ELECTROCHEMICAL PATHWAYS FOR SUSTAINABLE MANUFACTURING

A roadmap for electrochemical science and technology in the chemical industry



PREPARED & DEVELOPED BY OHIO UNIVERSITY

FUNDED BY THE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY UNDER GRANT NUMBER 70NANB14H052



THE RUSS COLLEGE OF ENGINEERING AND TECHNOLOGY Create for Good. This report was prepared with funding from the National Institute of Standards and Technology's Advanced Manufacturing Technology Consortia (AMTech) program under award number 70NANB14H052.

<u>Funded Participants</u> Dr. Gerardine G. Botte (Primary Author) *Ohio University*

Polymer Ohio

With special thanks to:

<u>Project Executive Committee</u> Michael Gallagher *Covestro (formerly Bayer MaterialScience)*

Dr. Paul Kohl Georgia Institute of Technology

Dr. Michael Lowe Dow Chemical

Dr. E. Jennings Taylor Faraday Technology Inc.

<u>Program Manager</u> Jean-Louis Steadmann National Institute of Standards and Technology

1. Table of Contents

<u>2.</u>	EXECUTIVE SUMMARY4
<u>3.</u>	GRAPHICAL ABSTRACT6
<u>4.</u>	BACKGROUND7
<u>5.</u>	ROADMAP DEVELOPMENT PROCESS
5.1.	ESTABLISHED ORGANIZATIONAL STRUCTURE: EXECUTIVE COMMITTEE (EC); ADVISORY BOARD (AB)10
5.2.	NEEDS ASSESSMENT
5.2.	1. NEEDS ASSESSMENT SURVEY AND INTERVIEWS
5.3.	Workshops
<u>6.</u>	ROADMAP RESULTS
6.1.	NEEDS ASSESSMENT - SECONDARY RESEARCH
6.1.	1. ACS PRESIDENTIAL ROUNDTABLE ON SUSTAINABLE MANUFACTURING REVIEW
6.2.	SURVEYS
6.2.	1. SURVEY RESULTS
6.2.	2. IN-PERSON AND TELEPHONE INTERVIEWS
6.3.	INDUSTRY WORKSHOPS
6.3.	1. INDUSTRY NEEDS ASSESSMENT WORKSHOP – DECEMBER 2014
6.3.	2. Alliances Workshop – April 2015
6.3.	3. INNOVATION WORKSHOP – JULY 201525
<u>7.</u>	<u>CHALLENGES</u>
_	
<u>8.</u>	OPPORTUNITIES
<u>9.</u>	ANALYSIS & RECOMMENDATIONS
<u>10.</u>	<u>REFERENCES</u>
<u>11.</u>	APPENDICES
11.:	1. APPENDIX A - NEEDS ASSESSMENT SURVEY
11.2	2. APPENDIX B - NEEDS ASSESSMENT INTERVIEW
11.3	3. APPENDIX C - FULL LIST OF POTENTIAL PROJECT AREAS
11.4	4. APPENDIX D - PARTICIPATING COMPANIES41

LIST OF FIGURES

FIGURE 1: EPSUM TECHNOLOGY ROADMAP PROCESS	9		
FIGURE 2: SUSTAINABLE MANUFACTURING GOALS ESTABLISHED BY THE EPSUM TEAM	15		
FIGURE 3: SURVEY OF TECHNICAL CHALLENGES IN THE CHEMICAL INDUSTRY	16		
FIGURE 4: SURVEY OF ECONOMIC IMPACTS IN THE CHEMICAL INDUSTRY	17		
FIGURE 5: RANKING OF IMPEDIMENTS TO WIDER ADOPTION OF ELECTROCHEMICAL TECHNOLOGIES	18		
FIGURE 6: COMPARISON OF TECHNICAL REACH VERSUS BREADTH OF IMPACT FOR THE 10 MAJOR INDUSTRY NEEDS IDENTIFIED	20		
FIGURE 7: COMPARISON OF POTENTIAL FOR PLATFORM TECHNOLOGY TO THE TIME HORIZON FOR DEVELOPMENT	21		
FIGURE 8: SIX POTENTIAL EPSUM CONSORTIUM INNOVATION ALLIANCES	21		
FIGURE 9: DOWN SELECTION OF POTENTIAL INNOVATION ALLIANCES	23		
FIGURE 10: DIAGRAM OF ADVANCED ELECTROWINNING PROCESS	25		
FIGURE 11: RESULTS OF LIFE SURVEYS BASED ON PARTICIPANTS THAT EXPRESSED AN INTEREST IN THE TECHNOLOGY PRESENTED. THESE			
RESULTS DO NOT INCLUDE PARTICIPANTS WHO ABSTAINED OR INDICATED NO INTEREST.	26		
FIGURE 12: HIGH PRIORITY CHALLENGES IDENTIFIED	27		
FIGURE 13: MULTI-GENERATIONAL PLAN FOR ELECTROCHEMICAL PATHWAYS INDUSTRY-UNIVERSITY RESEARCH PARTNERSHIP.			
GENERATION 1 IS BASED ON EXPERIENCE FROM THE NATIONAL SCIENCE FOUNDATION INDUSTRY-UNIVERSITY RESEARCH.	30		

LIST OF TABLES

TABLE 1: RESULTS OF EXPERT INTERVIEWS	
TABLE 2: SUMMARY OF INDUSTRY AND ELECTROCHEMICAL TECHNOLOGY	NEEDS

2. Executive Summary

There are opportunities to support, sustain, and enhance the U.S. manufacturing capacity by designing and developing processes that utilize electrochemical science and technology to address major technical barriers in the manufacturing of chemicals and materials. Electrochemical (EC) processes can be applied to the chemical manufacturing industry to gain improvements in efficiency, sustainability, and production capacity. Despite the demonstrated economic and technological advantages to applying electrochemical processes to chemical manufacturing including in advanced electronic device fabrication, component plating, metal refining and the chloralkali industry, the general chemical industry has been slow to adopt electrochemical techniques. This is mostly attributable to a lack of understanding of electrochemistry and its possible applications.

The Electrochemical Pathway for Sustainable Manufacturing (EPSuM) technology roadmap was the first of its kind to fully explore the potential applications for electrochemistry in the chemicals industry. The complexity of the chemicals industry, as well as the broad range of possible applications of electrochemistry to manufacturing, meant that the EPSuM roadmap had to develop a process that would allow for the evaluation and down-selection of an enormous amount of potential areas. The roadmap activities revealed countless opportunities where electrochemistry could be applied to the chemical manufacturing industry.

Through EPSuM's comprehensive workshops, examination of technical capabilities, and data analysis, the area of Water Treatment and Recycling emerged as the single most promising area of focus. Electrochemistry has the ability to address issues associated with the separation of aqueous process streams, and recovery of downstream chemicals and materials. Process water streams may contain constituents that could be re-used in the manufacturing facility or possess commercial value for sale to others. Removal of contaminants would allow re-use and recycling of process water without causing adverse reaction of chemical products or harm to processing equipment. Current processes are energy intensive, moderately efficient, and complex, however, chemical manufacturers are reluctant to adopt new technologies until they are proven and demonstrated.

The nature of chemical manufacturing requires that processes be reliable, continuous, and operate at volumes sufficient to accommodate the wastewater associated with process activities. All of the industry participants in the roadmap activity agreed that in order to encourage the adoption of electrochemical processes in manufacturing the fundamental research and development would first need to be de-risked in order to demonstrate practical applicability. Building demonstration capabilities will be essential in bringing these highly promising technologies to the industry, primarily owing to the issue of chemical manufacturing scalability. To date, most EC processes have operated on a relatively small scale, except for a few exceptions such as the chloralkali and aluminum processes. While evidence exists that EC processes can operate reliably at larger scales, the chemical industry will need to have demonstrations of this capability prior to significant investment.

The EPSuM roadmap resulted in a framework for the demonstration of proprietary research that will lead to platform technologies adaptable to the complex needs of the chemical manufacturing industry. The foundation of this framework is based on the concept of Innovation Alliances, industry-driven consortia designed to advance specific research and development programs. This concept was

developed as a result of important feedback from industry participants that investment in external research must have direct applicability to a specific need. Industry research and development budgets are continuing to decline and it is becoming increasingly difficult to invest in very early stage research that is not related to a specific challenge. Developing Innovation Alliances around very specific topic areas would allow industry to invest their money wisely, maintain intellectual property, and would provide a forum to solve research challenges that will be directly applicable.

