



Chemical Engineering Laboratory II (ChE 414.2)

Laboratory Manual

**Department of Chemical
Engineering**

2011-2012

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COURSE INFORMATION

Course objectives

In this course, experiments are chosen from the fields of biochemical engineering, unit operations and mass transfer. Students will collect and analyze the experimental data using the theoretical principles of heat and mass transfer, unit operations and biochemical engineering. Additionally, students will learn to conduct a laboratory experiment safely and will have the opportunity to improve their communication skills through preparation of laboratory reports.

Experiments

For the purpose of conducting experiments, students are to form groups (two students per group). Each group will be required to carry out four experiments to complete the course. The experiments for this course are as follows:

1. Fermentation: Kinetics of yeast growth
2. Filtration: Separation of calcium carbonate from water
3. Distillation: Batch separation of methanol from water
4. Liquid-Liquid Extraction – Partially miscible liquids
5. Boiling and condensing heat transfer
6. Fluidization: Pressure drop and heat transfer

Laboratory reports

Each partner in the group is to submit one formal report (maximum 15-16 pages excluding appendices), one brief technical report (maximum 8-10 pages excluding appendices) and two technical memos (maximum 2 pages with an appendix of one sheet containing a maximum of three figures) to complete the requirements of the course. The partners are not allowed to submit the same kind of reports for the same lab and whenever one partner hands in a technical memo, the other partner must submit either a formal report or a brief technical report. For groups with more than two partners these rules will be relaxed. The guidelines for preparation of reports can be found in the laboratory manual. Although every group performs the same experiments, your data, interpretation, analysis, background review, etc. should be unique and based on your own ideas. Plagiarism is not permitted; use your own analysis and thoughts! In addition to submission of a printed copy, students are expected to email an electronic copy of their report to the instructor (section 1: [Naveenji Arun](#) section 3: [Dr. Glyn Kennell](#)).

Laboratory notebooks

All original observations should be recorded clearly and neatly in a bound notebook. Data should be kept in an orderly and reasonably neat form. Students submitting a technical letter must carry out at least a sample calculation and include it in the notebook. All laboratory notebooks must be examined, dated and initialed by the laboratory demonstrator before you leave the lab. All laboratory notebooks must be handed in at the end of the term and will be assigned a mark.

Safety

Students must have studied the Department's Safety Handbook prior to performing experiments. (www.engr.usask.ca/index.php?cmd=tree_nodeID921). Each student must hand in a signed safety release form to the Laboratory Coordinator before they will be permitted to perform experiments in ChE 414. The form is available on the website indicated above. Safety regulations must be followed at all times while working in the Chemical Engineering Laboratories. The wearing of hard hats is mandatory in Room 1D25.1A. The hats are available in that room. These hats are not allowed outside this room and must be returned before you leave.

Evaluation and mark distribution

Careful measurements, correct calculations, logical deductions and clear conclusions are all necessary for a good report. However, even if all these are present but the report is not well written, some of the positive effects of the investigation will be lost. Technical content, clarity, innovative interpretations, and conciseness are important. Proper spelling, grammar and correct use of the English language are also important and will have an effect on the final mark. Although every group performs the same experiments, your data, interpretation, analysis, background review, etc. should be unique and based on your own ideas. Plagiarism is not permitted; use your own analysis and thoughts!

The formal report will be worth 35 marks, the brief technical report, 25, and the technical memo, 10 marks. The lab demonstrators will be reviewing your performance while you are in the lab and will assign a mark (out of 2.5) at the end of each lab period. The deadline for receiving reports and technical letters without any penalty will be two weeks after the date that you performed the experiment. A penalty of 10 % per week will be deducted from late reports or letters (or 1.4% per day, including weekends). In this course, each student will be given 7 “free” late hand-in days (weekend inclusive) to apply to reports as they wish, but submissions will not be accepted after the last day of classes.

Reports handed in after the last day of classes will not be marked and will be counted as zero! In order for late reports to be approved for marking a valid, written reason must be presented to the head of the department and must be accepted by the department in committee. This will not occur until at least one month after the end of the term.

