Chemical manufacturing creates products by transforming organic and inorganic raw materials using chemical processes. There are over 100,000 chemicals in the marketplace. Chemicals can very broadly be classified into two groups: commodity chemicals and specialty chemicals. Commodity chemical manufacturers produce large quantities of basic and relatively inexpensive compounds (up to $1 per kg) in large plants, often built specifically to make one chemical. Commodity plants often run continuously, typically shutting down only a few weeks a year for maintenance. Specialty-batch or performance chemical manufacturers produce smaller quantities of more expensive chemicals ($1 to $1,000 per kg) on an “as needed” basis in plants that are used less frequently. Often there is only one or a limited number of suppliers producing a given product. In contrast to the production of commodity chemicals, batch manufacturing requires that the raw materials, processes, operating conditions, and equipment change on a regular basis to respond to the needs of customers.

Despite the large number of chemicals available in the market, electrochemical synthesis of chemicals has been limited to a narrow spectrum. The reasons for this have been previously attributed to a lag in the education of chemists and engineers in electrochemistry and chemical engineering, a lack of suitable resources for cell construction, and most importantly the prohibitive costs involved (in many cases) in electrochemical synthesis. However, over the past 40 years, there have been significant developments in electrochemical synthesis and methods due to the advances in materials science and nanotechnology, the development of in-situ spectroscopy techniques, and progress in multi-scale modeling. As a result, it is timely to revisit some industrial electrochemical processes and to introduce examples of new economic opportunities for the electrochemical manufacturing of chemicals.

### Chlor-Alkali

The Chlor-Alkali industry is one of the largest chemical processes worldwide. Its two main components – chlorine and caustic soda – are indispensable commodities that are used for a wide range of applications. Nearly 55 percent of all specialty chemical products are used less frequently. Often there is only one or a limited number of suppliers producing a given product. In contrast to the production of commodity chemicals, batch manufacturing requires that the raw materials, processes, operating conditions, and equipment change on a regular basis to respond to the needs of customers.

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### Aluminum

The electrochemical production of aluminum is one of the most successful examples of how electrochemical reactors can reduce the cost of commodities. Before the implementation of electrolysis, aluminum was as expensive as silver. Today, aluminum (average 2014 price $0.84 per lb) is about 400 times cheaper than silver (average 2014 price $340 per lb). However, primary aluminum production today is ranked among the most energy and CO\textsubscript{2} intensive industrial processes. Specifically, it is among the world’s largest industrial consumers of energy, having an energy cost which accounts for approximately 30% of its total production cost.

All of the primary aluminum production occurs through one industrial practice, which consists of three steps: (a) mining of bauxite, (b) production of alumina (Al\textsubscript{2}O\textsubscript{3}) known as the Bayer process, and (c) reduction to Al metal known as the Hall–Héroult process. Please see pages 36 and 37 of the summer 2014 issue of Interface for an ECS Classics article on the Hall–Héroult process by past ECS Editors note.
The Hall–Héroult process include a) direct emissions (from the reaction of oxygen with the carbon-based anodes) and indirect emissions (from the use of fossil fuel based electricity) of CO₂, and b) emissions of perfluorcarbons (PFCs) like CF₄ and C₂F₆, released due to the anode effect in the cryolite bath.

In the Hall–Héroult process alumina is reduced electrolytically to aluminium in a molten cryolite bath while carbon is consumed (oxidized) during the electrolytic process and supplies part of the energy necessary for the reduction of alumina, according to the following reaction:

\[ 2\text{Al}_2\text{O}_3 + 3\text{C} \rightarrow 4\text{Al} + 3\text{CO}_2 \]

The theoretical minimum energy requirement for the reduction of alumina in a carbon cell is 6.16 kWh/kg Al (with CO₂ emission at 960 °C). The theoretical decomposition voltage is 1.7 V; however, in practice the cell operates at about 4.7 V mainly because of ohmic losses throughout the components. High-grade alumina is dissolved in a molten bath consisting mainly of cryolite (Na₃AlF₆) at a temperature of about 960 °C. Consumable carbon anodes are employed in the electrolytic cell to produce molten aluminum, which is periodically withdrawn from the cathode by vacuum siphoning. The electrolytic cells used in the process need to be periodically replaced, producing a carbon-based solid waste (0.02 kg/kg Al) known as Spent Pot Lining (SPL), which is classified as a hazardous waste due to its chemical composition (12% F and 0.15% CN).