The EPSuM team is proposing the development of the first-of-its-kind Innovation Alliance and demonstration facility in the area of Process Water Treatment and Recycling. The Innovation Alliance will be designed to overcome innovation challenges associated with developing effective water treatment and recycling technology and implementing this technology in-processes to ensure that the technology is robust in the chemical manufacturing environment. This unique framework will allow for the development of both platform technologies and unique solutions designed to meet industry needs, while maintaining industry intellectual property. The most important research needs have been identified as system scale, system reliability, consumable consumption, energy consumption, system selectivity, and systems costs. These broad areas of interest will be first applied to four potential areas for further research including:

- Chemical Manufacturing Plant Process Water Salts Remediation
- Hydraulic Fracturing Waste Water Treatment and Recycling
- Chemical Process Water Utilization Efficiency and Conservation (Cooling Tower Water, Condensate Recovery, Make-up Water Treatment, other processes)
- Hybrid Processes for Water Treatment and Selective Reactivity and Separations

There is strong support within the chemical manufacturing industry for a demonstration-scale facility that would have the capability to develop technology platforms that can be modified to each unique process. Industry would require access to a set of resources, including both personnel and equipment, for execution of follow-on projects to perform research and development on unique applications of electrochemical processing. This resource should have a body of expertise in process chemistry, electrochemical processing, system engineering, electrode materials, and overall system operation. A wide range of processing equipment, including reaction cells, throughput, and power levels, should also be available. It is envisioned that this facility would contain electrochemical reactors at a considerable scale (e.g., 1 m³/day) and simulation capacity to modify the processes according to industry needs.

Ohio University (OHIO) is well-recognized as an international leader in electrochemical engineering research, and is the location of CEProTECH, an NSF I/UCRC research center. Over the last 7 years, OHIO with federal, state, and industry support has invested \$10 million in advancing the field of research and commercialization of electrochemical technologies. OHIO is perfectly positioned to attract the resources needed to develop a demonstration-scale reactor facility in order to bridge the adoption gap needed for electrochemical processes to be utilized in chemical manufacturing and to cooperate with other organizations to add other areas of expertise such as design of membranes and power electronics. The roadmap process, findings, and recommendations are presented in this document.

3. Graphical Abstract



4. Background

The Chemical and Allied Products (ChEAllieds) industry represents a key component of the U.S. Gross Domestic Product (GDP), supporting 17% of the value added by the manufacturing industry in 2015.¹ Furthermore, the industry has experienced a year on year growth rate of 3 – 4% over the past three years. Overall, the gross output of the industry translated to \$830 billion dollars in 2015. However, the chemical industry is faced with major challenges that hinder and jeopardize industry growth and affect global competiveness. These include macroeconomic forces that affect supply and demand, the cost and availability of feedstock, and extremely tight profit margins. In addition, chemical processes often require large amounts of water, feedstocks (including a large proportion of petroleum products), and energy, while producing wastes that require treatment and disposal. As such, sustainable manufacturing is seen as a key component in addressing many of the major challenges within the chemical industry.

According to the American Chemical Society (ACS), next generation chemical manufacturing methods are needed to address the practical performance limits of current technologies.² Incremental improvements are not achieving the necessary advancements in performance and price required by customers. In order to develop the next generation of manufacturing methods, significant investment in Research, Development, and Demonstration (RD&D) is required. Deploying new manufacturing methods will achieve significant advantages including reducing energy and raw material usage, as well as decreasing waste and emissions. Most importantly, the implementation of sustainable manufacturing processes can have a significant impact on process efficiency, waste reduction, and co-product utilization, directly addressing the major challenges facing the industry.³ New transformational, disruptive, and enabling technologies are needed that will provide sustainable, long term solutions beyond incremental manufacturing improvements. The development of new manufacturing practices and technologies is key to the chemical industry's future success in meeting society's economic, environmental and quality of life goals in a sustainable manuer.

Addressing these RD&D challenges will require implementing technologies that have the ability to significantly impact the way that chemicals are manufactured. Electrochemical (EC) technologies and processes now represent a relatively untapped frontier of opportunity for unique, enabling, and transformative solutions that can benefit the chemicals industry. In order to examine the highest potential opportunities in applying EC technologies to chemical manufacturing, an industry-led working group was formed to develop a roadmap that could lead to the establishment of a consortium to advance this technology. The working group on Electrochemical Pathways for Sustainable Manufacturing (EPSuM), comprised of members from industry, academia, and the research community, was formed to develop a technology roadmap and to lay the pathway to collaborate on technology development projects via a consortium. This consortium would be formed to support, enhance, and sustain chemical industry manufacturing capacity by designing and developing processes utilizing electrochemical science and technology.

The EPSuM mission is to support, enhance, and sustain the US manufacturing capacity in the Chemical and Allied Products Industries by addressing major technical problems and industry growth inhibition via process design, development, and innovation using electrochemical science and technology. It is the vision of EPSuM working group that the creation of the Roadmap and subsequent Industry Consortium will enable chemical industry companies to accelerate product and process development of globally competitive and sustainable manufacturing methods.

The EPSuM team includes chemical industry leaders and scientific leaders from the electrochemical engineering field - encompassing industry, academic and association leaders, including:

- OHIO, Center for Electrochemical Processes and Technology (CEProTECH). CEProTECH is an industry-driven consortium at Ohio University, with state-of-the-art research facilities in the field of electrochemical engineering. CEProTECH is a National Science Foundation Industry University Cooperative Research Center (20,000 ft² facility with over \$10 million in infrastructure) that has created a comprehensive, consolidated hub for electrochemical research, where world-class researchers and high profile industry leaders come together and utilize the versatile discipline of electrochemical engineering to create marketable solutions for a varying array of issues.
- **PolymerOhio**. PolymerOhio is a NIST Manufacturing Extension Partnership affiliate with demonstrated leadership in roadmap development and strategic planning and led the consortium through the roadmapping process.
- **The Electrochemical Society (ECS)**. The ECS is a leading international educational association concerned with a broad range of phenomena relating to electrochemical and solid-state science and technology. ECS contributed to the roadmapping process by expanding the participation of electrochemical science and technology experts in the field.

5. Roadmap Development Process

The EPSuM roadmap process was based on identifying the intersection of industry needs and technology capability via data collection through primary and secondary research. This EPSuM roadmap was developed through: 1) Gathering input and secondary research from the chemical industry and its supply chain, 2) Identifying critical industry needs - to drive technology selection and development, 3) Determining technology alternatives that can satisfy critical manufacturing needs, 4) Selecting appropriate technology alternatives, and 5) Generating and implementing a sustainable plan to develop and deploy technology alternatives. As data was collected, a process of consolidation and downselection was used to determine areas of highest opportunity.

This Section (5.1 to 4.3) describes the process for Roadmap development. The results of the Roadmap development process can be found in Section 6 below.



Figure 1: EPSuM Technology Roadmap Process

In order to achieve the goals of the EPSuM roadmap, an **Organizational Structure** was established (Section 5.1) to aid in guiding the roadmapping process including vetting collected data and objective analysis. A **Needs Assessment** was performed via primary and secondary research (Section 5.2) and the collected data was consolidated and prioritized to determine the areas of highest opportunity. **Workshops** (Section 5.3) provided a venue for needs identification and in depth technical discussions, and an overall **Analysis** (Section 9) was performed to generate this final roadmap.

5.1. Established organizational structure: Executive Committee (EC); Advisory Board (AB)

The Executive Committee (EC) was created to oversee decisions about the program and the roadmapping process at an executive level. The EC provided critical guidance and oversight on the execution of the roadmap, particularly around decisions of consolidation and down-selection of opportunities. The EC also provided periodic progress review, and approval of all content of the program. The EPSuM Executive Committee was comprised of five (5) members: Dr. Gerardine Botte (Program and Technical Director); three industrial members: Michael T. Gallagher (Covestro), Dr. Michael Lowe (Dow Chemical), Dr. E.J. Taylor (Faraday Technology Inc.), and a technical expert (from nonprofit and/or academic) nominated by the Electrochemical Society: Dr. Paul Kohl (Georgia Institute of Technology). This provided industry leadership and expertise while balancing the governance of EPSuM. The Executive Committee also received industrial and technical input from the Advisory Board (see below).

The EPSuM Advisory Board (AB) was assembled to provide project, industrial, and technical guidance throughout the process. The AB consisted of representatives from key industrial company representatives (Appendix D). This group provided overall guidance to the project, reviewed interim progress reports, and provide strategic guidance. Industry representatives were selected to include members from all sectors of the chemicals industry as well as various company sizes.

5.2. Needs Assessment

The purpose of the Needs Assessment was to develop a thorough understanding of the trends and drivers in the chemical industry and identify the current state-of-the-art in electrochemistry to extract areas of promising opportunities. The Needs Assessment first focused on the task of narrowing down potential applications. The broad applicability of electrochemistry, combined with the highly complex nature of chemical manufacturing, resulted in countless potential applications. The EPSuM team developed this through a two-stage process of intensive research. The first stage included secondary research into the current drivers in sustainable manufacturing. The second stage expanded this to primary research through surveys developed for industry experts, and validated through industry workshops.

