

SPARKING Students Interest in Electrochemical Engineering

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Abstract

A new course in Electrochemical Engineering was given at Rowan University using an inductive teaching format. This format consisted of incorporating electrochemical engineering and electrochemistry experiments into the lecture. For this class we used an inductive presentation style of the material which started with an experiment. If an experiment was not possible then the results of an experiment were shown or a demonstration was given. Next a lecture was presented on the material using the experimental results followed by a presentation of the theory.

Introduction

There is a tremendous potential for the use of electrochemical engineering in growth areas such as fuel cells, bio-sensors, electrochemical sensors and batteries. In addition many new green chemical reactions are being proposed to replace traditional organic reactions. New environmental separation processes are being used for the removal of metals from wastewater. In addition to these new processes, traditional areas of chemicals production that have a major market share, such as that of the chlor-alkali industry, need to have further improvements made by process engineers to remain competitive. All of these areas have a high potential growth and chemical engineers will have a large impact in these areas.

Electrochemical engineering tends to be perceived as a relatively daunting course in chemical engineering. Students see topics using concepts that are very unfamiliar. A typical chemical engineering student will have seen some concepts related to ions in their chemistry courses such as the dissociation of ions and they will have balanced a redox reaction, but this is typically done in the freshman year and is covered near the end of the chemistry course. Since most electrochemical engineering courses are given as electives in the senior year the student is very unfamiliar with this material.

The electrochemical engineering course developed at Rowan University has been designed to introduce various topics of electrochemical engineering through an integration of experiments, projects, homework and lectures.

Current Electrochemical Engineering Education within Chemical Engineering

In many cases aspects of electrochemical engineering have been relegated to subsections of standard texts such as in Perry's 7th Edition of the Chemical Engineering Handbook. A relatively small section on fuel cells under the title, Electrochemical Energy Conversion (6 pages), and then another section on electrophoresis and electrofiltration in the Alternative Solid/Liquid Separations (8 pages). In the index sections on electrodialysis (6 pages),

electroanalytical instruments (3 paragraphs), electrostatic separators (5.5 pages). After that there is only passing mention in mass transfer correlations, and electrochemical oxygen sensors. Sections on the Thermodynamics of Electrochemical Cells and electrolytic solutions have been dropped. And this is at a time when process simulators are just beginning to include thermodynamic models of these systems.

A common area of chemical engineering related to material properties is a section on corrosion. This is normally covered in either a materials course or in a senior design course on selection of materials. In 1985 the University of South Carolina presented a paper on the integration of an electrochemical engineering and corrosion course to reinforce the electrochemistry fundamentals in these two courses.¹ In this course 2 important areas are covered that are of continual concern to chemical engineers.

A text is available as an extended version of a DECHEMA Experimental Course electrochemical engineering.² This text is based on 12 years of experience of giving experimental courses in electrochemical engineering. Its major emphasis is on using experiments to illustrate the basic principles of electrochemical engineering. The book contains 23 experiments ranging from basic electrochemical techniques to electrochemical processes found in separators and reactors. These experiments are on an advanced level and require various electrochemical instrumentation and apparatus.

Aspects of electrochemical engineering have been incorporated in various aspects of the chemical engineering curriculum. In the unit operations or senior laboratory course experiments in metal recovery using electrodeposition on a porous carbon cathode³, In the process control lab, Jan Talbot has initiated student designed experiments in electrochemical engineering.^{4,5} In this course the students must plan design and build and experimental apparatus. Then they must demonstrate that it works and analyze the data. Finally they give an oral presentation in addition to a technical report. In this class, projects such as a methanol-air fuel cell, electrolytic gas evolution, reciprocating paddle electroplating, electroforming process for inkjets, packed-bed electrode system for waste treatment of printed circuit board waste, electrokinetic soil remediation, electrochemical oxidation of organics, electrochemical ion exchange separator. This course is an excellent approach to teaching aspects of electrochemical engineering and the current course developed at Rowan University was based in part on this work.

Emerging areas of chemical engineering such as semiconductor processing and biomedical engineering can be a format for introducing electrochemical engineering. Mark Orazem introduces principles learned from mass transport, thermodynamics and heterogeneous and homogeneous kinetics to electrochemical processes within a semiconductor.⁶ Alvin Salkind incorporates recent progress in therapeutic and rehabilitative devices and ongoing research based on electrochemical engineering. These include the design of stimulating electrodes, heart pacers, bone and wound repair devices, sensors and pain control techniques. Professor Salkind remarks in this paper the educational and material science parameters necessary for additional future progress in biomedical engineering.⁷

Inductive Approach to Electrochemical Engineering

Instructors can teach inductively by presenting familiar phenomena, practical issues, or experimental observations before presenting a general principle. This procedure is unfamiliar to most professors since they were taught using a deductive style in graduate and undergraduate school. Surprisingly, most textbooks still use an exclusively deductive approach, proceeding from first principles and governing equations to specific applications. With the exception of the DECHEMA experimental course², there are relatively few textbooks that are written using an inductive approach and a professor's prior experience is deductive; this makes implementation of the inductive method a challenge. Another challenge is that students typically will not have a wide range of experience or intuition needed to begin the inductive process. A simple laboratory experiment or demonstration will provide the foundation (observations or data) from which the inductive process is initiated. It is interesting to note that in elementary school the science subjects are being taught use a text written in an inductive style.⁸

We have integrated inductive learning into our coverage of Freshman Clinic, fluids, heat transfer, mass transfer and transport phenomena.⁹ In heat transfer, for example, simple heat exchanger design is the first topic addressed in the course. Discussion of the significance of the overall heat transfer coefficient provides a meaningful framework for introduction of topics such as conduction and convection, which are introduced later in the course. In mass transfer, presented in the context of a transport phenomena course, students start with the design of a gas absorption tower. They are shown both laboratory equipment and pictures of industrial towers used to remove an impurity from a gas stream. If possible, they perform experiments on a laboratory gas absorption tower and observe the gas and liquid flowing over the packing material and measure inlet and outlet gas concentrations. Next, they use a simple model of the tower with a constant overall transfer unit height. The students then proceed to learn about the overall mass transfer coefficients, individual phase coefficients, and the film theory of mass transfer. This portion of the material ends with an experiment in diffusion, Fick's law of diffusion and the measurement and prediction of a diffusion coefficient. Our early efforts in using inductive learning have been presented in 2 previous papers.^{10, 11}

In presenting course material in an inductive frame work we have found that a new preparation of the material must be done. Our experience is in the creative use of experiments with interactive lectures in chemical engineering.

