ChE 402 Notes: Spring 2003

Our Senior laboratory is (or can be) one of the CAP STONE courses in your education in Chemical Engineering. It exposes you to a series of experiments that demonstrate the principals that you have learned in your classes. Each experiment reflects a combination of concepts that you have learned in different courses, e.g., thermo., mass balances, fluids, heat & mass transfer, kinetics, separations, control and design, etc. Specific examples such as polymerization, catalysis and bioengineering are represented in the experiments, which reflect the applications of Chemical Engineering in industry and research. You will learn to apply combinations of principals you have seen to real processes. You will find that there are often many practical differences between the idealized theories that you learned in class and the realization of these concepts. This is a crucial aspect to your education. The concepts are sound and correct. As an engineer, you are challenged to evaluate the application of these principals to complex processes.

As Seniors in your last semester, you may believe that you already have learned all that you need to succeed as Chemical Engineers, the last courses are just “icing on the cake”. However, employers are looking for Chemical Engineers who are multidimensional and know the applications and well as the limits to the basic concepts you have seen in your courses. They are looking for engineers that can bring together and extend these basic principals to real problems that they have. Further, you will need to communicate your studies to others in their organization. Our senior laboratory addresses these combined needs in concert and is, thus, a CAP STONE course of your chemical engineering education!

EXECUTIVE REPORTS: see attached

This semester you will submit “Executive Reports” in contrast to the more lengthy reports you submitted last semester. These are shorter and more to the point. Further, they can be easily summarized or extracted for circulation to upper management. Thus the conclusions and recommendation are up front. The reader proceeds progressively into the report depending on how much they need to know. An outline for executive reports is attached.

The most important aspect of an executive report is a reduction of B.S.: Where’s the beef? Do not “pad” your presentations or final reports with extraneous information. Focus on what you have done that is unique or represents differences from prior groups.

COURSE ADMINISTRATION

There are a few adjustments this semester in the teaching of the laboratory course. I am the only professor. This necessitates several additional constraints on the manner in which the laboratory is conducted so that you can gain the most out of your laboratory experience.

I applied several constraints in my setting up of the lab schedule. At most, there would be no more than five presentations or proposals on a given day (except the final day); otherwise, we would all get worn-out. The laboratory should end as early in the semester as possible, to allow you to finish your other courses and to enjoy your graduation.

Fortunately, we have four TA’s, which will be primarily responsible for 2-3 experimental stations each. The TA responsible for the experiment you are next to run should be consulted before you propose or run the experiment. They will also be available for final questions when you first run an experiment, during the first hour.
Several other modified requirements and operating procedures for this semester are outlined below.

**Preparation for Laboratory is Crucial!**
You must become familiar with the experiments and equipment before you actually enter the lab.
- Check out all the information on the website.
- Bring copies and be prepared to answer questions.
- Read the laboratory reports from prior groups!
- Find your references and read them.
  - You will be asked questions which you need to be able to answer!
- Know the Equipment and the software before you first enter the Lab.
  - Visit with the other groups in the prior session and ask questions.
  - Ask questions in the presentations and reports of the prior group(s).
  - Use the make-up days to ask questions of the lab techs (Les, Gary and Joe).

*If you are not familiar with the experiment and/or are not prepared, you will not be allowed to perform your experiments and 10% will be deducted from your grade on that experiment for each day you fail to be prepared.*

**Analyses Throughout the Laboratory**
You should not collect four sets of data for the four periods that you will be in the laboratory for a specific experiment and then analyze these data the night before your reports are due. You might have done this in the past, *but not now!* Before you come into your third period for a laboratory, you will need to have analyzed the results from your first two sessions. You will be required to show evidence of these preliminary analyses, including graphs of your data. The analyses do not need to be complete, but, they must be sufficiently detailed to determine if the experiments are running well and if you should be able to meet your stated objectives. Otherwise, we can modify the objectives and/or the experiments to be performed for the last two laboratory sessions. I will be available to discuss any potential changes during my office hours.
*You should take advantage of this!*

*If you have not analyzed the results from your first two sessions prior to your coming into your third session in the laboratory, you will not be allowed to perform your experiments and 10% will be deducted from your grade on that experiment for each day you fail to be prepared.*

The purpose of this requirement is to assure that you accomplish a meaningful set of objectives in your studies. These objectives can be modified due to unanticipated factors: experimental glitches or over-ambitious proposals. Ongoing analyses of the data will enable you to synthesize these into pragmatic objectives. Otherwise, you might find that you have accomplished little due to problems that occurred early-on but were not seen until the night before your final presentation.
INSTRUCTIONS FOR THE LABORATORY TECHS ARE REQUIRED!