5.2.1. Needs Assessment Survey and Interviews

The ACS Presidential Roundtable on Sustainable Manufacturing Roadmap was the foundation for identifying industry needs. However, the roadmap was produced in 2009 and the EPSuM team needed to use primary research to: 1) ensure the focus areas identified in the ACS Roadmap were still high priority areas for the chemical industry, and 2) categorize the degree of importance of each of the six areas. A written survey (Appendix A) was developed that was designed to be a fast and simple way for industry experts to rank the importance of each of the areas identified in the ACS Roadmap. The first opportunity to obtain industry input was during the 2014 American Chemical Society National Meeting. The surveys were distributed to 14 experts. Companies surveyed included representatives from:

- Materials and ceramics
- Oil & Gas

- Pharmaceutical
- Polymers and Plastics

The results of the survey were compiled and used to develop a more comprehensive interview guide that was used in both the workshops and for more in depth primary research interviews. The interview process was designed to stimulate discussion and elicit information from individuals representing companies at multiple points in the chemicals industry supply chain. The purpose of this interview process was to develop a better understanding of potential technology drivers, development paths, system requirements, technology alternatives, timelines and other commercialization issues. This information was used to down-select topics of interest that would be explored in depth during industry workshops.

The results of the primary and secondary research were consolidated by PolymerOhio and presented to the AB. In this report, industry needs were characterized on several dimensions including: industry impact – both breadth (how many establishments could deploy) and depth (economic impact per establishment), time horizon to commercial implementation, major technical barriers, technical approaches available, and expected investment. This AB feedback was subsequently presented to the EC for review, allowing the EC to down-select the topics that would advance to the workshops for further discussion and analysis.

5.3. Workshops

The workshops were designed to identify the major technological and related barriers that inhibit the growth of advanced manufacturing in the chemical industry, organize and prioritize these barriers and propose possible solutions using appropriate electrochemical methods. Within these context, three workshops were organized to meet these goals.

Prior to the first workshop, a webinar was held on October 22nd 2014, in partnership with University-Industry Demonstration Partnership (UIDP) and Government-University-Industry Research Roundtable (GUIRR) (<u>http://sites.nationalacademies.org/PGA/guirr/PGA_080979</u>). The webinar was used to advertise the EPSuM road-mapping effort, invite interested stakeholders to the workshop and provide examples of electrochemical innovations for industrial applications.

The first workshop was held on December 16th 2014 with 29 attendees from 7 companies. The workshop was used as a brainstorming and ideation session and provided an opportunity to explore and validate the information obtained through the Needs Assessment and through the first round of surveys and interviews. Therefore, the goals of the workshop were to:

- Identify new product and critical process development needs that will impact sustainable manufacturing
- Identify unmet product and process needs that may be addressed by electrochemistry and electrochemical engineering solutions
- Characterize these needs by breadth and magnitude of impact
- Identify competing approaches

The participants were divided into breakout groups, with each group assigned to one technology gap identified through the Needs Assessment and Surveys:

- Water Management and Remediation
- Energy Efficiency and Renewable Feedstocks, and
- Carbon-based materials and Products

Each group had a facilitator and a scribe to document the process and the groups consisted of representatives from across the supply chain as well as electrochemical engineering experts. Each breakout group discussed the overall approach and provided suggestions on technology paths, challenges, hurdles, and potential opportunities for improvement. Breakout groups also began to explore potential structures for the EPSuM consortiums in the form of industrial "Innovation Alliances". This concept of Innovation Alliances was developed because of important feedback from industry participants that the investment in external research must have direct applicability to addressing a specific need. These Innovation Alliances formed the foundation for discussion for the second workshop and the results from this first workshop are described in the results section (**Section 6.3.1**).

The second workshop was held on April 16th 2015 with 15 attendees from 8 companies. The second EPSuM workshop outlined and reviewed key learnings to date and focused on further refining the concept of the Innovation Alliances and selecting Innovation Alliances for further exploration. This workshop was also used as a forum to identify the solutions needed to tackle the problems in each Innovation Alliances. The feedback generated during this session was used to define the solutions required for tackling the issues discussed in the first workshop. Key experts i.e. technology providers were subsequently recruited by the EPSuM team to give presentations on these solutions at the third workshop. These recruitment efforts included advertising in the Interface Magazine of the Electrochemical Society, a booth exhibit at the 227th ECS Meeting in Chicago (May 24th – 28th 2015), one-on-one conversation with prospective experts during the ECS conference and follow-up emails and phone calls to over 40 individuals in industry and academia with electrochemistry and electrochemical engineering expertise. The results from the second workshop are described in the results section (Section 6.3.2).

The final workshop, entitled the Innovation Workshop, was held on July 8-9, 2015 with attendees from 15 companies and 9 universities. This workshop was used as a forum for solution providers to discuss proposed technologies and for industrial attendees to vote on technologies of interest and impact in their respective arenas. This votes were recorded as feedback using the L.I.F.E. (Level of Interest and Feedback Evaluation) Forms, typically used by NSF IUCRCs to evaluate projects during their board meetings. These forms, so named, were used to gauge interest while also allowing industrial members to give individualized feedback on improvements or modifications to the proposed ideas. The results from this third workshop are described in the results section (**Section 6.3.3**).

6. Roadmap Results

The EPSuM Roadmap was the first of its kind to explore the potential for applying electrochemistry technologies to the chemical manufacturing industry. The main challenge in developing the roadmap was the broad applicability of electrochemistry and the extensive nature of the chemicals industry. The team began with hundreds of potential areas of intersection and were tasked with narrowing that down to the most promising areas of research. In order to do that, the EPSuM team worked closely with industry to coalesce and explore major themes that emerged throughout the process.

The EPSuM team emerged with interesting insight on how to structure industry-university partnerships – via the concept of Innovation Alliances. Innovation Alliances are industry-driven consortia designed to continue the research and development programs recommended in the roadmap. This concept was developed as a result of important feedback from industry participants that the investment in external research must have direct applicability to addressing a specific need. Industry RD&D budgets are continuing to decline and it is becoming increasingly difficult to invest in very early stage research that is not related to a specific application. Developing Innovation Alliances around very specific topic areas allows industry to invest their money wisely and provides a forum to solve research challenges that will be directly applicable to real-world applications.

6.1. Needs Assessment - Secondary Research

The first phase of the Needs Assessment included secondary research to identify high priority needs of the sustainable chemical industry. Prior to review, the EPSuM team defined the requirements needed for the analysis to ensure that only relevant data was selected. All data included in the Needs Assessment had to meet the following requirements:

- Included in the production of chemicals and allied products (identified by NAICS Code 325)
- Include manufacturers of all sizes
- Time horizon for implementation of 10 to 15 years
- Address sustainable manufacturing requirements of: energy reduction, carbon footprint reduction, material utilization efficiency

6.1.1. ACS Presidential Roundtable on Sustainable Manufacturing Review

The ACS Review identified six areas of improvement as summarized below:

Area 1: Alternative Feedstocks

This area requires decreased dependence on fossil-based feedstock, development of infrastructure and techniques for alternative feedstock acquisition, chemical pathways and processing combined with investments in research, development and demonstration (RD&D) efforts and increase in workforce development for realistic sustainability.

Area 2: Energy Efficiency

This area requires improvements to existing equipment by incorporating materials and systems that maximize current operational economics e.g. leveraging low-quality heat emissions through the use of sensors, process control and software. In addition, risk perception and economic viability would aid in

widespread integration of practical energy efficient technologies and practices for optimal energy and material flows.

Area 3: Materials for Sustainable Manufacturing

This area requires overcoming the physical limits of existing materials through investments in RD&D. The CheAllieds largely reap these benefits as they supply both raw and finished materials and can leverage the benefits in the energy consumption of their processes.

Area 4: Next Generation Chemical Manufacturing

This area requires overcoming the performance limits of existing established technologies e.g. by building on recent innovations of microelectronics, computational science, genetic and biological engineering etc.

Area 5: Waste Reduction and Recovery

This area requires moving towards more integrated and closed-loop processes as already demonstrated by efforts in post-consumer paper, plastics and oils. These advancements require technical expertise due to the complexity of recovery in different material states, i.e. solid, liquid or gas.

Area 6: Water Conservation, Recycling and Reuse

This area addresses the CheAllieds consumption (in 2009 when the roundtable was conducted) of 10 billion gallons per day in heating, cooling, and processes used by moving towards zero discharge systems, e.g. minimizing water inputs and leveraging low-quality heat, while improving recycle/reuse technologies.