The energy consumed by the Hall–Héroult process (processing energy) and the resultant GHG emissions are summarized in Table 2. The total energy consumption and the GHG emissions depend on the source of energy used. A significant increase in both is observed when electricity from coal is used vs. hydroelectric. Independently of the electricity source, the electrochemical reaction is the highest source of energy consumption, accounting for up to 75% of the energy consumed and for 50% of the GHG emissions generated.

Despite the successful use of electrolysis for the production of aluminum, research efforts are still needed to improve energy efficiency, to minimize the use of consumable anodes, and to minimize waste generation. Recently, ARPA-E, under the modern electro/thermochemical advances in light-metal systems (METALS) program, has sponsored research and development projects for the implementation of new technologies in the electrolysis of aluminum (e.g., advanced electrolytic cells with power modulation and heat recovery, dual electrolyte and electrolytic membrane extraction, and pure oxygen anode electrode). Hence, aluminum production offers another example of an established electrochemical manufacturing process that nevertheless offers challenges and opportunities for the future.

Adiponitrile (ADP) is a key intermediate for the production of nylon 6, 6 polymers. It is used for the synthesis of hexamethylenediamine (HMD), which along with adipic acid are the raw materials for the production of nylon 6, 6 fibers and resins. The electrochemical route for synthesizing adiponitrile is employed by Solutia in Decatur, Alabama in the U.S., Asahi Chemical in Nobeoka, Japan, and by BASF at Seal Sands, UK. Chemically ADP is produced by DuPont in Texas, U.S. and by Butachimie (joint venture between DuPont and Rhodia) in Chalampé, France.

According to the report from ICIS, a petrochemical market information provider, in 2000, world production for the adiponitrile was 1.375 million metric tons per year of which 32.8% was produced via an electrochemical route. In a 2005 report, the global production increased to 1.564 million metric tons per year but the contribution from the electrochemical route dropped to 30.8% (0.481 million metric ton per year). An assessment from PCI Nylon, a market research consultancy, is that 1.197 million metric tons of ADP was produced in 2010 and only 29% of the ADP produced was from acrylonitrile. The demand for nylon 6, 6 is the driving force for the production of HMD and ADP, and this has remained fairly consistent over the past decade. However, the electrochemical synthesis of ADP has suffered slightly in its contribution towards global production mainly due to the reduced prices for the raw materials used in the butadiene (chemical) route of ADP production. Natural gas is used in the butadiene route to synthesize ADP and in recent years the price for natural gas has also come down.

### New Vistas for Electrochemical Manufacturing of Organic Compounds

The ADP electro synthses process is a classical example of electrochemical manufacturing of organic compounds. Due to environmental regulations, combined with advances in materials, the opportunity now exists to capitalize on the electrochemical synthesis of organic compounds from unconventional organic sources.

