicals. The process has not been found to be applicable without further development to the rinsing of caustic following mercerization treatments—one of the most important applications economically. This is because of the high conductivity of the caustic solutions, which dissipates the high field strengths required for electro-osmotic transport. This applications requires more development and industrial based testing for process optimization. If implemented to the mercerization rinsing stage following mercerization (treatment with caustic), then a potential exists for reducing fabric cost by about 5 cents per sqyd, or on the order of \$100 M annually. The ancillary benefits of the process are reduced cost of treatment of effluents, lower rates of discharge of waste materials into flowing streams and increased availability of prime runoff water in the Eastern Seaboard states.

E. Benefits to DOE

This process benefits the development of transport modeling at LLNL, with very wide-scale applications. Electro-osmotic transport is one of several electro-kinetic transport phenomena whereby an interaction of an externally-applied field with the space charge in the double layer causes movement of either the liquid or the solid phase.

Applications in other DOE projects include: electro-kinetic transport for subsurface remediation of contaminated soils, enhancement of electrical tomographic images of underground plumes and fluid flow, fabrication of certain composite materials that are compatible with aqueous solutions but which require processing at ambient temperatures, electro-osmotic valves for controlled injection of water into hydride reservoirs for generation of hydrogen gas for fuel cells, and control of transport in engineering materials with porosity such as filters or barriers. The mathematic models describing these applications are analogous.

The tasks performed by the National Laboratories under the TReC project helped maintain the dual use capabilities of these Laboratories developed and used by other Department of Energy programs. Separation sciences being advanced for recovery of colorants and auxiliaries as well as for the recovery of fibrous solid wastes utilized expertise from DOE Waste Management and Defense programs, and could benefit the Department with increased capabilities for the separation of waste materials resulting from energy and defense production activities. Research both in metals speciation analytical development and air emissions monitoring sensor development used the expertise developed at the Laboratories in methods and instrumentation development. Such efforts were consistent with the dual use nature of the work.

The fiber and textile industry need to understand the behavior of hazardous metals such as antimony that are used in fiber manufacture and treatment. These metals may be volatile under high temperature conditions and, if so, disqualify the textile from environmentally friendly certification in certain markets. DOE was interested in the volatility of these materials because of high temperature mixed waste treatment technologies including thermal treatment processes to produce final waste forms for residues from treating mixed waste.

This work could benefit both DOE Energy Research and Defense Programs efforts by providing through the National Laboratories increased capabilities for the monitoring and determination of discharges from energy and weapons production facilities. These capabilities had applications in both the environmental compliance and treaty verification areas of the Department of Energy. Low waste chemical application methods as well as the development of alternative techniques for cleaning would provide several benefits to the Department of Energy.

Reduction in waste volumes due to more efficient chemical application and parts and equipment cleaning methods results in lower waste production and higher process efficiencies in energy and weapons production systems. For example, electro-osmosis technology, included in this effort by LLNL for low waste applications of surface applied chemicals, was being developed and used in the DP complex for cleaning of concrete contaminated with radionuclides and heavy metals.

Other uses of electro-osmosis technology included both the regeneration of spent porous filters and the enhancement of filtration in porous media for radioactive and hazardous waste. The enhancement of this type of technology, applicable to reduction of waste in both the textile industry and DP complex would also be especially useful in the full detailed planning necessary for the fossil energy plant of the future (Combustion 2000 program) and the weapons complex of the future. In a similar area, the systems analysis and modeling capabilities being developed under the strategic planning efforts for process modification and energy conservation would be applied to the more efficient planning and evaluation of defense and energy technologies and processes.

F. Industry Area

Any industry using or employing textiles can benefit from this technology, including the textiles production industries, automotive industries, filtration industries, ground-water measurements and remediation, and industries fabricating exotic composite materials.

G. Project Status

The project was completed in 1997.

H. LLNL Point of Contact for Project Information

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Livermore, CA 94550
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Fax: 925-422-8020
Email: koopman1@llnl.gov

I. Company Size and Point(s) of Contact

The Institute of Textile Technology in Charlottesville, VA was founded in 1944 to become a privately financed, not-for-profit, institute for textile research. Innumerable companies participated in the partnership in an oversight function. Particularly active was Milliken and Company, Spartanburg SC, one of the largest single cotton fabric manufacturers in the US.

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804-296-5511
Fax: 804-296-2957

J. Project Examples

We have video as well as color photographs showing the removal of a red tint material using electro-osmotic transport implemented on a pilot plant level (translation of 0.5 m wide fabric at rates up to 23 m/min). These and other photographs are published in the final report of the project. A collection of high quality digital photographs, professionally taken, are available on a CD ROM.

K. Intellectual Property

Subject Inventions:

CRADA Article I defines Subject Invention as any invention of LLNL or ITT or ITT's agent conceived or first actually reduced to practice as defined under the United States patent laws in the performance of work under this CRADA.

Under CRADA Article XIV the Parties agree to disclose to each other each and every Subject Invention, which may be patentable or otherwise protectable under the Patent Act. The disclosures should be in such detail as to be capable of enabling one skilled in the art to make and use the invention under 35 USC 112.

LLNL Subject Inventions:

A patent has been issued covering this work:

John F. Cooper, "Electro-osmotic transport in wet processing of textiles," US Patent No. 5,810,996 Sep. 26 1998.

ITT and ITT Agent Subject Inventions:

None.

Copyrightable Computer Software:

CRADA Article XV provides that each Party shall own the title in Intellectual Property, defined as patents, copyrights, trademarks, and mask works, created solely by its employees or agents, and that title in jointly made intellectual property shall be jointly owned.

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LLNL Computer Software developed under this CRADA:

ITT and ITT Agent Computer Software developed under this CRADA:

Jointly developed Computer Software developed under this CRADA:

Licensing Status:

Under CRADA Appendix C LLNL grants ITT an option to enter into a License Agreement containing provisions granting ITT and its member entities an exclusive, worldwide, paid-up license to practice, including the right to sublicense, in the Field of Use specified in Appendix C, Intellectual Property

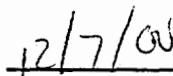
owned solely by LLNL which is developed under this CRADA. ITT and its member entities grant to LLNL an option to enter into a License Agreement containing provisions granting LLNL an exclusive, worldwide, paid-up license to practice, including the right to sublicense, outside the Field of Use specified in Appendix C, Intellectual Property owned solely by ITT which is developed under this CRADA. Appendix C also provides for cross-licensing of jointly-developed Intellectual Property. The term of the options granted in Appendix C is the term of the CRADA plus 6 months after the conclusion of work under the CRADA.

L. Release of Information

I certify that all information contained in this report is accurate and releasable to the best of my knowledge.



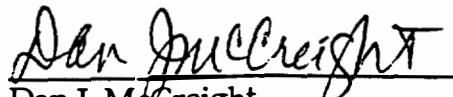
Karena McKinley, Director
Industrial Partnerships
and Commercialization



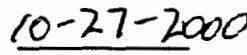
12/7/00
Date

RELEASE OF INFORMATION

I have reviewed the attached Project Accomplishment Summary prepared by Lawrence Livermore National Laboratory and agree that the information about our CRADA may be released for external distribution.



Dan J. McCreight
Institute of Textile Technology



10-27-2000
Date