Mark distribution

Component	Number	Percent for each	Final percent
Formal report	1	35	35
Brief report	1	25	25
Technical memo	2	10	20
Lab performance	4	2.5	10
Lab notebook	1	10	10
Total mark			100

The mark distribution is only approximate. Final grades will be assigned at the discretion of the instructor subject to the University Council and College Regulations on Examinations. **Students should be aware of and follow the University of Saskatchewan Academic Conduct and Integrity definitions, rules and procedures that are available on the web at www.usask.ca/honesty.**

GUIDELINES FOR PREPARATION OF TECHNICAL REPORTS

A good technical report is an essential part of any experimental study. Employers in industry often complain about the poor quality of the reports prepared by graduate engineers. No matter how good a technical investigation or study may be, the work is deemed a failure if the facts and ideas developed in it are not communicated effectively to the supervisor and others who can make use of the results.

Although the type or style of a report may vary from one organization to another, the object is always to communicate clearly and concisely. A number of suggestions from the point of view of people in industry can be found in the book entitled, "Effective Communication for Engineers" [31]. Proper technical report writing is nicely described in the book "Technically Write" [2]. One further suggested reference concerns the writing styles in undergraduate reports [3]. Here are a few suggestions:

- Organize the information in the report in a logical manner so that the reader can understand what you are trying to say.
- Use graphs and tables to communicate results whenever possible. Graphs that illustrate your important findings should be located in the main body of the report. In full reports, as opposed to technical letters, the same results should also be

presented in tables in the Appendix. Arrange graphs, other diagrams and printed outputs in such a way that they help to illustrate your points.

- Output data must be presented neatly and each chart titled to describe its conditions. Graphs and figures can be used very effectively to support your comments and conclusions.
- Provide meaningful conclusions and material supporting the conclusions. In stating the conclusions, draw the reader's attention to supporting data or results. If possible, offer a reasonable "theoretical" explanation for the conclusions. Make statements as quantitative as possible.
- Do not omit any essential information or explanation. Include safety and chemical hazard information.

Formal reports

Formal reports are full reports and should include the following:

- 1) Title page
- 2) Abstract
- 3) Table of contents
- 4) Nomenclature
- 5) Introduction
- 6) Review of theory or literature
- 7) Description of apparatus (with sketches)
- 8) Experimental procedures
- 9) Results and discussion
- 10) Conclusions
- 11) Recommendations
- 12) References
- 13) Appendix

(Maximum 15-16 pages excluding appendices)

A technical report should tell the reader what was done, what calculations were made, and what conclusions were reached. Sufficient explanation should also be provided so that the reader can follow the logic of the writer. The report should be reasonably complete so that it is not necessary for the reader to refer to the laboratory notebook. The main equations used in the analysis should be stated. Symbols must be clearly defined. Derivations should not be given except possibly in an appendix. References should be given to relevant theory in appropriate reference books. Large amounts of data should be excluded from the main part of the report and put in an appendix if needed. The main part of the report should be complete in itself so that it is not necessary to read the appendix unless further details are needed.

The report should begin with a title page, which will give the course number and course title, title of the experiment, your name and your partner's name, the date the experiment was performed, the due and submission dates, the Department address, your email address and your signature. An abstract should follow the title page and should contain a brief statement of the purpose of the investigation, a brief explanation of the system and how the results were obtained, and a concise, quantitative description of the main results and conclusions. It should be no longer than one page (**no graph or table in the abstract!**). A Table of Contents should follow the abstract and then a Nomenclature page. All pages in the report should be numbered (except title page) with the Abstract through Nomenclature being numbered i, ii, iii, . . . and the main part of the report starting on page 1.

The organization of the balance of the report is left to the student's discretion. However, the following should be kept in mind. The leading paragraphs of the main part of the report should include information about the purpose of the investigation, its importance in industry and sufficient theoretical background to inform the reader of the fundamental laws which apply to that particular experiment. The sources of equations and information used in the theoretical background should be referenced. The references should be listed on the last page of the main body of the report and just before the first appendix. Details of the theory and derivation of equations should be referenced rather than included in full in the text of the report.

A schematic diagram of the apparatus and a complete description of the equipment and material used should be included in the next section of the report followed by the procedures. The procedures should be presented in paragraph format (using complete sentences) and provide the reader with sufficient information to repeat the experiment (**do not repeat the instructions from the manual, describe what you did**). Technical brochures which describe the equipment and/or procedural manuals which include operating details may be referenced rather than summarized.

The raw data obtained from the experiment should be included next. If a large amount of data was collected, it should be presented in a table in the appendix. In that case, the most significant data and results can be included in one or two tables in the body of the report. A sample calculation should be given in the appendix of the report and should be presented in a logical sequence with accurate referencing to the experimental data used (what values

were used and where they are found in the report, e.g. table number). Results of calculations should be given, usually in the form of tables and graphs as appropriate, and fully discussed (discussion of experimental error, comparison to theory or other literature, conclusions drawn, etc). Conclusions should be summarized following the results. State the conclusions clearly and concisely. They may be presented in numbered statements (no discussion). Sources of error or suggested modifications in the procedure may be included in recommendations for future work. Ultimately, the experiment was conducted for a specific purpose. The report must indicate what was determined from conducting the investigation with respect to this purpose.