Previous work in this area is highlighted in a discovery course in electrochemistry by Michael May and Vijay Gupta¹². This course was developed at Central State University, to give undergraduates hands-on experience with electrochemical measurements and prepare students for summer research in Fuel Cell and Battery technology. Eight electrochemical experiments were used in this course: Construction of Zinc-Copper battery stacks, Lead Acid Battery discharge-charge cycles, Conductimetric titration of aspirin with Ammonium Hydroxide, Ion Selective Electrode determination of Fluoride in water, Cyclic Voltammetry of Potassium Ferricyanide solution, Cyclic Voltammetry of Sulfuric acid on Platinum working electrode, Anodic Stripping Voltammetry of Lead ion in solution, Differential Pulse Polarography of Lead ion in solution.

Many other experiments have been described in chemistry education journals that help to introduce concepts in electrochemistry. One such notable experiment is the lemon cell¹³. Experiments on electricity for children contain many hands-on activities that are useful in a review of basic electrochemical principles.^{14,15,16} Science kits can be purchased in electrochemistry on-line.¹⁷ This web site has experiments on the

1. Two-Potato Clock
2. The Orange Juice Clock
3. Conductivity Indicator Kit
4. Brass Alloy & Metallurgy Kit
5. Science Kit Metal Plating Sets
6. Effective Education: Tracking Hydronium Ion Concentration
7. Science First Conductivity Tester
8. Kemtec Gel-Cels: Experimenting in Electrochemistry Lab Activity (electrode potentials in safe agar media electrochemical cells)
9. Lab-Aids Introduction to Conductivity Experiment Kit
10. Lab-Aids Conductivity of Solutions Kit
11. Effective Education: Electrochemistry Demo: Using A Salt Bridge
12. 10 Level Conductivity Indicator
13. Audio & Visual Conductivity Indicator
14. Aldon AP Chemistry: ElectroChemical Cells

General chemistry sets can be purchased that include electrochemical experiments such as the Smithsonian Microchem Chemistry Sets.¹⁸ Other websites give description of electrochemistry experiments such as that at Fun Science Gallery¹⁹ or electrochemistry experiments within a chemistry course. One notable website maintained by Peter Keusch contains links to thousands of demonstrations experiments in chemistry.²⁰ Other websites give links to more elementary chemistry experiments.²¹

Rowan Universities Electrochemical Engineering Course

The course was a 3-hour combined undergraduate and graduate audience. To try to attract graduate students from industry the course meets officially once a week for 3 hours. Students are required to complete some laboratories outside of class and are all assigned a laboratory project.

The topics covered in our electrochemical engineering course include

1. Fundamentals of electrochemistry through experiments
2. Mass and Heat Transfer of Electrolytic Cell Systems
3. Energetics of Electrolytic Cell Systems
4. Rate Processes and Reaction Models
5. Reactor Models
6. Fuel Cells
7. Batteries
8. Ion Selective Electrodes
9. Electrochemical Sensors and biosensors

For this electrochemical engineering course we used the textbook by F. Goodridge and K. Scott titled, *Electrochemical Process Engineering: A Guide to the Design of Electrolytic Plant*, published by Kluwer Academic/Plenum Publishers, March 1995. (TP255 .G66 1995, ISBN 0-306-44794-0). This text was chosen because of the practical focus of the book. The authors emphasize that this is a practical guide rather than a textbook and many examples are given to further explain the material. Based on this applied focus the book was deemed to be appropriate for a primarily undergraduate audience. There are many excellent books on electrochemical engineering that were used as reference material for this course.²²

The experiments that were performed in the class were

- Basic Experiments in electrochemistry
- Experiment: Survey of Voltammetric Methods
- Experiment: Electrodialysis
- Hydrogen Fuel Cells
- Experiment: Battery Construction
- Experiment: Electrochemical Sensor

Course Details

The course started off with simple experiments that would refresh the students current knowledge in electrochemistry. These experiments illustrate to the students several basic electrochemical phenomena. The students were given a syllabus and the experimental write-up and moved to the lab to begin the experiments.

The first set of experiments was titled “Introduction to Electricity and Electrochemistry: Experiments for Children.” This series of experiments were designed to motivate the students to examine complicated processes, in an easy to understand format.

The first experiment in this series was a review of basic electronics such as Ohm’s Law, Power, and Series Power sources. In this experiment simple circuits were constructed and measurements were made of voltage, resistance and current. Figure 1 shows the basic circuits that were constructed. In addition to the review of basic electronics the students were able to learn how to use the electronic multimeter which would be used for nearly all of the experiments in the class.

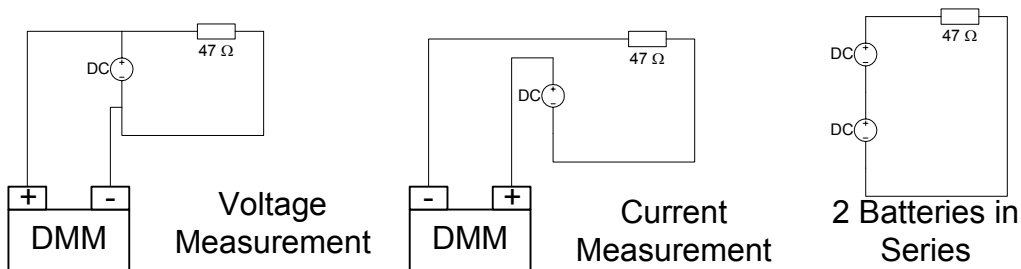


Figure 1: Basic Circuits

The second experiment was an electrolytic cell in which a paper clip was coated with copper. This experiment was based on the book by Michael Flaherty.²³ In this experiment a standard paper clip was coated with copper as shown in Figure 2. In this experiment the students investigated the effect of adding salt, and then vinegar. Next the students measured the change in mass of the 2 electrodes in preparation for a lecture on Faraday's law.

The third experiment was a galvanic cell made from lemons and then a series of beakers of vinegar. This experiment was based on the articles by Swartling¹³ and Stroebel and Myers²⁴ and the text of Glover.²⁵ In the first part lemons