Fill out preparation sheets for each day in the laboratory and submit them by 3:30PM of the lab day before you will be in the lab: e.g., Monday before Wednesday in the Laboratory or Friday before Monday. Make sure the instructions are dated when submitted and include instructions for specific days.

PRESENCE IN THE LABORATORY DURING ASSIGNED PERIODS.

You will have four periods in which to accomplish your studies. These start at 12:30 and end at 4:30. You are to be in laboratory during these periods unless you have an excused absence. Requests for these are to be submitted by email at least 24 hours in advance and/or doctors notes should be provided if for health reasons. For interview trips, you are required to inform me and your fellow group members a week in advance. If you miss laboratory with out prior approval or a doctors note, you will be penalized an additional 10%.

It is essentially impossible to finish your laboratory studies in fewer than four periods! The group cannot skip a laboratory or leave laboratory early (say before 4PM) and will be appropriately penalized. The unusual exceptions to this would be if the group is involved in detailed analyses such as running the Instron or GPC for the polymer laboratories. Alternately, you might be conducting a simulation; although, it is hard to envision that this could best be accomplished during the laboratory period. These will only be allowed if you have prior permission to do so.

SAFETY ISSUES

You must operate in the laboratory in a safe manner. You had some safety training last semester. Those of you that did not must complete the Laboratory safety and hazardous waste training immediately, before you participate in the laboratory. This is available through the UMASS web site for EH&S. You must wear your safety glasses in the laboratory or you will have to leave the laboratory. The TA’s have been instructed to assure compliance with this and any other safety issues, including dangerous or inappropriate behavior in the laboratory. You are dealing with equipment that can be operated in a manner that endangers you and all the others in the laboratory and we must insure that this does not occur. In industry, you will readily lose your job if you do not observe the safety requirements while you are in the laboratory. SAFE forms are required for each experiment and need to be prepared before you first enter the laboratory.

COMMENTS ON PRESENTATIONS

The presentations will be on Powerpoint® employing the computer in the conference room. These should be limited to 15 minutes + discussion. The presentations should start with an outline by which you let us know what is coming and encourage us to pay attention to certain points in your presentation. The focus will be on the new aspects of your studies, the new theory behind it and a discussion of the relations between what you intend to do (or have done) and the theory. You should end with a conclusions slide(s) where you summarize your accomplishments or the approach you will be taking. A recommendations slide will be used at the end or a report and a safety slide at the end of a proposal. Do not use too many words on each slide, abbreviate and then explain. Use variable font sizes and colors to make the slide interesting and focus our attention on the key words or concepts. A copy of your slides (4-6 slides/page) will be presented to me after your presentations.
COMMENTS ON OTHER GROUP-, SELF- AND PEER- EVALUATIONS

Group leaders must send in evaluation for others by email (to Hari) on the next day or give them to me or Hari after the presentations. I will track those other group leaders and a 5% decrease in your personal evaluation will be assessed for late reports, 10% for no reports! If you are excused (at least one day before the presentation or with a doctors note and email before) you will be responsible to designate another group member to fulfill your duties.

Self-evaluations for both the proposals and final oral presentations are due at the same time as are the final report. These should be given to me or sent by email. Penalties for not providing these in a timely fashion will be assessed, as above. These are personal penalties and will not affect the grade for other group members, but they can be significant.

Peer evaluations are required from all group members for each experiment and are due at the time of the final report. These are to be given Again, similar penalties apply for lateness or failure to submit these peer evaluations. I will, however, accept individual statements that “Each member of the group Y(A->I) contributed significantly and equally to the conduction and report for experiment X (1->10) submitted on (date).” Alternately, I would accept a statement such as, “The conduction and report for experiment X (1->10) submitted on (date) was primary the product of group members X, Y and Z while the contribution of Q was (significantly) less.” These must be submitted with the final report or submitted by email at the same time (or before) the report is submitted.