Overall, ACS estimated a total investment of \$3.2 billion over 5 years to address these challenges. With that level of investment, the benefits are estimated to be:

- 65% reduction in fossil fuel use
- 34% usage of renewable fuel sources
- 63% reduction in GHG emissions
- Savings equivalent to 500,000 new jobs
- Increase in production capacity

6.1.1.1. ACS Roadmap Analysis Results

The ACS Roadmap was an important foundation for the EPSuM roadmap in identifying the most important opportunities in sustainable chemical manufacturing. The 6 areas identified in the Roadmap focused on the highest potential impact. Through the EPSuM analysis, it was determined that the development of electrochemical pathways has the potential to advance sustainable manufacturing goals through new processes for the next generation of existing chemical products, innovative products, and novel process systems for novel new products. The EPSuM team developed a set of goals (Figure 2) that could potentially be achieved through the use of electrochemical strategies. Since the broad application of electrochemistry in chemical manufacturing was essentially a nascent concept, the EPSuM identified

a series of metrics that seemed reasonable and impactful. The reasonableness of these targets was tested throughout the course of the workshops.



Figure 2: Sustainable Manufacturing Goals established by the EPSuM team

6.2. Surveys

The main goal of the primary and secondary research activities was to identify key drivers in the CheAllieds industry and areas of potential technical capability where electrochemistry might be able to address these needs. While the research provided the basis for making initial assumptions, the EPSuM team needed to validate the information from this research to continue to formulate a formal Needs Assessment. The first step in this process was to develop a survey to validate and inform the information found in the ACS Roadmap.

The ACS meeting in August of 2014 was an ideal venue for distribution of the survey to multiple industry leaders. The EPSuM team contacted over 14 experts prior to the meeting to arrange a time to perform the survey. The survey was designed to be simple, effective, and take less than 15 minutes to complete. The questions were as follows and the results are provided in Figures X:

- 1. Rank 1 -6 which of these problems poses the greatest TECHNICAL CHALLENGE to the Chemicals Industry:
 - a. Development of alternative feedstocks (to petroleum and natural gas)
 - b. Improving energy efficiency
 - c. Development of new materials for sustainable manufacturing
 - d. Waste reduction and recovery in chemicals production
 - e. Water conservation, recycling and re-use in the chemicals industry
 - f. Development of next generation chemical manufacturing processes
- 2. Rank 1 -6 which of these problems will have the greatest ECONOMIC IMPACT on the Chemicals Industry:

- a. Development of alternative feedstocks (to petroleum and natural gas)
- b. Improving energy efficiency
- c. Development of new materials for sustainable manufacturing
- d. Waste reduction and recovery in chemicals production
- e. Water conservation, recycling and re-use in the chemicals industry
- f. Development of next generation chemical manufacturing processes
- 3. Rank 1 -3 which areas you see as the greatest impediment to wider use of electrochemistry methods and technology in the Chemicals Industry:
 - a. Lack of electrochemistry education and knowledge
 - b. Lack of resources and institutions for research and development
 - c. Lack of resources for cell construction

6.2.1. Survey Results

Question 1 - Rank 1 - 6 which of these problems poses the greatest TECHNICAL CHALLENGE to the Chemicals Industry.

The results of the survey indicated that the most important technical challenge in the industry was water conservation, recycling, and re-use (1e), followed closely by development of new materials for sustainable manufacturing (1c). There was better consensus about water conversation due to the lower standard deviation in comparison to the development of new materials. Waste reduction and recovery as well as development of next generation chemical manufacturing processes were also both regarded as highly important.

- 1. Rank 1 6 which of these problems poses the greatest TECHNICAL CHALLENGE to the Chemicals Industry:
 - a. Development of alternative feedstocks (to petroleum and natural gas)
 - b. Improving energy efficiency
 - c. Development of new materials for sustainable manufacturing
 - d. Waste reduction and recovery in chemicals production
 - e. Water conservation, recycling and re-use in the chemicals industry
 - f. Development of next generation chemical manufacturing processes



Figure 3: Survey of Technical Challenges in the Chemical Industry

Interestingly, the results of the survey were in direct opposition to the ACS Roadmap in identifying the highest priority needs. The ACS Roadmap was developed during a period of high energy costs, and high petroleum prices, and therefore, was identified as a much higher priority for the industry. In 2014 during the course of these surveys, the cost of petroleum and natural gas had dropped significantly and energy costs were down as a result.

Question 2 - Rank 1 - 6 which of these problems will have the greatest ECONOMIC IMPACT on the Chemicals Industry

The survey indicated that the most impactful areas would be the development of new materials for sustainable manufacturing (2c) and the development of next generation chemical manufacturing processes (2f). On the other hand, energy efficiency improvement (2b) and waste reduction and recovery (2d) were ranked lowest.

- 2. Rank 1 6 which of these problems will have the greatest ECONOMIC IMPACT on the Chemicals Industry:
 - a. Development of alternative feedstocks (to petroleum and natural gas)
 - b. Improving energy efficiency
 - c. Development of new materials for sustainable manufacturing
 - d. Waste reduction and recovery in chemicals production
 - e. Water conservation, recycling and re-use in the chemicals industry
 - f. Development of next generation chemical manufacturing processes



Figure 4: Survey of Economic Impacts in the Chemical Industry

These responses appear to indicate the importance of material and process innovation as better drivers of economics in the chemical industry over efficiency. It also agrees with the aforementioned analyses, i.e. cheaper energy availability reduces the economic impact of energy efficiency.

Question 3 – Rank 1 -3 which areas you see as the greatest impediment to wider use of electrochemistry methods and technology in the Chemicals Industry:

The general consensus suggests that lack of resources for cell construction is the greatest impediment, while the lack of electrochemistry education and knowledge was identified as the lowest impediment to the use of electrochemistry in the chemicals industry.



- a. Lack of electrochemistry education and knowledge
- b. Lack of resources and institutions for research and development
- c. Lack of resources for cell construction



Figure 5: Ranking of Impediments to wider adoption of Electrochemical technologies

6.2.2. In-Person and Telephone Interviews

In addition to the brief surveys conducted at the ACS meeting, the EPSuM team conducted more in depth interviews with 16 additional experts at 15 companies. These interviews (see Appendix B) were designed to identify specific areas of technical interest for the Needs Assessment that could be explored further during the workshops. The interviews focused on three major categories: 1) specific areas of potential application of electrochemistry, 2) areas of industrial operation where electrochemistry could be applied, and 3) highest impact areas of application. The tables below summarize the findings of the interviews.

Table 1: Results of Expert Interviews

Specific Areas of Potential Electrochemistry Application
Recycling of chemical process by-products and petroleum waste products
Specialty additives for formulated polymers products
Electrosynthesis of nanostructured additives for coatings, adhesives, sealants, and film products
Next generation barrier films and encapsulants for solar, battery and super capacitor applications
Processing of coal to produce graphene
Electrosynthesis of monomers from natural gas and natural gas stream by-products
Electrochemical pathway to monomers and functional additives from coal and natural gas streams
Electrochemical pathway to polyols from coal and natural gas streams

Specific Areas of Potential Electrochemistry Application

Bio-Electrochemical Engineering approaches to multi-stage polymerization processes

Electrosynthesis of lubricant additives and motor oils from biobased feedstocks

Industrial Operation Areas

Synthesis of bulk chemicals

Synthesis or modification of specialty chemicals (including pharmaceuticals)

Corrosion and/or environmental protection

Environmental remediation (including air and water treatment)

Energy generation

Areas of Highest Potential Impact

Synthesis of chemicals from renewable feedstocks

Separation of aqueous waste streams

Recovery of downstream chemicals/materials

Modification of hydrocarbons

The EPSuM team used the results of these interviews to aid in the ideation and brainstorming during the first workshop.

6.3. Industry Workshops

Three workshops were held:

- 1. An Industry Needs Assessment workshop serving as a brainstorming session (Section 6.3.1)
- 2. An Alliance workshop serving as a consortium structuring session (Section 6.3.2)
- 3. An Innovation workshop serving as a solutions assessment session (Section 6.3.3)

6.3.1. Industry Needs Assessment Workshop – December 2014

The ideation session led to the identification of 100+ industry needs and numerous enabling electrochemical engineering solutions. These needs were grouped into 11 categories of industrial needs and 5 categories of enabling electrochemical technology needs.