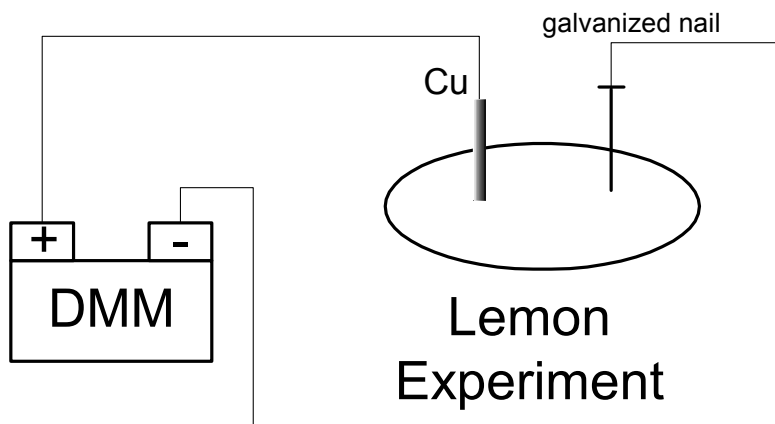
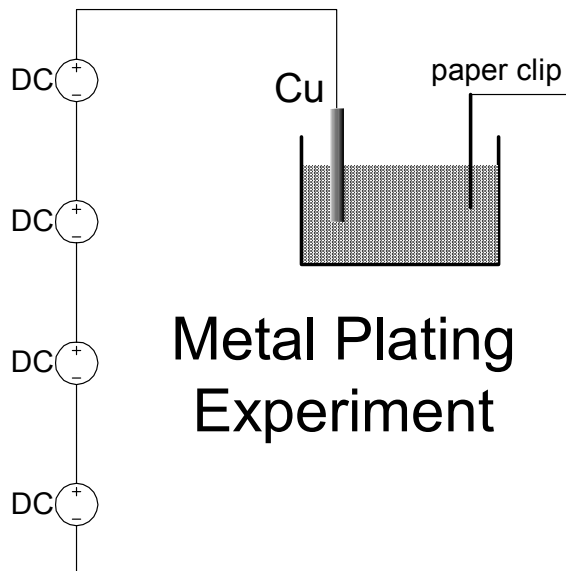


Figure 4: Lemon Experiment



Figure 3: Students Conducting Electrochemistry Experiments

were used to generate a current to light an Light emitting diode. As shown in Figure 4 a copper and a galvanized nail were used for electrodes. In the second part of this experiment 3 beakers of vinegar were used to generate a small current

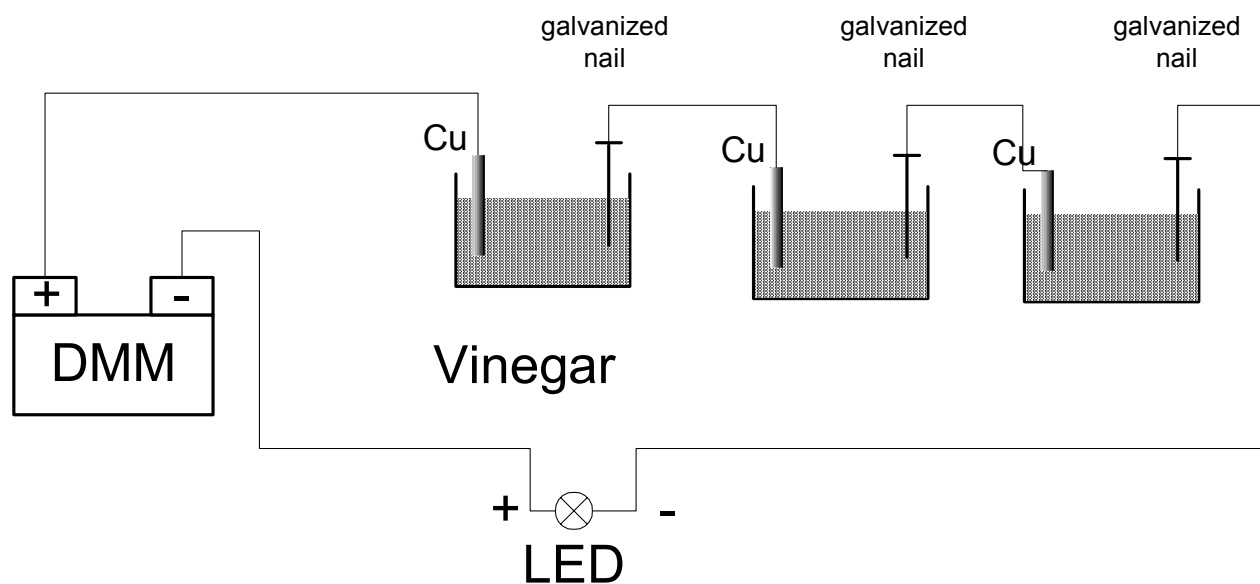


Figure 5: Vinegar Battery

After this first series of experiments were performed then a lecture was presented on the fundamentals of electrochemistry.

In this lecture the basics of electronics were not covered,

instead the lecture started with a discussion of the 2nd and third experiments. Results from these experiments were plotted and analyzed. For example the students were asked to put a strip of zinc metal into copper sulfate and asked to make observations of the result similar to that shown in Figure 6. From this simple demonstration a discussion of spontaneous reactions was undertaken and the concept of Gibbs Free energy and standard electrode potentials was reviewed.



Figure 6: Spontaneous Reaction with Zn and CuSO₄

This lecture was followed by a second set of experiments that were given by Giorgio Carboni.¹⁹ In this set of experiments a Daniell's cell was constructed using a copper and zinc electrode immersed in 1 M Copper Sulfate (CuSO₄) and 1 M of Zinc Sulfate (ZnSO₄) solutions as shown in Figure 7. In these experiments we used both a porous clay planting pot and a salt bridge made by from a Tygon "U" shaped tube filled with saline solution. After this a series of experiments on galvanic deposition was conducted

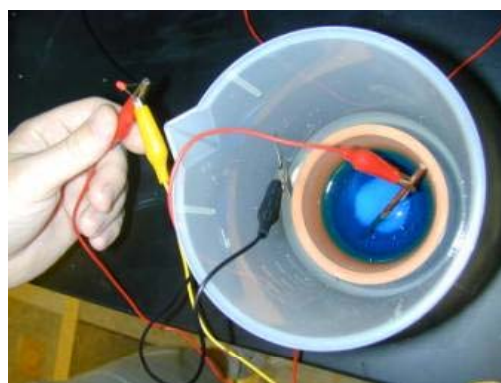


Figure 7: Daniell Cell

using zinc, copper and stainless steel electrodes were performed in solutions of copper sulfate.

These experiments were again followed by a lecture in which the data obtained was analyzed and aspects of electrode reactions, competing reactions, and conductivity were discussed.

Process Specific Experiments.

The previous experiments were general electrochemistry experiments that could have been done previously in general chemistry or physical chemistry laboratories. The following 2 experiments show examples of electrochemical engineering and require specialized equipment.