### Table II. Processing energy and GHG emissions produced by the Hall–Héroult process.

<table>
<thead>
<tr>
<th>Type of Energy</th>
<th>Part of the process</th>
<th>Energy Consumption (kWh/kg Al)</th>
<th>CO₂ emissions (kg CO₂/kg Al)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydroelectric</td>
<td>Reaction</td>
<td>14.50</td>
<td>3.82</td>
</tr>
<tr>
<td></td>
<td>Carbon anode baking</td>
<td>6.01</td>
<td>3.83</td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td>20.51</td>
<td>7.65</td>
</tr>
<tr>
<td>Coal Based electricity (3 kWh for every 1 kWh required)</td>
<td>Reaction</td>
<td>46.86</td>
<td>13.50</td>
</tr>
<tr>
<td></td>
<td>Carbon anode baking</td>
<td>6.51</td>
<td>4.30</td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td>53.37</td>
<td>17.80</td>
</tr>
</tbody>
</table>
Examples of this new avenue include the electrolysis of carbon dioxide\textsuperscript{26-33} and the electrolysis of coal and solid fuels (e.g., pet coke, paper pulp, biomass).\textsuperscript{34-40} These opportunities are briefly discussed below.

Electrolysis of carbon dioxide.—Carbon dioxide is currently generated at rates that far outweigh its removal/conversion to raw materials such as fuels and chemicals. The possibility of converting this abundant waste to useful products has created an avenue of interest from the perspectives of both sustainable energy and environmental decontamination.\textsuperscript{3} Although several chemical and catalytic methods have been tested for converting carbon dioxide to fuels, including reaction with hydrogen, hydrocarbons and organic carbonates, the use of electricity provides a more sustainable approach, due to the possibility of renewable sources (solar PV, wind) being used as the source of electricity.\textsuperscript{26} While alternative chemical routes for CO\textsubscript{2} reduction have shown disadvantages such as deactivation of catalysts due to the presence of water (methanol and ethanol formation) and high reaction temperatures (carbon monoxide formation from C – O dissociation), electrochemical CO\textsubscript{2} reduction can be performed under ambient conditions.\textsuperscript{27-28}

The electrochemical reduction of CO\textsubscript{2} has been investigated in aqueous and non-aqueous solutions. Various metal electrodes (Pb, Hg, Tl, In, Sn, Cd, Bi, Au, Ag, Zn, Pd, Ga, Cu, Ni, Fe, Pt, and Ti with CO\textsubscript{2} reduction potentials vs. SHE of -1.63 V, -1.51 V, -1.60 V, -1.55 V, -1.48 V, -1.63 V, -1.56 V, -1.14 V, -1.37 V, -1.54 V, -1.20 V, -1.24 V, -1.44 V, -1.48 V, -0.91 V, -1.07 V, and -1.60 V, respectively) have been explored for this reaction.\textsuperscript{41,3}

### Table III. Examples of industrial organic electrosynthesis processes at commercial and pilot plant stages of operation. Adapted with permission from Table 2 in Sequeira, CAC and Santos DMF (2009) Electrochemical Routes for Industrial Synthesis. Place: Brazilian Chemical Society.\textsuperscript{22}