Table 2: Summary of Industry and Electrochemical Technology Needs

Ind	ustrial Needs	Ena	abling Technology Needs
1.	Separations in chemical manufacturing - materials from	1.	Novel anion exchange membranes
	aqueous streams	2.	Novel electrodes
2.	Separations in non-chemical manufacturing - materials from	3.	Electrochemical cell longevity
	aqueous streams	4.	Electrowinning/electrogalvanizing
3.	Separations for reduced energy requirements		cost reduction
4.	Selective organic reduction	5.	Next-generation reactor design
5.	Polymer depolymerization		
6.	Water electrolysis		
7.	Electrochemical materials synthesis from renewable		
	feedstocks		
8.	Spent/unused chemicals recovery		
9.	Carbon nanotubes separation		
10.	Modification of hydrocarbons modification		
11.	Other opportunities		

Following the Industry Workshop, the EPSuM team evaluated each of the 10 identified Industrial Needs (Item number 11 "Other opportunities" was excluded from this analysis) to determine the potential impact (breadth and magnitude), technical reach, time horizon to market readiness, potential as platform technology, potential as pre-competitive technology, potential as "game-changer". The results of this analysis are found below.



Figure 6: Comparison of Technical Reach versus Breadth of Impact for the 10 Major Industry Needs Identified



Figure 7: Comparison of Potential for Platform Technology to the Time Horizon for Development

Based on the findings from this analysis, the EPSuM team was able to formulate a framework for six potential Innovation Alliances for further exploration in the next workshop (Figure 8).



Figure 8: Six Potential EPSuM Consortium Innovation Alliances

A summary of each innovation alliance is as follows:

- Water Treatment (Including Oil/Gas/Hydraulic Fracturing): Under this innovation alliance, opportunities would include reducing the water usage footprint in the chemical industry via recycling of wastewater streams e.g. recycling process chemical additives and improved treatment of process water streams e.g. electrochemical processing of "frack water" (flowback and produced water) at lower cost, higher energy efficiency and higher selectivity. Per the ACS sustainable manufacturing review, this applies to Areas 2, 5 and 6.
- **Bio-Renewables Electrochemical Process and Product**: Under this innovation alliance, opportunities would explore the electrosynthesis of specialty chemicals, monomers, multifunctional additives and polymers from renewable oils and carbohydrates. Per the ACS sustainable manufacturing review, this applies to Areas 1, 3 and 4.
- Solar PV Module Recycling: Under this innovation alliance, opportunities would explore the recycling of thin-film photovoltaic modules containing cadmium in the form of CdTe and other compounds like CIGS, as well as electrochemical extraction processes that are cost-effective and applicable to multiple material systems in solar cells. Per the ACS sustainable manufacturing review, this applies to Area 5.
- **Transformational Electrowinning**: Under this innovation alliance, opportunities would explore chemical stream separations (recycling, recovery and remediation) via electrochemical methods outside of the needs in the solar industry e.g. electronics recycling and paint pigment remediation. Per the ACS sustainable manufacturing review, this applies to Areas 2, 5 and 6.
- **Coal to High Performance Materials, Additives/Fillers**: Under this Innovation Alliance, opportunities would explore the conversion of coal to carbon-based materials. It was advised that this innovation alliance should be considered an enabling area instead of a major thrust, due to pre-existing carbon-based efforts. Per the ACS sustainable manufacturing review, this applies to Areas 1 and 3.
- Electrochemical Enabling Technologies: Under this innovation alliance, emphasis would be placed on enhancing electrochemical processing technologies (See Section 3.1.1.1) i.e. equipment, materials etc., that aid to reduce costs and improve efficiencies. In other words, this would focus on a technology push to enable industry needs. Per the ACS sustainable manufacturing review, this applies to Areas 2, 3 and 4.

In addition, a list of 120 needs used in structuring these innovation alliances are provided in Appendix C.

6.3.2. Alliances Workshop – April 2015

Based on the interests and expertise of invited participants to the second workshop, four Innovation Alliances were highlighted and discussed in detail, with two of these innovation alliances -Transformational Electrowinning and Water Treatment – suggested as a combined platform technology.



Figure 9: Down Selection of Potential Innovation Alliances

6.3.2.1. Biorenewables Innovation Alliance

In these discussions, critical issues related to technology development were highlighted as follows:

- Controversy due to nexus between Food-Fuel-Chemicals systems
- Cost-competitiveness of renewable building blocks due to shale gas supply economics
- Complications associated with capital investment due to competing feedstock innovation strategies
- Technical Expertise required for R&D in selectivity, catalysis and membrane materials
- Gaseous carbon-containing feedstocks leading to downstream carbon dioxide formation
- Retrofitting strategies with existing chemical company infrastructure
- Complexity associating with scalable processes

In addition to these highlighted needs, metrics for success and outcomes were discussed along the lines of:

- Reduction in Carbon Footprint
- Reduction in Production Cost
- Value-added chemicals development to meet specific company needs
- Electrode, reaction and process design for multiple products
- Integration of established bio-based building blocks for innovation.

Overall, industry realized the need for a carefully structure and "beta-tested" alliance to manage the risk of the "front-end of innovation" as well as "new product development" processes for bio-renewable products. As such, implementation of electrosynthesis and electrochemical engineering unit operations could offer new pathways and be examined under this alliance.

6.3.2.2. Solar Module Recycling

In these discussions, critical issues related to technology development were highlighted as follows:

- Next Generation process for removal of EVA polymer encapsulant
- R&D for translation from batch to continuous reactors
- Cost reduction in recovery processes
- Improved metal recovery i.e. energy efficiency and process selectivity.

In addition to these highlighted needs, metrics for success and outcomes were discussed including:

- Develop cost reduction metrics
- Evaluation of government (local, state and federal) regulations and environmental health and safety (EH&S) metrics
- Leveraging established systems e.g. First Solar's Unrefined Semiconductor Material Processor, for metrics
- Integrated process with cost and energy efficient recovery of polymer, glass and metal recovery

6.3.2.3. Water Treatment Technologies Innovation Alliance

In these discussions, critical issues related to technology development were highlighted as follows:

- Treatment of Cooling Tower Water
- Water Shortage Issues (Process Innovations for Reduction of Volume of Water)
- Quality of water issues (for production use and post-production)
- Evaporative water loss during production process
- Electrolyze water to produce ozone
- Water treatment cell design electrodes, reactors and equipment

Based on these ideas, example projects were discussed as platform technologies

- Chemical Manufacturing Plant Process Water Salts Remediation
- Hydraulic Fracturing Waste Water Treatment and Recycling
- Chemical Process Water Utilization Efficiency and Conservation (Cooling Tower Water, Condensate Recovery, Make-up Water Treatment, other processes)
- Hybrid Processes for Water Treatment and Selective Reactivity and Separations

Subsequently and to further integrate water technologies with other waste streams, Ohio University proposed a potential platform technology for process stream treatment, combining water remediation with metal recovery.

6.3.2.4. Transformational Water and Electrowinning Innovation Alliance

A platform technology leveraging process streams with relatively low concentrations could incorporate ion reduction with waste oxidation and vice versa. This reductant stream could be waste metals, waste solar modules or more generally electronic waster (e-waste) as shown in Figure 10.



Advance/Transformational Electrowinning

Figure 10: Diagram of Advanced Electrowinning Process

Due to the need for water recycling with proprietary compositions, this platform technology gained enthusiastic responses from the attendees and was proposed as a technology for further discussion in the final workshop.

Finally, to address the critical needs and opportunities for the innovation alliances, technology providers were sought with expertise in the following areas of electrochemistry and electrochemical engineering:

- 1. **Membranes** e.g. electrochemical separations using membranes, next generation ion exchange membranes etc.
- 2. **Organic electrochemistry** e.g. mechanistic organic electrochemistry, novel solvent systems, electropolymerization etc.
- 3. **Electrochemical Reactor Systems** e.g. AC/DC power supplies, electrochemical control modes, advanced electrochemical reactor configurations etc.
- 4. **Novel Electrodes Processes and Architecture** e.g. adsorbate selective electrodes, advanced manufacturing of electrodes, well-ordered systems etc.

6.3.3. Innovation Workshop – July 2015

A total of 11 technology solutions were presented and discussed during this workshop. The titles (verbatim) of the presentations are listed below with the results of the feedback provided in Figure 11.

- 1. Transformational Water/Selective Electrowinning
- 2. Electrosynthesis of Hydrocarbon Base Oil from Bio-Based Feedstock
- 3. Highly Active and Scalable Porous Electrodes for Chemical Conversion
- 4. Development of Slurry Electrodes for Industrial Processes

- 5. Ceramic Membrane Reactor for the conversion of Free Fatty Acids to Hydrocarbons
- 6. Ion Conductive Membranes: Current Applications and Future Prospects
- 7. Bio-inspired and Biological Electrocatalysis

Very Interested

- 8. Development of a new Reaction Medium for Organic Electrochemistry
- 9. Novel Electrocatalysts for CO2 and Oxygen Reduction and Oxygen Evolution
- 10. Organic Electrochemistry: Using Sustainable Sources of Energy for the Generation of New Reactive Intermediates



11. Powerful Connections: Managing Volts and Amps in the Sustainable Electrochemical Grid



Interested with Change

Interested

These results indicate the strongest interest in a solution for simultaneous water and waste management (Technology Solution 1 in Figure 11), followed closely by interests in both innovative catalyst structures (Technology Solution 3 in Figure 11) and organic conversion and separation (Technology Solution 5 in Figure 11). Thus, an appropriate Innovation Alliance would be the Transformational Water and Electrowinning Innovation Alliance (**Section 6.3.2.4**) discussed in the Alliance Workshop.

