Topical 5: Pilot Plant Design and Optimization (T5)

#161 - Pictures of Pilot Plants and University Unit Operation Labs (T5001)

PAPER 161c -- The Ohio State University -- Chem Engr Ug Teaching Lab
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Annotated List of Unit Operations in UG Chemical Engineering Lab for ChBE-630

The Ohio State University – Department of Chemical and Biomolecular Engineering
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E-1 -- Plate & Frame Heat Exchanger

The objective of this investigation is to determine the effects of several operating conditions and process variables on the heat transfer between two fluids in a plate heat exchanger.

Plate heat exchangers are an important choice for industrial heat transfer when the process fluids are subject to fouling, are easily contaminated, and especially if there is a relatively low temperature difference. The system examined will operate at steady-state. Some analysis will be applied to a system in a dynamic mode which resembles heat transfer applications, such as cooling batch reactors, which are increasingly prevalent in the chemical, plastics, food, and pharmaceutical industries.

The hot and cold process fluids for investigating the plate heat exchanger are city water and steam heated city water. A direct steam injection water heater is used to provide warm water for the hot process fluid. The hot water is cooled by city water in a plate heat exchanger. Depending on the plates installed a plate exchanger can operate in co-current or counter-current flow with single or multi-pass configuration. The specific unit has plates installed that are set for single pass operation but the flows can be configured to be either co-current or counter-current.

E-2 -- Shell & Tube Heat Exchanger

The objective of this investigation is to determine the effect of flow rate and flow configuration on the performance characteristics of a shell and tube forced convection heat exchanger.

The general purpose is to construct a correlation of observed heat transfer performance as a function of flow, properties, and configuration. Correlation are expressed as the Nusselt number or Colburn j-Factor versus Reynolds number and possibly the Prandtl

number. Since the latter quantity may not vary by much, the students consider applying a widely accepted form of dependence. Along with heat transfer, the students consider the effect of pressure drop, perhaps in terms of friction factor versus Reynolds number.

One method of correlating data is through a Wilson plot, in which data are taken by varying the flow rate of one fluid at a time. From each set of experiments, $1/U_o$ is plotted versus Re_F^{-a}, where "F" refers to the fluid for which the velocity was varied, and "a" is an exponent to be determined by nonlinear regression. If a reasonably consistent value of "a" is obtained for a range of flow rates (all in the same regime), the "other" film coefficient can be determined from the intercept of the lines obtained at different, but constant values of that fluid's flow rate.

A process fluid, water, is to be heated in a U-tube heat exchanger by condensing steam on the shell side. Steam condensate will be collected and weighed. The heated water (i.e. process fluid) is cooled by cold water in a shell & tube heat exchanger.

The heated process fluid is to flow on the tube side while the cooling water will flow on the shell side. The process fluid flow will be measured by a calibrated rotameter located on the instrument board near the centrifugal pump unit. The cooling water flow is measured by a calibrated rotameter located on the front side of the shell & tube heat exchanger.

E-3 -- Liquid-Liquid Extraction

Acetic acid (5 wt%) is removed from n-hexanol with water in a Karr reciprocating-plate column.

The objective of this experiment is to determine the effects of operating variables on the performance parameters of a pulse-plate column (Karr column) for the extraction of acetic acid from *n*-hexanol into water. Operating variables include (but are not limited to) feed and solvent flow rates and pulse amplitude.

Agitation is essential in the design of any liquid-liquid extraction equipment, in order to achieve high mass transfer coefficients and to reduce the size of the drop in order to get a higher ratio surface/volume, again to increase the mass transfer rate. Even though this can be done by mechanical agitators such as rotary stirrers, the amount of agitation must be controlled carefully because excessive agitation causes that the size of the drops of one of the liquid phases becomes so small that it can be dragged by the countercurrent flow of the other liquid phase, causing flooding of the column

The process variables to be studied in this experiment are water and hexanol flow rates and the pulse frequency. The experiment includes four runs. The first two vary inlet water flow rates, the third one involvse variation of the agitator frequency and the last one involves change in the flow of hexanol. All subsequent calculations and conclusions are made from these runs. Additional runs can be performed in the event that the students are unsure of your results or want to establish reproducibility of the performance of the column, as time permits.

E-4 -- Continuous Distillation

15 wt% Ethanol in water is separated in a 12-tray single-bubble cap glass column. Trays are teflon.

The objective of this investigation is to determine the effect of operating conditions (feed location and reflux rate) on the separation efficiency of a twelve-tray bubble cap distillation column using an aqueous ethanol feed.