<table>
<thead>
<tr>
<th>Product</th>
<th>Starting Material</th>
<th>Company</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetoin</td>
<td>Butanone</td>
<td>BASF</td>
</tr>
<tr>
<td>Acetylenedicarboxylic acid</td>
<td>1,4-Butynediol</td>
<td>BASF</td>
</tr>
<tr>
<td>Adipoin dimethyl acetal</td>
<td>Cyclohexanone</td>
<td>BASF</td>
</tr>
<tr>
<td>Adiponitrile</td>
<td>Acrylonitrile</td>
<td>Monsanto (Solutia), BASF, Asahi Chemical</td>
</tr>
<tr>
<td>4-Aminomethylpyridine</td>
<td>4-Cyanopyridine</td>
<td>Reilly Tar</td>
</tr>
<tr>
<td>Anthraquinone</td>
<td>Anthracene</td>
<td>L. B. Holliday, ECRC</td>
</tr>
<tr>
<td>Azobenzene</td>
<td>Nitrobenzene</td>
<td>Johnson Matthey Company</td>
</tr>
<tr>
<td>p-1-Butylnaphthaldehyde</td>
<td>p-1-Butyltoluene</td>
<td>BASF, Givaudan</td>
</tr>
<tr>
<td>L-Cysteine</td>
<td>L-Cystine</td>
<td>Wacker Chemie AG</td>
</tr>
<tr>
<td>1,4-Dihyronaphthalene</td>
<td>Naphthalene</td>
<td>Clariant</td>
</tr>
<tr>
<td>2,5-Dimethoxy-2,5-dihydrofuran</td>
<td>Furan</td>
<td>BASF</td>
</tr>
<tr>
<td>Hexafluoropropyleneoxide</td>
<td>Hexafluoropropylene</td>
<td>Clariant</td>
</tr>
<tr>
<td>m-Hydroxybenzyl alcohol</td>
<td>m-Hydroxybenzoic acid</td>
<td>Otsuka</td>
</tr>
<tr>
<td>p-Methoxybenzaldehyde</td>
<td>p-Methoxytoluene</td>
<td>BASF</td>
</tr>
<tr>
<td>Perfluorinated hydrocarbons</td>
<td>Alkyl substrates</td>
<td>3M, Bayer, Clariant</td>
</tr>
<tr>
<td>Salicylic aldehyde</td>
<td>o-Hydroxybenzoic acid</td>
<td>India</td>
</tr>
<tr>
<td>Succinic acid</td>
<td>Maleic acid</td>
<td>CERCI, India</td>
</tr>
<tr>
<td>3,4,5-Trimethoxytolyl alcohol</td>
<td>3,4,5-Trimethoxytoluene</td>
<td>Otsuka Chemical</td>
</tr>
</tbody>
</table>

**Commercial Process**

1-Acetoxynaphthalene | Naphthalene | BASF |
2-Aminobenzyl alcohol | Anthranilic acid | BASF |
Anthraquinone | Naphthalene, butadiene | Hydro Quebec |
Arabinose | Gluconate | Electrosynthesis Co. |
1,2,3,4-Butanetetracarboxylic acid | Dimethyl maleate | Monsanto |
3,6-Dichloropicolinic acid | 3,4,5,6-Tetrachloro-picolinic acid | Dow |
Ethylene glycol | Formaldehyde | Electrosynthesis Co. |
Glyoxylic acid | Oxalic acid | Rhone Poulenc, Steetley |
Hydroxymethylbenzoic acid | Dimethyl terephthalate | Clariant |
Monochloroacetic acid | tri- and di-Chloroacetic acid | Clariant |
Nitrobenzene | p-Aminophenol | India, Monsanto |
5-Nitronaphthoquinone | 1-Nitronaphthalene | Hydro Quebec |
Partially fluorinated hydrocarbons | Alkanes and alkenes | Phillips Petroleum |
Propionic acid | Propargyl alcohol | BASF |
Propylene oxide | Propylene | Kellog, Shell |
Substituted benzoic acids | Substituted toluenes | Hydro Quebec, W.R. Grace |

**Pilot Process**

(continued on next page)
There are numerous products that arise during CO₂ electrochemical reduction based on the number of electrons transferred over the course of the reaction. In fact, one study has shown as many as 16 products in varying amounts (formate, carbon monoxide, methanol, glyoxal, methane, acetate, glycoaldehyde, ethylene glycol, acetaldehyde, ethanol, ethylene, hydroxyacetone, acetone, allyl alcohol, propionaldehyde, and 1-propanol). However, while the reduction potentials for the respective reactions indicate mostly favorable thermodynamics, the poor kinetics necessitate overpotentials upwards of 1.0 V to yield these products. In most instances, multiple products are formed leading to poor selectivity. The bottleneck in CO₂ reduction has been postulated to be due to the formation of a radical anion:

\[ \text{CO}_2 + e^- \rightarrow \text{CO}_2^\cdot \ \ (\text{-}1.90 \ \text{V vs. SHE}) \]

In addition to this limiting step, a competing reaction is hydrogen evolution reaction in aqueous environments:

\[ 2\text{H}_2\text{O} + 2e^- \rightarrow 2\text{OH}^- + \text{H}_2 \ \ (\text{-}0.41 \ \text{V vs. SHE}) \]

These issues with electrode kinetics and the nature of the chemical environment as well as the issues associated with poor selectivity indicate that further fundamental understanding of CO₂ electroreduction is required before viable electrochemical manufacturing of useful products from CO₂ can be realized at a commercial scale. However, this is undoubtedly an area with immense potential and opportunity for further exploration at the laboratory and perhaps pilot scales.