Electrodialysis Experiments

Electrodialysis is an electrochemical separation technique for ionic solutions that has been utilized in industry for several decades.²⁶ Electrodialysis can be used in the separation and concentration of salts, acids, and bases from aqueous solutions, the separation of monovalent ions from multivalent ions, and the separation of ionic compounds from uncharged molecules. Electrodialysis is a selective transport process in which the ions are removed

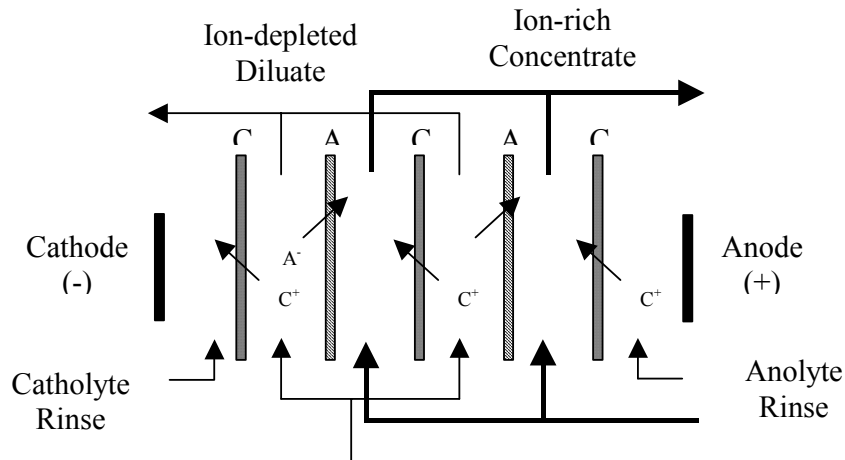


Figure 8: The principle of electrodialysis

from the feed stream through a physical barrier or membrane therefore, a phase change is not necessary for the separation to occur. Other separation methods such as distillation and freezing involve a change of phase of the solvent to accomplish the separation.²⁷

Electrodialysis is employed in several industries for a variety of applications which include: the production of potable water from brackish water, demineralization of whey, deacidification of fruit juices, production of boiler water, and the removal of organic acids from fermentation broth.^{26, 28, 29, 30} Electrodialysis can be used for either electrolyte reduction recovery of ions from feed streams.^{31, 28, 29, 30}

In a typical chemical engineering undergraduate curriculum students are exposed to traditional separations methods with a heavy emphasis on distillation and adsorption processes. The driving force for these typical separation techniques are concentration gradients and phase changes. Electrodialysis is a very unique separation method in that electrical potential is the driving force for the separation to take place. This experiment is designed to provide students with an

understanding of the fundamental principles, design and applications of electro dialysis technology. The experimental data collected from these experiments demonstrate the concepts of the rate of ions transferred from the feed vessel, efficiency, Faraday's law, and energy consumption.

Fundamentals

The principle that governs electro dialysis is an electrical potential difference across an alternating series of cation and anion exchange membranes between an anode and a cathode. The feed solution containing both positive and negative ions enters the membrane stack to which a voltage is applied, thus causing the migration of the ions toward their respective electrodes and into the diluate or concentrate streams. The cation exchange membranes allow the transfer of cations but inhibit the transfer of anions. Conversely, anion exchange membranes allow the transfer of anions but inhibit the transfer of cations. The result is alternating compartments containing streams of dilute ion concentration and streams rich in ion concentration. An ionic rinse solution is recirculated past the electrodes to maintain the conductivity of the membrane stack but prevent potentially corrosive ions from the feed solution from contacting the electrodes. This concept is illustrated in Figure 1 with a feed solution of ammonium chloride.

Electro dialysis is commonly performed in either constant-voltage or constant-current mode.

Calculations relating voltage and current to power consumption and efficiency of operation are of critical interest.

The cell voltage and current within a membrane stack are related through Ohm's Law:

$$V = I \times R \quad (1)$$

where V is the voltage (V), I is the current (A), and R is the resistance of the membrane stack (Ω). The resistance of the membrane stack is due to the resistance of the ions flowing through the membranes and the aqueous solution while being transferred from one solution to another.³⁰ At high voltages, the system does not follow Ohm's Law and the interested reader is referred to standard texts such as Mulder²⁶ for the description of other regimes.

The power consumption necessary to for the removal of ions from the feed solution is proportional to the current and the resistivity of the stack. The necessary power, P (J/s) is represented by equation (2). This equation does not account for power necessary to pump the feed, rinse, or concentrate streams.²⁶

$$P = VI \quad (2)$$

Current is the rate of charged passed and expressed as:

$$I = \frac{dC}{dt} \quad (3)$$

where, C = charge passed (C) and t is time (s). For a system operated at constant voltage the power consumption will change throughout the run as the current changes. The total energy consumption for an experiment is the power integrated over time, and (1)-(3) can be combined to yield an expression for total energy consumption, E (J):

$$E = \int P dt = \int V \frac{dC}{dt} dt = V \times C \quad (4)$$

The efficiency of the membrane stack is a measure of the systems ability to utilize the current effectively in the removal of ions. The number of moles of ions transferred through the stack is related to the theoretical rate of charge passed, C_{theor} (C/s) by Faraday's Law.

$$C_{theor} = Fn_{theo} ([NH_4^+]_{V_{feedo}} - [NH_4^+]_{V_{feed}}) \quad (5)$$

Where F is Faraday's Constant (96,485 C/mol e^-), n_{theo} is theoretical number of moles of electrons per mole of ion passed, and V_{feed} is the volume of the feed fluid (vessel + holdup). The feed stream passes between the cation and anion exchange membrane pairs and ions are removed from the feed and pass to the concentrate solution. In Figure 1, there are 2 pairs of membranes and for every mole of electrons passed to the electrode 2 moles of cations and anions will be

removed from the feed solution. For Figure 1 the value of n_{theo} is given as $n_{theo} = \frac{1 \text{ mole } e^-}{2 \text{ mol ion}}$. In your experiment you have 5 pairs of membranes plus an additional cation membrane to protect the anode. The theoretical charge passed, C_{theor} , is divided by the actual charge passed to obtain the efficiency the current efficiency, $C.E.$ of the membrane stack in the removal of ions²⁸.

$$C.E. = \frac{C_{theor}}{C_{actual}} \quad (6)$$

An efficiency of less than one indicates that not all of the charge passed by the electro dialysis system was utilized to transfer ions from one stream to another. Potential causes of efficiencies less than one include a non ideal selectivity of the membranes for their respective ions, the potential of parallel current paths within the membrane stack, and the transfer of water molecules.²⁷