7. Challenges

The key drivers for electrochemical technology implementation center around four considerations: (1) technology awareness, (2) market needs for innovative processes, (3) competitive balance between electrochemical and chemical processes, and (4) scale up and cost. More specifically, technical challenges related to Process Solutions and/or Materials will need to be addressed. These challenges were identified as high priority areas that would need to be addressed for wide-scale adoption (Figure 12).

Concerns around scale up and cost seemed to be one of the major limitations for the chemical industry to accept the potential transformational solutions that electrochemical technologies could enable for the industry. More details are provided in Section 7.



Figure 12: High Priority Challenges Identified

8. **Opportunities**

Overall, there were several major research needs identified as opportunities to electrochemistry so it can be widely applied to chemical manufacturing. These include:

System Scale – Most developmental electrochemical systems in operation to date have operated at the 100 mW power level. For practical use in the chemicals industry, systems must be demonstrated at the 1 kW range, an increase of 4 orders of magnitude. At these power levels, the systems must be shown to process material on the order of several hundred gallons per minute.

System Reliability – To be used in chemicals production, systems must be capable of running 24 hours per day for weeks or months at a time without interruption. Few electrochemical processes have demonstrated this level of continuous operation to satisfy industry needs. These tests would need to include all system elements including power supplies, controls, electrodes, etc.

Consumables – The true level of consumable consumption, and resultant cost, is unknown at the scale and operating levels required by most chemical manufacturing operations. Electrode consumption, degradation, replacement requirements and other operating parameters must be established.

Energy Consumption – True energy consumption at scale including required system uptime and reliability levels must be assessed.

Selectivity – To recover valuable wastewater constituents, electrochemical systems must demonstrate suitable selectivity in contaminant removal that will warrant recovery over fresh material supply.

Capital Costs – Once demonstrated at scale, capacity and suitable reliability levels, system capital costs must be developed.

In addition to the information outlined above, chemical companies in the EPSuM program indicated they would require access to a set of resources, including both personnel and equipment, for execution of follow-on projects. This access would facilitate research and development on unique applications of electrochemical processing. This resource should have a body of expertise in process chemistry, electrochemical processing, system engineering, electrode materials, and overall system operation. A wide range of processing equipment, including reaction cells, throughput, and power levels, should also be available.

9. Analysis & Recommendations

The Chemical and Allied Products Industries, similar to other mature industries, are challenged by the ever-increasing pace of economic changes - both local and global. These changing dynamics impact how the chemicals industry produces its products, pricing levels the market will support, the attractiveness of new products, and overall profitability. When this project was first conceived, much of the initial challenges and needs assessment was based on the Vision 2020 document from the American Chemical Society released in 2009. At the time, oil and energy prices were high and there was significant pressure to reduce energy consumption and identify alternative feedstocks for producing chemicals. Within 5 years, due to market dynamics in the oil and gas industry, the perspective on availability and long term

pricing of hydrocarbon fuels has changed dramatically. As a result, interest in energy efficiency and alternative feedstocks has greatly reduced.

The investment strategies of companies in this industry have also begun to change. The increased pressure to deliver results with every investment dollar, including R&D funds, has forced companies to critically scrutinize every investment. Companies demand knowledge of expected results in their R&D expenditures and a clear assessment of the risks involved. As such, companies are less likely to join consortia or collaborations with a portfolio of prospective projects, some of which may be of little or no interest to the company. If investments in consortia are made, they will be a small portion of the company's overall R&D budget (e.g. <2%) with an expectation that results will be produced on a regular, recurring basis quarterly or semi-annually. If a technology area appears to have promise and fits within the company's overall strategy, more sizable investments (say 5%-20%) are possible. However, these are performed on a contract research basis, with the company controlling all IP and release of information.

There would have been considerable difficulties if the EPSuM team attempted to develop a traditional consortium to develop electrochemical technologies specifically for the chemical industry. Electrochemical processes are relatively unknown to the industry and the benefit would have been unclear. This prompted the concept of Innovation Alliances that are focused on demonstration efforts with emphasis on a single platform technology with broad applicability to the whole industry. Industry participants in the roadmap activity repeatedly stressed the importance of needing demonstration level technologies prior to significant funding or time investments.

To demonstrate this new Innovation Alliance, the team has selected water conservation, recycling and re-use as the first focus. As discovered in this roadmap, water recycling is extremely important and proprietary in the chemical industry. Demonstration scale reactors would focus on effluent water from industrial process such as metal and gas retrieval. This effluent water may contain constituents that could be re-used in the manufacturing facility or re-sold to external entities for commercial value. Removal of process contaminants would allow re-use and recycling of process water without causing disruption in production e.g. through adverse product selectivity or downtime to processing equipment. The use of electrochemical technology and systems have shown some technical promise in this area; however, there are a number of issues that require further development and demonstration before the private industry will be willing to make sizable investments in technology development and adaptation for company and process-specific application.

The estimated investment required can be considered moderate as the consortium will be selfsustaining within 4 years via memberships. Furthermore, the consortium will capitalize on the infrastructure established by Ohio University and the Center for Electrochemical Processes and Technology (20,000 ft² facility with over \$10 million in state-of-the art equipment) as well as coinvestment of the University, state and federal agencies. The EPSuM team envisions the overall investment would be approximately: Year 1: \$1.5 million, Year 2: \$575 thousand; Year 3: \$375 thousand; Year 4: \$185 thousand. There would be at least 10 companies participating by the end of Year 4 that would pay an annual membership of \$100,000 per year.

Figure 13 shows the differences between the two models. Generation 1 is based on OHIO's Center for Electrochemical Processes and Technologies that was established following the Industry/University

Cooperative Research Agreements requested by the National Science Foundation (NSF). Generation 2 is the evolution to a stronger industry/university partnership via Innovation Alliances that enable access to complete technology with the platform focused on: 1. Design, scale-up, optimization and demonstration of electrochemical reactors; 2. Process control and simulation; 3. Methods for *in-operando* trouble-shooting; and 4. Access to specialized electrochemical equipment and expertise. In addition, Generation 2 would be strengthened by complementary expertise of researchers and engineers from different academic institutions, on a case-by-case basis, to address the needs of the industry. For example, when external investigators develop novel catalyst materials and membranes, the EPSuM leadership would work with these investigators to develop methods for scale-up and demonstration in the pilot-scale facility. OHIO is perfectly positioned to attract the resources needed to develop a demonstration-scale reactor facility and cooperate with other organizations with complementary expertise such as design of membranes and power electronics. This OHIO capability has been demonstrated via the Innovation Workshop and will be imperative to bridging the adoption gap needed for electrochemical processes to be utilized in chemical manufacturing.

Partnership Type	Generation 1: Industry- University Cooperative Research Center	Generation 2: Innovation Alliance
Technology Scale	Bench-scale (mW)	Pilot-scale (kW)
Technology Capacity	mL/min	m³/day
Technology Readiness Level	1-3	4-6
Technology Platform	Shared Electrochemistry	Shared reactor models, expertise, characterization and <i>in situ</i> cells
Intellectual Property	Pre-competitve	Proprietary
Membership Level	\$50,000 per year	\$100,000 per year

Figure 13: Multi-generational plan for Electrochemical Pathways Industry-University Research Partnership. Generation 1 is based on experience from the National Science Foundation Industry-University Research.

Based on the sustainability metrics adopted by the EPSUM for 2030 (shown in Figure 2), project selection for the innovation alliance on water conservation, recycling and re-use should be based on the potential impact towards the following metrics:

- 50% waste and by products reduction
- 40% carbon dioxide emissions and effluent reduction

- 30-50% reduction in manufacturing costs
- 40-50% energy utilization reduction

10.References

- 1. U.S. Bureau of Economic Analysis (2016). Industry Data: GDP by Industry (<u>http://bit.ly/BEA-IndustryGDP</u>, Last Accessed March 2017).
- 2. American Chemical Society (2013). Sustainable U.S. Manufacturing: Chemical and Allied Industries; Technology Area 4: Next Generation Chemical Manufacturing (<u>http://bit.ly/ACS-Sustainability-Area4</u>, Last Accessed March 2017).
- 3. American Chemical Society (2013). Sustainability and the Chemical Enterprise (<u>http://bit.ly/ACS-Sustainability-Chem-Enterprise</u>, Last Accessed March 2017).