Comparison with Prior Experiments:

All groups must be familiar with the results of prior groups for the same experiment. This is crucial as you must present these in your proposal and will compare your results to theirs in your report. You do not necessarily have to accept their results and their interpretation in total; indeed, alternate interpretations for prior results will be awarded extra credit if they are reasonable and supported by your reasoned analysis. This will not result in a re-grading of the prior reports by the earlier groups (unless you demonstrate that their results were improper).

PROPOSED EXPERIMENTS

Your are to choose and defend a proposal for your research. This differs from last semester where you were given a set of requirements. I welcome and hope (read this as you shall) you discuss your proposal with me (and others, as noted) before you present it to all of us. There will be several options and you need to propose enough to keep you busy for four lab periods but not too much. You will be productively working in the laboratory for four periods. In all cases you must relate your results to those from prior groups of which you need to be familiar before your proposed studies.
Experiments

1. Characterization & control of a heat exchanger  
   **HeatEx**  
   TA: Hari
   
   The focus for this semester is control. One is first to measure the process dynamics and then to propose a control scheme. There are several options for the control and you are to choose one. These options involve control of any of several temperatures (of the process or cooling water at various points) employing any of several variables (steam, cooling and process water). The process can be operated in co- or counter-current flow modes for the water-water heat exchanger. At first simple PID control will be employed. Simulation should be employed to represent your results (with and without control) as soon as you can. You should test the estimates of the best control constants (from any of several approaches in your control text). Show us the practical limits of the various constants and the consequences for being outside these limits. Demonstrate the differences for P, PI and PID control for the system you propose.

2. **Fermentation**  
   **Ferment**  
   TA: Gupta
   
   The fermentation laboratory will grow from last semester. Professor Forbes has agreed to advise you on the series of experiments this semester and Sarwat Khattak (skhattak@ecs.umass.edu, office 577-0134) has also agreed to provide some advice as she was TA last semester. There is a new manual for this that was assembled by Professor Roberts (who will have little time this semester). Two directions that may be taken are to investigate the use of dry yeast for the growth process. This might give a faster growth kinetics, cutting down on the reaction time. The second is to optimize the production of alcohol, which involves two steps: growing the yeast and then having the yeast produce ethanol from sugar. The first process is aerobic while the second is anaerobic. You have several choices as to how you wish to run the reactor to produce the most alcohol in the shortest period of time. You will need to test and understand the changing kinetics of growth and ethanol production to determine the optimum mixing, feed and temperature protocols for this.

3. **Ion Exchange**  
   **IonEx**  
   TA:Aadil
   
   The Ion exchange experiments will compare a variety of ion-exchange resins for the removal of Copper Sulfate from an aqueous solution. There will be four new resins available, all strong acid cation resins produced by Dow. Two of these represent different particle sizes for the same resin that was studied last semester. Another represents a different degree of cross-linking. And, finally, there is a product called, “Marathon”. Each new group is to choose a resin to compare to the prior results and to show and explain the differences. It is obviously required to understand the differences based on their composition and morphology, as extracted from DOW. One will need to understand the basic theory as it relates to the potential differences. Finally, you will compare your results to the theory and to the results from prior research groups. The theory and the data from prior studies will be part of your proposal.

4. **Membrane Separation by Permeation**  
   **Perm**  
   TA: Hari
   
   The Permeation experiments studies the enrichment of oxygen from binary and tertiary gas mixtures flowing in series or in parallel into two permeation bundles. Former groups have investigated separation of $\text{O}_2$ from $\text{N}_2$ and $\text{Ar}$. You will have mixtures of $\text{O}_2$ in $\text{He}$ and $\text{O}_2$ in $\text{CO}_2$ as well as ternary mixtures of $\text{N}_2/\text{O}_2/\text{CO}_2$ and $\text{N}_2/\text{Ar}/\text{CO}_2$, often with different mole fractions. You will propose to compare the enrichment of oxygen as a function of flow-rate/back-pressure.
from different feeds and to compare these to prior separations (employing different feed compositions).

5. **Polymer Injection Molding**  
**InjMold**  
**TA:** Aadil

The obvious purpose of polymer injection molding is to produce a part (plastic dogbone or spiral) with certain properties. These properties are subject to testing by an Instron mechanical testing instrument (Ramas Sankaranarayanan in PSE will run these, coordinated with Ramon) or to other tests you can devise. Normally, the goal is to produce the strongest part but it could also be to produce a part that breaks within a given region of force. There are several variables that can be manipulated in this process that change the strength, uniformity and/or mechanical performance of the final part. These are ideally suited to experimental design to optimize this process. We will have three polymers to choose from for extrusion. You will study one of these in contrast to the other polymers to be studied by other groups.