The equipment used in this experiment is a twelve-tray bubble-cap distillation column (plus a reboiler) with liquid sampling capability. Ethanol composition analysis will be performed using the IR meter and balance. The material used in the continuous distillation experiment is an aqueous ethanol solution (about 10-30 wt% ethanol).

In the chemical and petroleum industries, distillation is the most common separation process. The production of most liquid products requires distillation, either for a precursor or the product itself. Therefore, an inherent understanding of distillation is important for chemical engineers. The purpose of this experiment is to provide you some hands-on experience with distillation. Furthermore, since the distillation column is glass, the students can actually see the operation in action and acquire a better understanding of column operation. In this experiment, the feed can be located on one of three trays and the reflux ratio can be adjusted. The students study how changing the feed location and adjusting the reflux ratio affects product purity and overall efficiency of the column.

The students investigate the effects of changing the feed tray location and reflux ratio on the column efficiency. The feed can be introduced on trays 3, 6, or 9 and the reflux ratio can be set anywhere between 0.4 and 1.5 or so. Five sets of data will be taken: feed to each tray with a constant reflux ratio (this is the first three data points), and then keep the feed on only one of the trays and take two sets of data with different reflux ratios (the last two data points). The major goals are to determine the overall column efficiency and the minimum reflux ratio required to perform this particular separation.

E-5 -- Continuous Stirred Tank Reactor (CSTR) Engineering

This experiment investigates the rate of a pseudo-first order reaction, the hydrolysis of acetic anhydride, which forms acetic acid in two reactors having different characteristics. Part A studies the reaction in a batch reactor and part B studies the reaction in a continuous stirred tank reactor. Reaction and kinetic theory learned in the Reaction Engineering and Chemistry courses can be tested by setting up different experiments using both the batch and continuous reactors.

PART A - Batch Reaction

The purpose of this experiment is to understand the effects of several operating conditions on the rate of reaction, rate constant, and activation energy during the hydrolysis of acetic anhydride. The overall goal is to determine the rate and propose explanations for the values found. The concepts covered in this study are important in order to satisfactorily design equipment to effect reactions on a commercial scale.

The experimental goals are to determine the extent of reaction at various times by measuring the concentration of a given component as a function of time. This will be accomplished by monitoring the pH of the solution as the reaction proceeds. Temperatures should range from 0°C to 30°C (running experiments twice near each extreme should be quite effective). One experiment should also be run in the middle of

this range. Thus, not less than five experiments are to be run. Note that the volume of water should be much greater than the volume of acetic anhydride in order for the reaction to be pseudo-first order (approximately 800:1).

This experiment determines the rate of reaction and the reaction rate constant at various temperatures. An Arrhenius plot is made in order to determine the activation energy of hydrolysis of acetic anhydride.

PART B - Continuous Reaction in Continuous Stirred Tank Reactor

The purpose of this part of the experiment is to determine the effect of reactor size, inlet flow rates and inlet concentrations on the outlet concentration during a pseudo-first order reaction in a continuous stirred tank reactor (CSTR). This may be studied simultaneously with the batch reactor, where the rate of reaction, rate constant and the activation energy will be studied for the same reaction.

The reaction to be studied is the hydrolysis of acetic anhydride. The initial acetic anhydride concentration will be low, to simulate a pseudo-first order reaction. The water to acetic anhydride should remain between 800:1 to 1,000:1. With these ratios, the reaction can be studied as a first order reaction with the concentration of acetic anhydride being rate-limiting. The reactor can be run with three different volumes and infinitely variable flow rates and inlet concentrations. The water flow rate can range from 0.8 to 5 L/min. and the acetic anhydride flow rate can be varied from 2 to 24 mL/min.

E-6 -- Plug Flow Reactor (PFR) Engineering

The objective of this experiment is to investigate the effect of operating variables on the performance parameters of a plug flow reactor for a pseudo-first order reaction.

This experiment investigates the rate of a pseudo-first order reaction, the hydrolysis of acetic anhydride, which forms acetic acid in two reactors having different characteristics. Part A studies the reaction in a batch reactor and part B studies the reaction in a plug flow reactor.

PART A - Batch Reaction

The purpose of this experiment is to understand the effects of several operating conditions on the rate of reaction, rate constant, and activation energy during the hydrolysis of acetic anhydride. The overall goal is to determine the rate and propose explanations for the values found. The concepts covered in this study are important in order to satisfactorily design equipment to effect reactions on a commercial scale.