11. Appendices

11.1. Appendix A - Needs Assessment Survey

Use Of Electrochemistry In The Chemicals Industry: Needs Assessment

- Rank 1 6 which of these problems poses the greatest TECHNICAL CHALLENGE to the Chemicals Industry: Development of alternative feedstocks (to petroleum and natural gas) Improving energy efficiency Development of new materials for sustainable manufacturing Waste reduction and recovery in chemicals production Water conservation, recycling and re-use in the chemicals industry Development of next generation chemical manufacturing processes
- Rank 1 6 which of these problems will have the greatest ECONOMIC IMPACT on the Chemicals Industry:

 Development of alternative feedstocks (to petroleum and natural gas)
 Improving energy efficiency
 Development of new materials for sustainable manufacturing
 Waste reduction and recovery in chemicals production
 Water conservation, recycling and re-use in the chemicals industry
 Development of next generation chemical manufacturing processes
- Rank 1 3 which areas you see as the greatest impediment to wider use of electrochemistry methods and technology in the Chemicals Industry:

 Lack of electrochemistry education and knowledge
 Lack of resources and institutions for research and development
 Lack of resources for cell construction

Others

- 4. How would electrochemistry methods and techniques be investigated, developed and evaluated for use within your company? Which organization, department, division would have that responsibility?
- 5. Would you like to participate in a NIST project to evaluate opportunities of electrochemistry in sustainable manufacturing for the Chemical Industry and Allied Industries?

Yes _____

No ____

11.2. Appendix B - Needs Assessment Interview







As a participant in the NIST EPSuM Technology Roadmap, answers to the following questions will enable us to develop a "Preliminary Needs Assessment and Gap Analysis" in preparation for the Technology Roadmap Workshops:

- 1. Which of the following opportunities pose the greatest <u>technical challenges</u> to the Chemicals and Energy Industry:
 - a. Development of alternative feedstock (replacing petroleum and natural gas feedstock)
 - b. Improving energy efficiency
 - c. Development of new materials for sustainable manufacturing
 - d. Waste reduction and recovery in intermediates, chemicals, polymers and formulated products, manufacturing
 - e. Water conservation, recycling and re-use in the chemicals and energy industry
 - f. Development of next generation chemical manufacturing processes?
- 2. Follow-up to question 1, what technologies and technology platforms do you see as having the greatest potential to address these opportunities and issues?
- 3. Which of these opportunities and issues will have the greatest <u>economic impact</u> on the Chemicals Industry and why:
 - a. Development of alternative feedstock (replacing petroleum and natural gas feedstock)
 - b. Improving energy efficiency
 - c. Development of new materials for sustainable manufacturing
 - d. Waste reduction and recovery in chemicals production
 - e. Water conservation, recycling and re-use in the chemicals industry
 - f. Development of next generation chemical manufacturing processes?
- 4. What do you see as the greatest impediment to wider use of electrochemistry methods and technology in the Chemicals Industry?
 - a. Lack of electrochemistry education and knowledge
 - b. Lack of resources and institutions for research and development
 - c. Lack of resources for cell construction
 - d. Lack of technology platform integration
 - e. Something else?

- 5. How would electrochemistry methods and techniques be investigated, developed and evaluated for use within your company? Which organization, department, division would have that responsibility?
- 6. Do you see a role for use of electrochemistry as a conversion or refining process for utilization of biomass feedstock? What are its limitations or unknowns?
- 7. Do you see opportunities for wider use of electrochemistry to offset or replace the significant energy requirements (thermal) in separation processes Replacing fossil fuels (natural gas and oil) with renewable (wind and solar produced electricity) energy?
- 8. Describe how electrolysis technology for hydrogen production could be improved?
- 9. Do you see a role for electrochemical engineering in the production of nanostructured materials and formulated products? Which nanomaterials have the greatest potential to impact the chemicals industry and your company, and how would they be used?
- 10. What are the limitations to wider use of electrochemical science to enable intensification of some chemical manufacturing processes enabling the economic use of smaller, lower capacity plants?
- 11. What opportunities are available to use electrochemical technology in separation processes for treatment of aqueous solutions, by-products, and waste materials? Are there opportunities in your company for application of electrochemical processes to the recycle/reuse of process water?
- 12. What is the potential for use of electrochemistry and by way of example, ionic liquid processing to improve separation processes in the chemicals industry and your company?
- 13. What are the barriers for reducing chemical synthesis process costs?
- 14. What are the barriers to reducing the amount of energy required for processing or production?
- 15. What are the trends for electrochemical milling methods to fabricate nanoparticles and nano-fibers?

11.3. Appendix C - Full list of potential project areas

- 1) Separation (electrowinning) of organic and inorganic materials from wastewaters and/or by-product liquids in chemical manufacturing for material recovery, re-cycle, or use by others.
 - a) Treatment of wastewater in closed loop chemical plants
 - b) Carbon-based membrane for filtration; electrochemical
- 2) Separations (electrowinning) of organic and inorganic materials from wastewaters and/or byproduct liquids in non-chemical manufacturing operations for purpose of resource recovery and feedstock replacement.
 - a) Process water recycling for metal finishing & printed circuit board
 - Water re-use from advanced manufacturing Electrochemical Machining (ECM) to Sludge Volume ratio of 300:1
 - c) Industrial wastewater treatment to reduce chemical consumption
 - d) Na₂SO₄ Recovery
 - e) Removal of metal from ECM waste
 - f) Waste stream management (recycle)
 - g) Electrowinning metals (2nd largest energy consumption)
 - h) Electrowinning of CdTe and other solar materials AK
 - i) First Solar presentation at April workshop
 - Re business model initially, each PV OEM will need to develop their own process to recycle panels and recover materials. Process will be chemistry specific. Possibly in future, third party "PV panel processing" industry could develop, similar to metal scrap.
 - i) ITO recovery
 - j) NiCd battery recovery/reversal
 - k) Metal recovery of electronics
 - I) Electrochemical process for remediating organics in class process and other water/fluid
 - m) Waste water treatment for metal recovery
 - n) Recovery of metals from sewage sludge
 - o) Electrowinning process for current formaldehyde free binders
 - p) Waste asphalt into graphite
 - q) Electrowinning process for recovery of chemicals and coating materials in glass recycling
- 3) Separation of chemical streams to reduce resource requirement
 - a) Separation of chemical streams to reduce/replace thermal (steam)
 - b) Design process to operate at lower temperatures to reduce water requirements
 - c) Reduce energy in separation processes (gases)
 - d) Electrochemical processes to replace chemical processes
- 4) Selective organic reduction
 - a) Portable water treatment systems to address intermittent biological problems
 - b) Electrochemical pathway to CO₂ thickening (for enhanced oil recovery)
 - c) Selective sacrificial reduction
 - d) Electrochemical CO₂ reduction (reduced CO₂ air emissions)
 - e) Polymer depolymerization

- f) Valuable pigments (TiO₂, etc) polymer depolymerization
- 5) Water electrolysis
 - a) Water electrolysis from nuclear heat
 - b) Efficient water electrolysis
- 6) Electrochemical synthesis of materials from bio-products and incumbent feedstocks for use in end products
 - a) Electrochemical synthesis of chemicals, oligomers and polymers from multi-functional feedstocks (bio-materials)
 - b) Synthesis of polymers in-situ w/ reinforcements for composite manufacture
 - c) Monomers to polyamides, polyimide, polyurethanes, PC, polyesters
 - d) Ester based plasticizer replacement for phthalate based plasticizers
 - e) CO_2 to C_x to develop new monomers and polymers
 - f) Electrochemical alternative to fermentation/bio-based feed stock
 - g) C₂ and C₃ to electrolytes and ionic liquids
 - h) Sealants/adhesives via electrochemistry processes
 - i) Electrochemical synthesis of aldehyde free binders for glass
 - j) Electrochemical alignment of fillers for transparent (flexible) composites
 - k) Electrosynthesis of polymers for ease of integration with additives
 - I) Integration of CO₂ capture and synthesis to higher order chemicals
 - m) Low cost or optimized precursor for CNT CO₂ capture
 - n) Layering carbon nanotubes (CNTs) with metal oxide/metal nanoparticles
 - o) Reversible or controlled electrochemical attachment of gas molecules to carbon
 - p) Cheap process for single-walled nanotube material
 - q) Low process cost to produce/isolate single layer graphene
 - r) End products include slow release coatings on fertilizer, pharmaceuticals, optically transparent carbon films on glass
- 7) Recovery of spent/unused chemicals by downstream users or consumers
 - a) Recovery of chemicals used in hydraulic fracturing: flowback and process water
 - b) Separation of antifreeze (glycol)
 - c) "Point-of-use" turnkey electrochemical process for post-consumer paint recycle vs. large plants
 - d) Frack water process next generation
- 8) Separation of carbon nanotubes
 - a) Efficient catalyst for carbon nanotube syntheses
 - b) Electrochemical process to segregate carbon nanotubes
- 9) Modification of hydrocarbons
 - a) Electrochemical modification of C–H hydrocarbons
- 10) Other opportunities
 - e) Plasma pyrolysis
 - f) Electrochemical planarization of Si
 - g) Point of generation methane reforming as new feedstock source
 - h) Molten salt electrolysis as feedstock source