Electrolysis of coal and solid fuels.—The controlled manipulation of complex hydrocarbon sources (e.g., coal, pet-coke, lignin, pulp paper, biomass) to high value products with minimum carbon dioxide emissions is a tantalizing prospect and presents an excellent opportunity for electrochemical manufacturing of chemicals from complex and messy feedstocks. One example is the electrolysis of coal, wherein researchers have demonstrated that through the implementation of advanced materials, nanotechnology, and appropriate cell design, significant improvements in process performance can be achieved. A case in point is the nearly 2 order of magnitude improvement observed over the past several decades from current densities of 4 mA/cm² in the late 1970s to up to 250 mA/cm² today.

The efficient production of hydrogen (22 kWh/kg of hydrogen) with 100% faradic efficiency, liquid fuels, and graphene has been demonstrated recently. The formation of the films can involve multiple complex reactions depending on the type of coal. 35 The electrochemical oxidation of coal is a complex process due to the heterogeneity of the material, it has been hypothesized that the anodic current is used for the direct oxidation of coal to carbon dioxide and for the formation of the films. Assuming that coal can be represented as carbon, the electrochemical oxidation of coal to carbon dioxide takes place according to:

\[ \frac{1}{2} \text{C} + \text{H}_2\text{O} \rightarrow \frac{1}{2} \text{CO}_2 + 2\text{H}^+ + 2e^- \ \ (1) \]

The majority of the current in the process (75-85% depending on the type of coal) is used for the oxidation of coal into hydrogen rich films that grow on the surface of the coal particles (for example, simplified reaction 2), while the electrochemical oxidation of coal to carbon dioxide (according to simplified reaction 1) is low. When Wyodak coal is used for the process, 75% of the coal is oxidized according to reaction 2, while 25% of the coal yields CO₂. On the other hand, at the cathode the evolution of hydrogen occurs with 100% faradaic efficiency.

Step 2 of the process, extraction of liquid fuels, capitalizes on the formation of the films on the surface of the coal to yield chemicals and fuels. It has been demonstrated that liquid fuels can be obtained through extraction using ethanol at 78 °C and ambient pressure with a yield of 0.2 g per g of coal. Using the combined electrolysis/liquid extraction process presents an opportunity to reduce costs for the production of liquid fuels and chemicals from coal. Another application for the use of the electrolyzed coal is the synthesis of carbon nanostructures, nanotubes, and graphene – step 3 of the process. For example, Vijapur et al. have demonstrated the synthesis of graphene films from coal using chemical vapor deposition.

The electrochemical manufacture of high-value chemicals from coal is another electrochemical manufacturing technology that is in a nascent stage and, like CO₂ electro-reduction, will benefit greatly from further research and development efforts.

**Outlook**

Due to recent technological advancements and a changing economic climate, electrochemical technologies and processes now represent a relatively untapped frontier of opportunity for unique, enabling, and translational solutions that can benefit the chemical industry. Electrochemical processes provide significant benefits including:

- **Easy integration with renewable energy (electricity) sources.** The scalability of the technologies, as well as their ability to easily operate in an on-demand mode, facilitates the technologies’ ability to interface with renewable, time-varying energy sources.
Fig. 1. (a) Electrochemical manufacturing of high value chemicals (hydrogen, liquid fuels, graphene) from coal. (b) Galvanostatic performance of the coal electrolytic cell at 100 mA/cm².
Minimization of purification and separation costs. Electrochemical synthesis and/or electrolysis potentially allow the direct production of pure fuels and/or chemicals.