The experimental goals are to determine the extent of reaction at various times by measuring the concentration of a given component as a function of time. This will be accomplished by monitoring the pH of the solution as the reaction proceeds. Temperatures should range from 0°C to 30°C (running experiments twice near each extreme should be quite effective). One experiment should also be run in the middle of this range. Thus, not less than five experiments are to be run. Note that the volume of

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This experiment determines the rate of reaction and the reaction rate constant at various temperatures. An Arrhenius plot is made in order to determine the activation energy of hydrolysis of acetic anhydride.

PART B - Continuous Reaction in a Plug Flow Reactor

The purpose of this part of the experiment is to determine the effect of reactor size, inlet flow rates and inlet concentrations on the outlet concentration during a pseudo-first order reaction in a plug flow reactor (PFR). This may be studied simultaneously with the batch reactor, where the rate of reaction, rate constant and the activation energy will be studied for the same reaction.

The reaction to be studied is again hydrolysis of acetic anhydride. The initial acetic anhydride concentration will be low, to simulate a pseudo-first order reaction. The water to acetic anhydride should remain between 800:1 and 1,000:1. With these ratios, the reaction can be studied as a first order reaction with the concentration of acetic anhydride being rate-limiting. The reactor can be run with infinitely variable flow rates and inlet concentrations. The water flow rate can range from 0.4 to 4.5 L/min. and the acetic anhydride flow rate can be varied from 1 to 20 mL/min..

E-7 -- Gas-Solid Fluidization

The objective of this experiment is to determine the basic properties which control the flow regimes of a solid and fluidized bed and develop an understanding of the principles of fluidization.

Fluidization phenomena include air flow through both packed beds and fluidized beds, as well as the pressure drop experienced by the fluid flowing across the beds. An appreciation for bed porosity, minimum fluidization velocity, minimum bubbling velocity, and terminal velocity will be gained. Also, the major flow regimes of fluidized beds, their industrial and experimental uses will be studied.

The main experimental objectives for this lab are to study fluidization of solids having different particle sizes and densities. The various things to be studied include, but are not limited to, pressure drop in a packed bed and then a fluidized bed with height of the bed, minimum fluidization velocity, minimum bubbling velocity, and terminal velocity. It is important to observe the difference in pressure change for increasing fluid flow as compared to decreasing fluid flow. Lastly, it is important to make very careful visual observations in order to compare the experiment to theory.

E-8 -- Gas-Liquid Column Dynamics

The objective of this experiment is to investigate the hydrodynamics of a gas-liquid system. The student's investigation constitutes the determination of phase holdup (or volume fraction of each phase) at different gas velocities. They also investigate the different regimes in a gas-liquid system.

Several industrial operations involve gas-liquid systems. Bubble columns hold several advantages over other reactors for gas-liquid operations. With no moving parts, maintenance of the bubble column is simplistic. Bubble columns also offer excellent heat and mass transfer. Finally, bubble columns give great gas-liquid contact. For those reasons, they are heavily used in industry for reaction and mass transfer operations. Moreover, slurry bubble columns employ micron-sized catalysts suspended in a liquid medium for three-phase catalytic reactions.

Absorption, fermentation, bio-reactions, coal liquefaction, and wastewater treatment are just a few examples that involve bubble columns.

Absorption is an obvious, self-explanatory use for bubble columns. Fermentation and bio-reactions fall under the category of gas-liquid reactions. Uses for bubble columns include the fermentation of acetic acid for wine vinegar production, production of cellulase, and lipase. Coal liquefaction converts coal into useful liquid products such as diesel and gasoline. Sasol, the main employer of coal liquefaction, uses slurry bubble columns to convert coal into liquid fuels. Bubble columns are used in wastewater treatment for removal of contaminants.

E-9 – Absorption – Ammonia from Air

The objective of this investigation is to determine the effects of gas and liquid mass velocities on the performance characteristics of the absorption of ammonia from air into water in a column packed with glass Rashig rings.

Absorption in a packed column represents virtually all of the important phenomena of gas-liquid contacting in a "differential" column, as opposed to a "staged" column. It also, of course, exhibits the relevant phenomena of absorption, and is closely related to stripping. The column installed in our lab is made of glass and is packed with glass Rashig rings. Though most columns in commercial applications are made out of metal, it does allow full view of flow distribution, flooding, entrainment, and liquid retention in the base of the column.

The purpose of this experiment is to study countercurrent flow of a gas and liquid in a packed column, and mass transfer and equilibrium effects that govern absorption of ammonia from air by water. The phenomena to be studied include, but are not limited to, pressure drop in a the packed bed as affected by both fluid flow rates and other conditions, flooding, mass and energy balances, and performance of the packed column as a mass transfer device. It is important to make careful visual and quantitative observations in order to compare the experiment to theory.