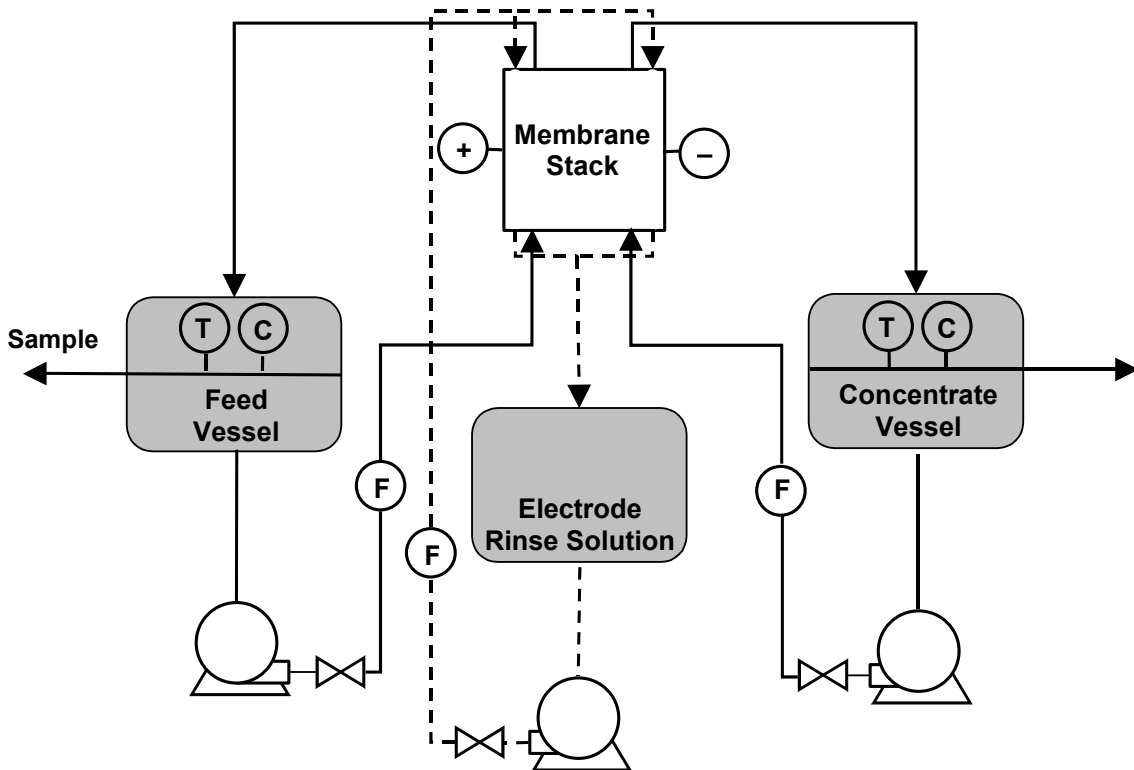


Figure 9: Scheme of the experimental electro dialysis system employed for the separation runs. T, C, F: Temperature, Conductivity and Flowrate on-line analyzers

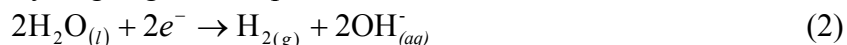
Experimental Procedure and Results

Ammonium cations and chloride anions are contaminating species that are commonly present in industrial process streams. In this investigation we considered the removal of ammonium chloride from an aqueous stream. Experiments were performed to study the effect of the following parameters in the removal of NH_4Cl from water: the initial concentration of the concentrate vessel, the applied cell voltage, and the selection of the membranes. Additional experiments could be conducted to investigate other process parameters such as number of membrane pairs, flow rate, feed concentration, and temperature (within the limits of the membranes). The system performance was evaluated using efficiency, selectivity, power, and energy consumption calculations.

The membrane stack was constructed with 5 pairs of cation and anion exchange membranes, with a cation exchange membrane adjacent to each electrode as shown in Figure 9. A solution of 0.5 M ammonium sulfate was used as the anolyte/catholyte rinse solution, in order to prevent the generation of chlorine or hypochlorite which would be hazardous. The feed solution was aqueous NH_4Cl or $\text{NH}_4\text{Cl}/\text{MgCl}_2$ with a total initial salt concentration of approximately 1.0 M. The concentrate stream was an aqueous NH_4Cl with an initial concentration between 0.05 and 0.2 M (specified for each run). At the anode, the expected predominant reaction in this system with a neutral solution is the formation of oxygen:



At the cathode, the formation of hydrogen gas is the predominant reaction in neutral solution:



The system was operated in a constant voltage, batch recirculation mode. Flowrates were 0.5 L/min for the feed and concentrate streams, and 0.7 L/min for the combined anolyte and catholyte streams. The following data were collected at regular intervals of ten minutes or less: Liquid volume in each vessel, conductivity of each stream, current, charge passed, and temperature. Completion of experiments took between 70 and 90 minutes, depending on the applied voltage and other operating parameters. The experiment was considered complete when the current dropped to approximately 1.0 A. The ion concentrations were determined using conductivity measurements.

Initial concentration of the concentrate vessel is important because it is a key parameter in the resistance of the membrane stack. If the concentrate stream initially has an extremely low ion concentration, water splitting may occur in order to provide the ions necessary to carry the current. (Water splitting generates hydrogen ions and hydroxyl ions, which are capable of migrating through the cation- or anion-selective membranes respectively; this may result in pH changes in the diluate and concentrate compartments.) Thus, the concentrate stream is usually “primed” with a low electrolyte concentration to facilitate transport of the desired ions from the feed stream. To illustrate this, the system performance is compared using different initial concentrate vessel concentrations. The effect of the initial ion concentration in the concentrate stream is shown in Figure 11. These runs were performed at a constant voltage of 13 V using a 1.0 M NH_4Cl feed and 0.5M $(\text{NH}_4)_2\text{SO}_4$ rinse, and the initial concentrate concentration was varied from 0.05M-0.2M NH_4Cl . The figure shows the decline in the feed ion concentration throughout the course of the runs. The rate of ion transport increases with increasing initial concentrate concentration. At higher concentrate concentrations the ionic conductivity of the

membrane increases, effecting a higher current for a given voltage drop across the cell stack, and thus resulting in a higher rate of transport of the ionic species.

Applied cell voltage is a critical operating condition in electro dialysis processes. As the cell voltage is increased, the ion concentration in the feed vessel is depleted more rapidly, thus reducing the duration of the experiment. However, increasing the cell voltage increases the energy consumption of the unit according to Equation **Error! Reference source not found.**. An interesting investigation can be performed by varying the voltage and comparing the system performance and energy consumption.

To study the effect of voltage, the voltage was varied between 8-13 volts, based on manufacturer recommendations.³² The system operated in the Ohmic region within this voltage range. The results of ammonium chloride removal at different voltages are shown in Figure 10, which plots the feed ion concentration throughout the run. This figure shows that at higher cell voltages the feed ion concentration is depleted more rapidly than at lower cell voltages. As the concentrate concentration increases at the start of the run, the current increases and a higher ion transport rate is observed. This can be observed through the change in the slope of the curves in Figure 10 (about 5-10 minutes into the run).