Ease of operation at low temperature and pressure. Electrochemical synthesis and/or electrolysis typically takes place at low temperatures and pressures as compared to traditional heterogeneous catalytic synthesis. This could represent significant cost savings.

Mid-term impact. The timeframe for implementation of these technologies could be mid-term to long-term (five to twenty years from now).

Ease of storage and transportation of feedstock and fuels. Liquid fuels catalyzed through these processes can be transported, stored, and used using existing technology and infrastructure.

The chemical and allied industries (ChEAllieds) confront technology challenges – e.g., reliability of energy supply, lack of energy efficient/transformative manufacturing technologies, waste reduction, and water conservation – that hinder and jeopardize their growth and affect their competitiveness worldwide. Current chemical industry production methods have approached their practical performance limits, therefore, new disruptive, and enabling technologies are needed that will provide solutions to the ChEAllieds beyond incremental manufacturing improvements. Electrochemical manufacturing could provide opportunities for the ChEAllieds and, given the advantages and outlook discussed above, this is an emergent area for research and development. The Electrochemical Pathway for Sustainable Manufacturing (EPSuM) Consortium69 funded by the National Institute of Standards and Technology (NIST) through the Advanced Manufacturing Technology Consortia (AMTech) Program is an example of an attractive collaborative model for pursuing translational advances in this sector. The overarching goal of this consortium is to develop a technology roadmap to support, sustain, and enhance U.S. manufacturing capacity in the nation’s chemical industry and allied sectors through innovative processes that utilize electrochemical science and technology to address major technical barriers. Partners on the Phase I of the program are the Center for Electrochemical Engineering Research at Ohio University,57 the National Science Foundation Industry University Cooperative Research: Center for Electrochemical Processes and Technology (CEProTECH),58 PolymerOhio Inc.,59 The Electrochemical Society,60 and multiple companies. Similar government-university-industry partnerships would greatly advance the electrochemical manufacturing sector and should be actively pursued.

About the Author

GERARDINE (GERRI) BOTTE is the Russ Professor of Chemical and Biomolecular Engineering at Ohio University, the founder and director of Ohio University’s Center for Electrochemical Engineering Research, and the founder and director of the National Science Foundation I/UCRC Center for Electrochemical Processes and Technology. Dr. Botte and members of her research group are working on projects in the areas of electrochemical engineering, electro-synthesis, batteries, electrolyzers, sensors, fuel cells, mathematical modeling, and electro-catalysis. Example projects include: hydrogen production from ammonia, biomass, urea, and coal, synthesis of carbon nanotubes and graphene, water remediation, selective catalytic reduction, ammonia synthesis, and electrochemical conversion of shale gas and CO2 to high value products. She has 116 publications (peer-reviewed, book chapters, proceedings, and patents) and over 190 presentations in international conferences. She is the inventor of 18 U.S. patents and 29 pending applications. She is the Editor in Chief of the Journal of Applied Electrochemistry. In 2010, she was named a Fellow of the World Technology Network for her contributions on the development of sustainable and environmental technologies. In 2012 she was named a Chapter Fellow of the National Academy of Inventors. Dr. Botte served as past Chair, Vice-Chair, and Secretary/Treasurer of the ECS Industrial Electrochemistry and Electrochemical Engineering Division. She received her BS in Chemical Engineering from Universidad de Carabobo (Venezuela) in 1994. Prior to graduate school, Dr. Botte worked as a process engineer in a petrochemical plant (Petroquimica de Venezuela) where she was involved in the production of fertilizers and polymers. She received her PhD in 2000 (under the direction of Ralph E. White) and ME in 1998, both in Chemical Engineering, from the University of South Carolina. Prior to joining Ohio University as an assistant professor in 2002, Dr. Botte was an assistant professor at the University of Minnesota-Duluth. She may be reached at botte@ohio.edu.

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