The equipment is comprised of a column with a diameter of 150 mm and packed to a height of about 1.3 meters with 10 mm diameter Rashig rings. Both the column and packing are made of borosilicate glass. For both the water and air two flowmeters are provided. You must be careful not to operate these out of their respective ranges or damage could occur. In particular, the low-level water flowmeter can handle only about 8 liters per minute (2.1 gpm).

E-10 – Adsorption – Acetic Acid on Carbon

The objective of this investigation is to determine the effect of particle size to column diameter ratio, feed rate and inlet concentration on the capacity and break-through curve characteristics of a granular adsorbent.

Adsorption is a rapidly growing operation that has the capability to purify liquids and gases and perform bulk separations. Cyclic adsorption combines the steps of uptake and regeneration and is typically conducted in a fixed bed with multiple identical columns in parallel. The objective of this experiment is to study the uptake step. In particular, the experiment may investigate the effects of feed concentration, flow rate, particle size, and/or column diameter on the capacity and break-through characteristics of commercial adsorbents. These experiments may include ranges of those variables in which other conditions are carefully controlled.

E-11 -- Reverse Osmosis

The purpose of this experiment is to determine the effects of several operating parameters and conditions (feed pressure, pressure differential across the membrane, salt content and salt type) on the performance of a reverse osmosis (R.O.) system.

A broader goal is to gain appreciation of membrane-based separations, which have made a dramatic impact on the chemical, food and beverage, paper, petrochemical, metal finishing, and pharmaceutical industries over the past 25 years.

The student is given a design extension problem assignment sheet at the Preliminary Meeting. It will outline a specific application for which a R.O. system is needed. The student team tailors the experimental program to ensure the theory (or empirical correlation) will be adequate for the specified conditions, and to accommodate the design data needs. Important variables are likely to be: feed concentration, pressure, flow rate, and temperature, product split (i.e. the ratio of permeate to retentate flow rates), permeate pressure, and permeate salt concentration. Some of these can be controlled; others depend strongly on other variables.

E-12 -- Batch Filtration

The objection of this experiment is to learn to operate a batch filtration unit to separate $CaCO_3$ solids from slurry formed by the reaction of $CaCl_2$ with $NaCO_3$. The student is required to: 1) Study the batch filtration process and the effect of pressure drop across the filter medium and filter cake on the operational performance and 2) Use the experimental data to determine the filtration parameters relevant to your system.

The following scenario is used to guide the investigation:
A plant has a battery of batch filtration units in operation. The Company's Engineering Department proposes to run a series of tests on one of them to determine the performance of this type of equipment under varying conditions. The data will be used as a basis for design of the proposed doubling of capacity of this plant. The present capacity is 2000 gallons of slurry per day.

There is available in the Development Department of this plant a small size batch filtration unit essentially the same type that is currently used in the plant. It is the thought of the engineering department that it might be possible to obtain a correlation between the small size filter in the Development Department and the filter in the actual plant

operation. If such a correlation is possible, it would have the following effects: (1) cut down the number of large scale tests to a minimum thereby disturbing plant production as little as possible; (2) afford a simple means of predicting and determining the optimum conditions for filtration on the units in operation; (3) permit the obtaining of data for predicting the effects of changes in operating conditions with the minimum amount of nuisance to the operating department, and (4) serve as a basis for the design calculations in future plant expansions.

E-14 -- Mixing Dynamics

The objective of this experiment is to determine the effects of tank geometry, baffle position and dimensions and Reynolds Number on the power number and blend number.

The student team is asked to investigate the design criteria for a mixing operation. The students are to consider themselves as part of the new design team. One of the first duties is to design the agitation system for other divisions within the company. It is not certain what type of agitators or what sizes of motors to buy.

The Lab is divided into two sections. The first section deals with viscosity measurement and the second with impeller characterizations.

Viscosity Measurement

A Fanning Couette viscometer is used for the viscosity measurements. The students are required to compare the measured values of viscosity of a glycerol/water mixture to established values. Use six data points for this comparison using 75% and 15% glycerol/water mixtures as the end points (i.e. 75% wt% glycerol in water). As glycerol is somewhat expensive, the students are not able to mix new batch for every measurement. Instead, they start with the 75 wt% and dilute the mixture with water each time they want a new data point, ending with the 15% solution. For each data point the students make four measurements, one each at 100, 200, 300 and 600 RPM's.

2) Impeller Characterization

In order to determine the optimum design for the agitation units, several parameters must be studied. Listed below are the operating conditions to be optimized.

Determine the relationship between the liquid (baffled) height and diameter, Z/T. Using the same diameter tank, vary the height of the liquid in the tank. Use 4 different Z/T values to determine the optimum. Use 3 different RPM values. Use the same impeller for all these tests.