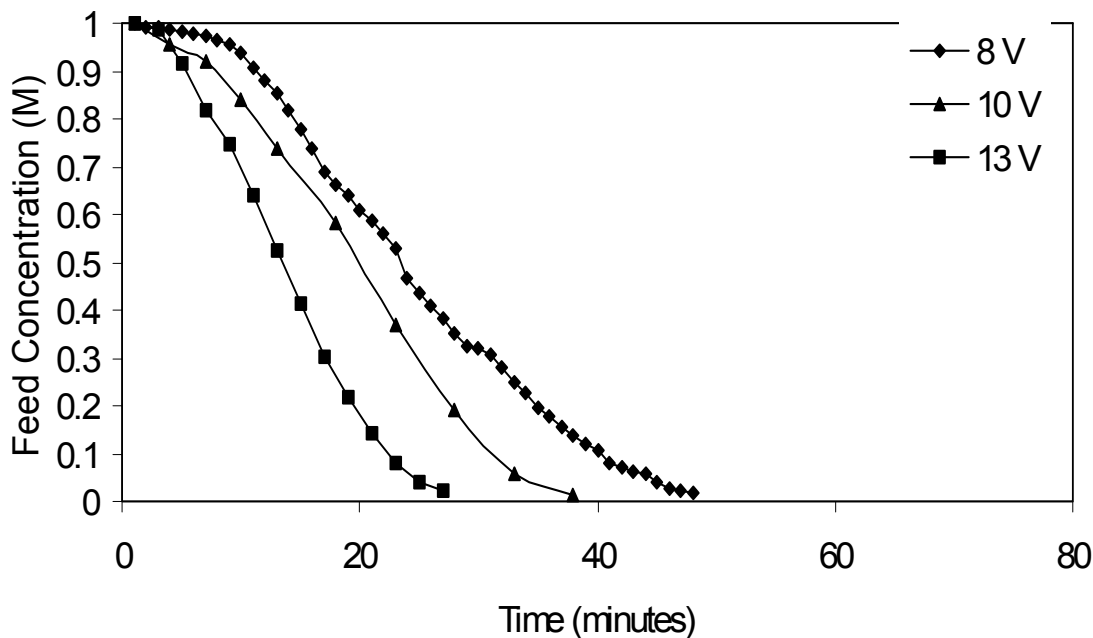


Figure 10: Effect of Voltage on the Feed Concentration. Process operating Conditions: 1 M Feed (initial), 0.1 M Concentrate (initial), AMX/CMX Membranes

The students can calculate the efficiency of the membrane stack in the removal of ions at the various cell voltages. At the conditions shown in Figure 10, overall efficiencies of close to 100% were obtained for the 8 V and 10 V runs, while the efficiency of the 13 V run was approximately 85%. The lower efficiency of the 13 V run was accompanied by an increase in temperature due to finite membrane resistance, which could damage the ion-selective membranes if cooling is not provided. Similarly, the calculated energy consumption for the 13 V run was significantly higher than that for the 8 V or 10 V run, as shown in **Error! Reference source not found.**. The

experimental results for efficiency and energy consumption could be used to estimate the energy requirements and membrane area necessary to achieve this deionization task for a given plant capacity, and to obtain a rough estimation of process costs.

Other experiments were performed and more details can be found in the paper by Farrell et al.³³

The experiment is completed when the feed conductivity drops to 1.5 mS. After the experiment is complete, the system is flushed with deionized water. Typical experimental results are shown in Figure 11.

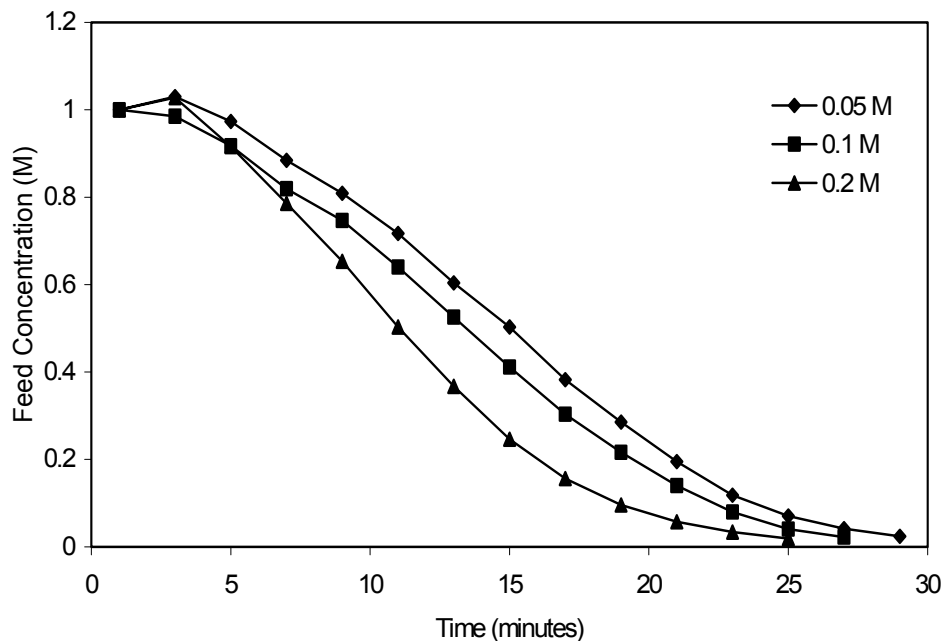


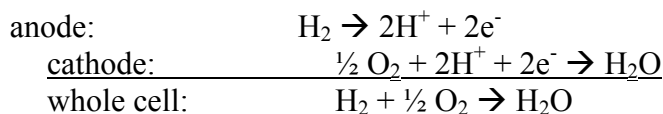
Figure 11: Effect of initial concentrate concentration at constant voltage. Process operating conditions: 13 V, 1 M Feed (initial), AMX/CMX Membranes

Fuel Cell Experiment

The experiments using a fuel cell were developed in conjunction with James G. Lertola of DuPont Central Research & Development, Wilmington, DE. The objective of this experiment is evaluate the mass balance, polarization curve, efficiency, and voltage decay of a hydrogen fuel cell stack.

Introduction

The fuel cell is a device for generating electricity directly from reaction of fuel and oxygen. Unlike traditional electric generators, fuel cells take the energy from a fuel like hydrogen or methanol and convert it directly into electric current without any mechanical engine or turbine in between. The chemical reactions involved are:



Proton-exchange-membrane fuel cells, which are being developed by nearby industrial companies like DuPont and W. L. Gore, may offer the best chance for fuel cells to grow in the marketplace. This type tends to be thin and lightweight, and they are able to operate at low temperatures and start up quickly. Applications will likely start with small electric devices like cell phones and portable computers, then expand into stationary generators and eventually automobiles.



Figure 12: BCS Fuel Cell System

This experiment employs a stack of eighteen hydrogen-powered fuel cells connected in series, which allows it to achieve a high enough voltage to power a small electric lamp. The experimenters will use it to indeed generate electricity, power the lamp, and run its own cooling fans. Finally, they will connect the stack to an electronic load box and measure its performance.