Needs of the Chemicals and Allied Products Industries

Notes from interviews and completed surveys incorporated into previously identified categories

- 1) Separation (electrowinning) of organic and inorganic materials from wastewaters and/or by-product liquids in chemical manufacturing for material recovery, re-cycle, or use by others.
 - a) Waste reduction recovery is always important, but typically realizes diminishing returns to further investment as you reduce waste. Much interest in this area is regulatory driven – will do it when everyone has to, as it's difficult to recover the added processing cost if done in isolation – would become non-competitive.
 - b) Only an issue for companies that are putting things in the water that can't be easily removed prior to release to surface impoundments
 - c) Use the oxidative process to recycle and reuse aqueous solutions. The ability to achieve selectivity of targeted elements would be a plus, but not a necessity in all situations.
 - d) Concentration of materials in solution are very low, requiring processing of large amounts of material and large cells. Other solutions to remove contaminants pose a significant obstacle for electrochemistry to overcome.
 - e) Not so much recycle /re-use, but mitigating materials in process water is an interest area. So inherently yes, that treatment, not so much separation, but treatment of aqueous solutions, by-product streams, and waste streams there is potential. It's not the gross amount of pollution you have in water, it's quite often a few bad actors so to the extent that you can selectively and efficiently treat those materials would be a boon, a cost savings, and a reputation savings.
- 2) Separations (electrowinning) of organic and inorganic materials from wastewaters and/or byproduct liquids in non-chemical manufacturing operations for purpose of resource recovery and feedstock replacement.
 - a) Use the oxidative process to recycle and re-use aqueous solutions. The ability to achieve selectivity of targeted elements would be plus, but not a necessity in all situations.
 - b) Must focus development on materials with high value to justify recovery costs, or high disposal fees that can be reduced.
 - c) The processing of frack water in the oil and gas industry is hugely important to the chemicals industry. It is important that the method of hydro-fracturing shale deposits continue – providing abundant, low cost hydrocarbons for feedstock. Will likely drive the industry for the next 20 years.
- 3) Separation of chemical streams to reduce resource requirement
 - a) The importance of energy cost reduction is dictated by the energy intensiveness of the incumbent process not all chemical production processes have a high energy component.
 - b) Must be able to reduce both the energy component and large capital investment required with large scale thermal processing.
 - c) The industry has been working on improving energy efficiency for several decades and will continue in the near future.
 - d) Energy efficiency always a driver and always an important driver. In fact we continuously have initiatives going on looking at energy efficiency and energy integration. How do you reduce the

overall energy consumption; how do you make sure you are making use of waste energy and waste heat; co-generation; all those things are extremely important.

- e) Compared to a thermal method there is another approach to reduce the activation energy to carry out a chemical reaction. The nature of the actual feedstocks that go into the process also influence the amount of energy required. If you go into the process with a higher energy content feedstock you have a lower energy conversion process. It's the thermodynamics or the energy of the input feedstock. Electrochemistry can potentially overcome both, probably more the kinetics than thermodynamics.
- f) Improvements in energy efficiency is both a significant technical challenge and has the potential to have a large impact on the chemical production process.
- 4) Selective organic reduction
 - a) Reducing the need for hazardous chemicals by replacing their oxidizing or reducing role in a chemical reaction system with electrochemical redox reactions.
- 5) Polymer depolymerization
- 6) Water electrolysis
 - a) The biggest costs for hydrogen production include the energy to produce it, compress it, and transport it. Production costs could be reduced through the development of more efficient catalysts or photo-electrochemical methods.
 - b) On-demand, on-site hydrogen production could also improve the cost of hydrogen generation. Improved catalysts and robust separators would help to reduce the costs of system acquisition and maintenance.
- 7) Electrochemical synthesis of materials from bio-products and incumbent feedstocks for use in end products
 - a) Alternate feedstocks is of great interest to many companies primarily due to an expectation of greater price stability and sustainability aspect. Allocating resources at this time, when oil and gas prices are severely depressed, is challenging. The chemical industry cannot achieve a price premium for using "sustainable" feedstocks or other "green" approaches.
 - b) Next generation chemical manufacturing is of interest photo-chemical processing? The ability to produce radical anions and cations could provide interesting development and synthesis opportunities.
 - c) Development of new materials for sustainable manufacturing is forward-thinking. This would involve replacing materials that have been used for maybe 100 years. Not only is it a challenge to come up with the material with the right properties, but to get customers and their customers to accept and convert will be a major challenge. For a material with regulatory approval, it will be very challenging to gain approval for a new material. Just the development of a new chemical to replace an old chemical is a huge expense, probably an unrecoverable cost. And then it needs to be on the TSCA (Toxic Substances Control Act) list?
 - d) The need to develop alternative feedstocks has greatly diminished in the last few years due to new projections on the availability and price of hydrocarbons. There will always be an "interest" in bio-based materials due to a fear of changes in the hydrocarbon market – a form of price hedge. Until there is a major change in the long-term view on hydrocarbon availability and pricing, the development of alternative feedstocks will be low on the priority list.

- e) Technologies that will deliver inexpensive feedstocks, possible selective processes, are of great interest.
- f) The ability to synthesize improved raw materials could lead to significantly differentiated product offerings. Many are evaluating bio-based feedstocks as an alternative to petroleumderived raw materials, however electrochemical routes to processing of these new raw materials have not been investigated due to lack of awareness of technologies that can convert bio-based feedstocks into the raw materials needed.
- g) A major consideration will be how chemicals and/or monomers from bio-based feedstock compare in terms of properties, especially contaminants, versus current sources? Trace contaminants will be different and have an impact on downstream users in the supply chain.
- 8) Recovery of spent/unused chemicals by downstream users or consumers
- 9) Separation of carbon nanotubes
- 10) Modification of hydrocarbons
 - Activation of carbon-hydrogen bonds to allow further chemistry e.g. alkanes. Transition metal chemistry is the current method used, but very expensive. Catalysts are hard to develop, and their structure becomes complex.
 - b) Take some basic raw material feedstock like ethylene or propylene feedstocks and create the radical anions and radical cations and try to understand how you control that so you could do polymerization reactions or substitution reactions.
 - c) Catalytic nanowires for chemical processing and surface area chemistry.
 - d) Use of nanowires to modify substrate surface before coating application. Modify the surface to create highly reactive surfaces; the ability to provide better flow characteristics; less turbulent flow with modified surfaces.
- 11) Other opportunities
 - a) Demonstrate that electrochemical processes can reduce the construction costs for new chemical processing capacity. Most chemical plants are built in the field from structural steel. If more of the plant could be built in a factory – in a controlled environment – and then simply assembled at the site this would reduce construction costs.
 - b) For chemicals with high distribution costs, the ability to build small-scale production facilities based on electrochemistry may be interesting. Thermal plants cannot be downscaled easily.
 - c) While there is quite a bit of interest in next generation manufacturing, investments have been limited to small-scale research and proof-of-concept. Few companies are investing in development.
 - d) The complexity of operations is always the driver. You are producing materials that are well known in the market place and the purity and processing standards for these products have been well-defined and it's a big inhibitor to new technology development and with some industry standards, if you change anything your downstream customers have to go through the requalification which is expensive and an inhibitor.
 - e) Treatment of exhaust emissions from vehicle (car, truck, bus, etc.) engines. Removal of particulate and/or Greenhouse gases. Processing can be accomplished at any point of the combustion process on the vehicle. Energy efficiency and package size are critical objectives for this application.

- f) On-board production of hydrogen for vehicles. Would require 100% selectivity toward hydrogen.
- g) The bigger opportunity for electrochemistry at this point is in the development of nanostructured surfaces that impart specific functionalities to materials. For example, electrochemical methods could be used to impart, add or remove functional groups from the surface of a material to make it more hydrophobic or hydrophilic. As another example, an electrochemical treatment on the surface of a composite could improve the adhesion between the fiber and a polymer resin to increase the strength of a composite.

11.4. Appendix D - Participating Companies

Capital Resin Corp DuPont (2 sites) PolyOne Sherwin Williams Ashland Chemical –Valvoline Worthington Industries **Owens Corning** Covestro (formerly Bayer Material Science) **First Solar IGS Energy** Eastman Chemical Tenneco Faraday Technology Inc. De Nora Tech IBM (Electrochemical Society) Archer Daniels Midland **Dow Chemical Graftech International** Zyvex Energizer