Determine the optimum ratio of clearance of the impeller from the bottom of the tank (baffled) to the tank height, C/Z. Using the same impeller, vary the clearance of the impeller from the bottom of the tank. Use 4 different C/Z values for 3 different RPM values.

Determine the effect of impeller design. Use 4 different impellers in the same tank. Select a mixture of axial and radial flow impeller types. Use 5 different RPM values for each impeller. The baffled tank is used.

Determine the effect of baffles by operating with and without baffles. Use 5 different RPM values. Use the same C/Z and Z/T values as in part C and choose one kind of impeller used in part C. Use the same impeller for all these tests.

Determine the effect of a different viscosity. Compare a solution of water with a solution of 50/50 wt% water glycerol. Use 5 different RPM values and the same impeller for each viscosity. Pick an impeller that will demonstrate the difference clearly.

Optimum operation condition to be studied for now are: a) the relationship between the height of liquid in the tank and the diameter of tank, b) the optimum ratio of clearance of the impeller from the bottom of the tank to the tank height, c) the effect of impeller design, and d) the effect of a different viscosity. Be certain that the parameter which is being studied is the only parameter which is being changed. For example, if you are studying the impeller characteristics, do not change the clearance of the impeller for each trial. The set of 5 RPM values could be 150, 225, 300, 425, and 500+. The set of 3 different RPM values could be 100, 250, and 500+.

E-15 -- Fermentation

The purpose of this experiment is to gain experience operating a fermentor on the 20L pilot plant scale to produce yeast biomass and to analyze the fermentation performance.

Chemical engineers can perform in a broad range of industries and careers in the bioprocess industry are expanding in popularity. One of the most common and important components of the bioprocess industry is fermentation. The production of Baker's Yeast in an aerobic fermentation process will be studied here. Baker's Yeast is actually the yeast Saccharomyces cerevisiae. The importance of Baker's Yeast in the bakery and alcohol industries is well established, recently, the demand for Baker's Yeast is recently expanding into other industries such as the nutraceuticals and homeopathic medical industries.

The process of growing or culturing of Saccharomyces cerevisiae biomass has many components to be optimized. Some important factors are the media composition, culturing temperature and pH, aeration and oxygen transfer rates, reactor type, and contamination. The media used for culture should include sources of carbon, phosphorus, nitrogen, trace minerals, and growth factors. The carbon sources are carbohydrates, which are also used for energy by the yeast. Example of carbon sources are glucose, sucrose, xylose, and dextrose. To supply the remainder of the required nutrients, each component can be added individually in known amounts or a complex substance can be used that provides all nutrients but in variable amounts. Examples of complex additives are yeast extract, cottonseed extract, and molasses. Saccharomyces cerevisiae has two metabolic pathways to utilize the carbon source: aerobic and anaerobic. In the aerobic pathway, the carbon source is utilized in the TCA cycle to produce energy, biomass, and carbon dioxide in a very efficient manner. Therefore, oxygen should be fed to the reactor to aerobically produce yeast biomass. If no oxygen is supplied, the yeast will use an anaerobic pathway to produce ethanol. This is an inefficient use of the carbon source and biomass growth will be inhibited. If the carbon source concentration is too high however, the yeast will activate both pathways, producing ethanol and biomass. This, too, is inefficient for optimal biomass growth. If the original carbon source diminishes, any

ethanol produced will be used as an alternate carbon source for limited growth. The agitator blade size, configuration, and speed are important factors in the supplying of oxygen too.

The operating temperature of the fermentor influences the growth of the yeast. At 32oC, the yeast grows at a maximum growth rate, but at 28.5oC, the highest yeast biomass yield is achieved. The operating pH range should be between 4 and 5.5. Low pH conditions help to inhibit contamination growth, while higher values of pH in that range yield a high quality product. The fermentation can be performed in different reactor set-ups. This experiment utilizes a batch process whereas a fed-batch fermentor, in which fresh media is supplied either in pulses or continuously, is most common in industry.

The success of fermentation, and most biological processes, depends greatly on the sterilization process. Sterilization of the reactor and media is essential before proceeding with an experiment to ensure that almost all of any contaminating microorganisms are killed. Sterilization is performed at 121oC and 15psi for a determined period of time. The sterilization time is calculated to ensure that the microorganism population is diminished to an acceptable level. Browning reactions and nutrient degradation are issues to consider when determining the sterilization time. Browning reactions can be avoided by sterilizing some components of the media separately and combining them afterwards. Sterilization can be done in an autoclave for smaller objects and/or on-site inside the reactor, if possible