Fundamentals

The fuel cell reacts hydrogen fuel at the anode and oxygen at the cathode to generate an electric current. In this experiment, we will initially observe the variation of the performance of the stack with time. We will develop the polarization curve, and will finally compare the mass and electric-current balances in the system.

It is typical, especially in a relatively brief experiment like this, to observe variation of the fuel cell performance with time. This will be caused by many factors, such as lags in heat transfer and equilibration of the water content in the membranes. In long term usage, fuel cells will be expected to deliver fairly steady voltage over 1000 to 50,000 hours of operation, depending on the application.

The cathode of the PEM fuel cell utilizes flowing air for two purposes. First, oxygen in the air is a direct reactant, combining with the protons from the membrane and the electrons from the electrical circuit to form water. Secondly, the air is a carrier gas, helping to remove the product water so that more oxygen can get in and react. In this stack, air is not pumped or blown through the cathode, but merely passes through its open, vertical channels by natural convection. Depending on the movement of the air in the hood, the rate of air flow may be enhanced by use of a clear plastic box open at the top and bottom. When this box is placed on top of the stack, it acts like a chimney, channeling the warm air upward and therefore drawing fresh air into the stack from the bottom. This effect increases the performance of the stack.

The stack temperature can be varied by means of four small DC-powered fans, whose source of electric current is the fuel cell itself. The fans are an example of "parasitic loads" which must be placed on a fuel cell system; other such loads in real applications are pumps, blowers, preheaters, and controllers. A well-designed fuel cell system will have minimal parasitic loads.

The use of these fans will cause the stack to operate at a lower temperature. When they are turned off, the higher temperature will change many factors affecting the performance of the cells, particularly the rates of the electrochemical reactions and the diffusivity of protons and water across the membranes. Generally, the cell will perform better at higher temperature.

The polarization curve is the clearest measure of a fuel cell's performance. It is a plot of voltage versus current density; as the current density increases, the voltage drops off, finally falling off to zero when the mass transfer rate of a reactant or product reaches its maximum possible value. A good cell has a high polarization curve. An alternative way of showing this data is as a plot of power density versus current density; power density p is simply the product of the voltage V and the current density i :

$$p = V i$$

In addition to voltage and current, the stack's performance can be looked at from the point of view of its hydrogen consumption. For this reason, flowmeters are connected to the inlet and outlet of the stack. A mass balance on hydrogen can be used to determine the rate of its consumption in the cells, and the rate of electric charge passed may be calculated based on the ratio of two electrons liberated for each molecule of hydrogen.

Power generation can be calculated from this mass balance by knowing the Gibbs free energy of the hydrogen oxidation reaction, which ranges from -237 kJ/mol at 25°C to -228 kJ/mol at 80°C . The power calculated in this manner can be compared to that calculated from the polarization curve, and a practical efficiency of the stack is the ratio of the two. Additional references can be consulted for more information.³⁴

Experimental Procedure Equipment

In this experiment we used a BCS Fuel Cell Stack (18 PEM cells, 50 cm^2 active area each) with attached fans and a transparent plastic box to serve as chimney for the gas flows. The gas flowrates were measured using rotameters and the load was controlled using an HP 6060B electronic load box.



Figure 13: Hewlett Packard 6060B Electronic Load Box

The students submitted the following after completing the lab:

1. Summary of experiment.
2. Polarization curve:
Plot of average cell voltage and average power density (two y-axes) vs. average current density for the test where the current setting was varied.
3. Handwritten sample calculations to obtain cell voltage and current density from the measured stack voltage and total current.
4. Mass balance:
Calculate the hydrogen

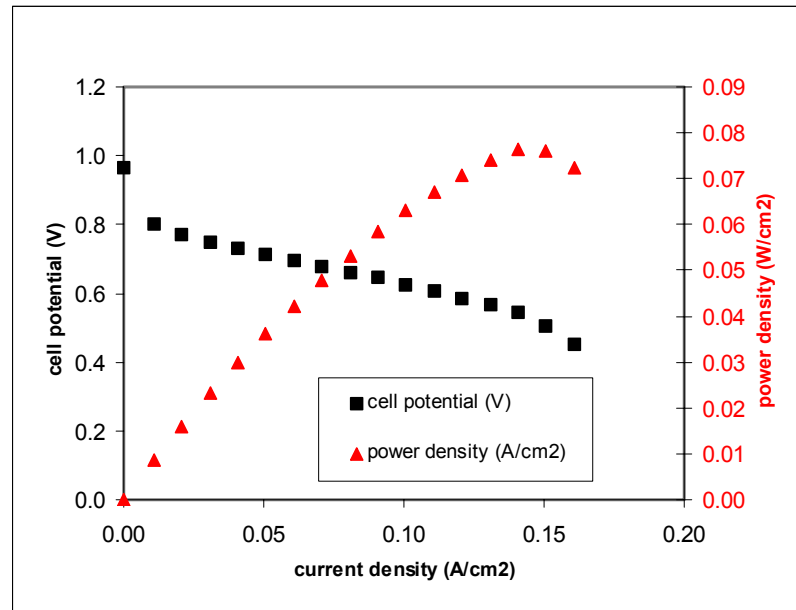


Figure 14: Polarization Curve from Fuel Cell Experiment

consumption rates for each current setting used in generating the polarization curve.

Use the downstream flowmeter readings for this, by comparing the current-flowing and the open-circuit-voltage conditions. Report the chemical energy consumed, based on the hydrogen flow measurements. Finally, calculate the pressure drop across the stack under open-circuit-voltage conditions based on the upstream flowmeter readings.

5. Efficiency: Consider the electrical energy calculated in plotting the polarization curve and the chemical energy calculated in the mass balance. Calculate the efficiencies of electric generation by the ratio of the two. Plot efficiency as a function of current density.
6. Speculate on the causes of any mismatch between the chemical and electrical energy calculations, and any departure of the polarization curve from what is expected.
7. Raw Experimental Data table.

An example of a polarization curve obtained from this experiment is shown in Figure 14. In Figure 15 an example of electrical efficiency is shown for the fuel cell stack.