6. **Binary Distillation**  
**Distill**  
**TA:** Gupta

This semester the focus is on distillation dynamics leading to control of the column. The primary control variables are the steam into the reboiler and the reflux flow. The dependant variables are the bottom and product compositions, respectively. Several variables dictate the dynamics and eventual control of product compositions. There are logical combinations control and manipulated variables: steam-bottoms or reflux-top. It is crucial that you present a proper energy analysis of this system. There are several options in the analysis of energy, depending on where you draw the boundaries. All involve measuring the rate of steam condensing in the reboiler. We will have a sight glass to monitor the level of liquid at the exit of the reboiler and you will need to work out a protocol to quantify the energy transferred from the steam.

7. **Methanation in a Fixed Bed Catalytic Reactor**  
**CatReact**  
**TA:** Ramon

The initial experiments last semester were flawed in that temperature gradients were found to be present at high flow rates. This semester we will at first employ He as a carrier gas to improve the heat transfer (the first group should explain and quantify this). Eventually, we hope to have a new reactor in place which will improve heat transfer before the catalyst bed. Finally, the last two groups may synthesize their own catalysts for methanation and test these compared to the commercial catalysts. The general kinetics (L-H kinetics), including activation energies, the regions for diffusion control, and catalyst deactivation (and regeneration) can be studied. There are several kinetic simulation packages for catalytic reactions that may be employed to represent this plug flow catalytic reactor.

8. **Polymerization Kinetics**  
**PolyKin**  
**TA:** Ramon

You will have options for both the monomer and initiator in this semester. Butyl-Methacrylate is the optional monomer while Benzoyl peroxide is the optional initiator. The same slate of solvents are available to you that were employed last semester, i.e., bulk, or in toluene, butanol, methanol, benzene and/or cyclohexane. In all cases, you are to focus on the differences between the results you find and those of prior studies in our labs or in the literature. In the case of literature results you will most probably confirm their findings. Differences in the kinetics should be studied; specifically, the activation energies will differ for certain optional changes. In other cases, the differences will be most reflected in the nature of the polymer product that is
produced. For polymer products, the first measure is the molecular weight distribution, which will be measured with GPC in the PSE department (you will need to understand the technique). We will work a proto-call whereby this characterization data can be obtained for a limited number of samples (e.g., < 8 in two sets of four). You should be familiar with those kinetic and mechanistic components that control the molecular weight distribution. These experiments are typical in the development of a new polymer product(s) wherein the range of reaction variables are studied to optimize a polymer for specific applications based on the polymer properties, MWD being the first.

9. **POLYMER EXTRUSION**
   **Extrusion**
   **TA:** Aadil
   The purpose of polymer extrusion is to produce a continuous strand of polymer of uniform dimension from pellets. Several dies of varying dimension are available. The conditions throughout the extruder control the nature of the product strand. This experiment should first employ experimental design to optimize the production process and its influence on dimension and uniformity for different die sizes. You will have three choices of polymer and will need to characterize the product (dimension and uniformity). A protocol should be developed for this. Die-swell can be a complicating factor for certain polymers under specific conditions. We may be able to develop access to DTA, differentia thermal analysis, to characterize differences in the polymer crystallinity which will depend on process conditions.

10. **TWO-PHASE FLOW**
    **2PhaseFlow**
    **TA:** Gupta
    The two-phase flow experiment involves air and water flowing co-currently through a packed column, packed with glass beads to disperse the flows. Such a system is often employed to scrub plant effluents (removing pollutants). You can independently vary the flow rates of the two fluids and as you do so the nature of the contact between the two phases changes from trickling to pulsing flow. Pressure transducers are placed throughout the column to measure the pressure drop and variations in pressure with time. These periodic variations in time can further be subject to Fourier analyses to characterize the nature of the pulsing flow. You will explain this to the rest of us. Finally, an ethylene glycol and water solution will be employed to vary the liquid viscosity and to determine how this changes the contact between the two phases, the nature of the flow regimes and the pulsing behavior.