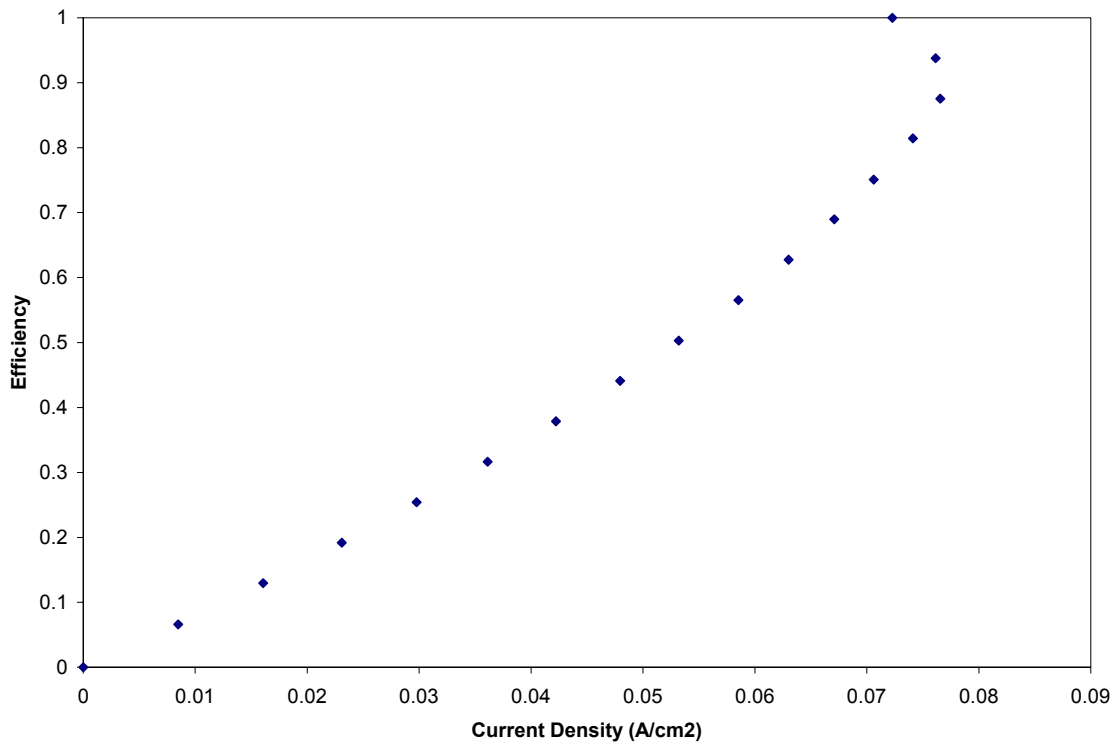


Figure 15: Electrical Efficiency of Fuel Cell Stack

Student Designed Experiments.

The final experiment conducted by the class was the experiments designed by students within the class. The following requirements were placed on this semester long design project:

Experiment Characteristics:

- **Timeframe:** The experiment should be performed by a team of students in one or two lab periods.
- **Economics:** The materials and supplies should be reasonably inexpensive.
- **Equipment:** If major equipment is required, an equipment description as well as vendor information and pricing should be included.
- **Analysis:** Use standard methods of analysis such as gravimetric, spectrophotometer, conductivity, etc. Keep in mind that this must be performed by students, not analytical chemists. Indicate the specific analytical method that will be used, and include a reference from literature.
- **Modeling:** The experimental data must be compared with a mathematical model. Fitting data to a correlation is also acceptable.

The Deliverables:

- A detailed laboratory handout, complete with introduction, objectives, theory & modeling, experimental methods, and results (including how to do data analysis). Hypothetical results, or results from literature should be used to illustrate how to present/analyze data.

- A list of materials and supplies, including vendor information and pricing
- A presentation

Examples of experiments submitted by the students are

1. Optimization of an Aluminum Air Fuel Cell³⁵
2. Fundamentals of Half Cell Reactions and Potential Determination
3. Electrodialysis of Salt Solutions in Food Products.

Summary

Overall the students enjoyed this course giving it a 4.69 out of 5. They enjoyed the experiments giving them a rating of 4.44 out of 5. Comments from the students included:

This paper discusses the use of the inductive style in teaching an electrochemical engineering course and gives examples of how traditional laboratories can be integrated with lecture material. In summary to convert a laboratory write-up to an inductive style the following should be done:

1. Handout a prelab given to peak the students interest. Have them hypothesize the trends in the data that will be collected.
2. The laboratory work should primarily consist of data collection and analysis using only graphical methods.
3. Discussion of the lab should take place in the classroom setting. Variable-parameter relationships should be identified.
4. Lectures on the variable-parameter relationships should be given.
5. Homework should be assigned based on the data taken in the laboratory.

In the procedure for an inductive presentation style of material starts with an experiment or shows results of an experiment and ends with the derivation of equations describing these results. We believe that this inductive style of teaching will result in large gains in the student's ability to synthesize and evaluate this material.

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BIOGRAPHICAL INFORMATION

Stephanie Farrell is Associate Professor of Chemical Engineering at Rowan University. She received her B.S. in 1986 from the University of Pennsylvania, her MS in 1992 from Stevens Institute of Technology, and her Ph.D. in 1996 from New Jersey Institute of Technology. Prior to joining Rowan in September 1998, she was a faculty member in Chemical Engineering at Louisiana Tech University. Stephanie's has research expertise in the field of drug delivery, and integrates pharmaceutical and biomedical topics and experiments into the chemical engineering curriculum. Stephanie won the 2000 Dow Outstanding Young Faculty Award, the 2001 Joseph J. Martin Award, and the 2002 Ray W. Fahien Award.

Robert Hesketh is a highly motivated professor in both undergraduate and graduate education and has received 9 education and 2 research awards, including ASEE's 1999 Ray W. Fahien Award. He has made major contributions in laboratory methods that demonstrate chemical engineering practice and principles. These highly visual and effective experiments, the most notable using the vehicle of a coffeemaker, are used to introduce engineering design and science to university and pre-college students. He has developed over 20 experiments employed throughout the curriculum at Rowan University. These experiments range from small scale coffee experiments to 25 ft distillation column experiments. His work has been presented at national meetings, workshops and published in journals and proceedings and his experiments are being used in over 15 institutions. He has attracted over 2 million dollars in external funding for research and educational activities.

C. Stewart Slater is Professor and Chair of Chemical Engineering at Rowan University. He received his B.S., M.S. and Ph.D. from Rutgers University. Prior to joining Rowan he was Professor of Chemical Engineering at Manhattan College where he was active in chemical engineering curriculum development and established a laboratory for advanced separation processes with the support of the National Science Foundation and industry. Dr. Slater's research and teaching interests are in separation and purification technology, laboratory development, and investigating novel processes for interdisciplinary fields such as biotechnology and environmental engineering. He has authored over 70 papers and several book chapters. Dr. Slater has been active in ASEE, having served as Program Chair and Director of the Chemical Engineering Division and has held every office in the DELOS Division. Dr. Slater has received numerous national awards including the 1999 and 1998 Joseph J. Martin Award, 1999 Chester Carlson Award, 1996 George Westinghouse Award, 1992 John Fluke Award, 1992 DELOS Best Paper Award and 1989 Dow Outstanding Young Faculty Award