Brief Technical Reports

A brief technical report should include:

1. Title page
2. Summary
3. Results and discussion
4. Conclusions
5. Recommendations
6. Appendix.

(Maximum 8-10 pages excluding appendices)

It is equivalent to the formal report but with the abstract replaced by a summary and the absence of the introduction, theory/literature review, materials and methods sections. The summary should include: a brief introduction stating the nature and purpose of the investigation, a brief explanation of the experimental system, procedures and a summary of the important results. There should be an appendix which includes only raw experimental data and a sample calculation.

Technical Memorandum

A technical memo is a brief memorandum to the supervisor/instructor. It should state concisely the experimental conditions, results, discussion, conclusions and recommendations. A brief table of results may be inserted into the text of the memo to support the conclusions. The text should not exceed two double-spaced typewritten pages. One page for an appendix containing a maximum of three figures may be included. See the Chapter “Informal Reports Describing Facts and Events” in Blicq [2].

Useful references for technical writing

1. Effective communication for engineers. McGraw-Hill Book Company. 1975 (Library Call No. T 10.5, E27 1975).
2. Blicq, R.S. Technically write: Communicating in a technological era. Prentice-Hall Canada. 1998 (Library Call No. T 11.B62)
3. Jeter, S. M. Writing style and standards in undergraduate reports. Glen Allen, Va.: College Pub., c2004. (Library Call No. LB2369 J48 2004).

Sample of evaluation sheets

ChE 414 – FORMAL REPORT GRADE SHEET

Student: _____ Experiment: _____

Date Due: ___/___/___ Date Rec'd: ___/___/___ Late Penalty: _____%

REPORT SECTION	CLARITY OF PRESENTATION G.P. x Wt. = Mk			TECHNICAL CONTENT G.P. x Wt. =Mk		
Title Page		2				
Summary		4			5	
Table of Contents Nomenclature		2				
Introduction & Theory		4			10	
Apparatus & Procedure		4			5	
Results & Discussion		8			15	
Conclusions & Recommendations		4			5	
References		2				
Appendices Experimental Data Calculated Results Sample Calculation		4			5 5 5	
Totals		34			55	

SEE THE REPORT FOR COMMENTS

Report Mark = (Total Mark) / 8.9 = _____ %

*** GRADE POINT (G.P.) DESCRIPTOR ***

10	9.5	8 - 9	7 - 7.5	6 - 6.5	5 - 5.5	0 - 4.5
Exceptional	Excellent	Very Good	Good	Satisfactory	Passable	Fail

ChE 414 – BRIEF REPORT GRADE SHEET

Student: _____ Experiment: _____

Date Due: ___/___/___ Date Rec'd: ___/___/___ Late Penalty: _____%

REPORT SECTION	CLARITY OF PRESENTATION G.P. x Wt. = Mk			TECHNICAL CONTENT G.P. x Wt. =Mk		
Title Page		2				
Summary		4			5	
Results & Discussion		8			15	
Conclusions & Recommendations		4			5	
Appendices Experimental Data Calculated Results Sample Calculation		4			5 5 5	
Totals		22			40	

SEE THE REPORT FOR COMMENTS

Report Mark = (Total Mark) / 6.2 = _____ %

* GRADE POINT (G.P.) DESCRIPTOR *

10	9.5	8 - 9	7 - 7.5	6 - 6.5	5 - 5.5	0 - 4.5
Exceptional	Excellent	Very Good	Good	Satisfactory	Passable	Fail

ChE 414 – TECH MEMO GRADE SHEET

Student: _____ Experiment: _____

Date Due: ___/___/___ Date Rec'd: ___/___/___ Late Penalty: _____%

REPORT SECTION	Clarity and Content G.P. x Wt. = Mk		
Title and Names		2	
Brief Background		4	
Technical Summary		8	
Conclusions & Recommendations		4	
Totals		18	

SEE THE COMMENTS ON THE MEMO

Report Mark = (Total Mark) / 1.8 = _____ %

* GRADE POINT (G.P.) DESCRIPTOR *

10	9.5	8 - 9	7 - 7.5	6 - 6.5	5 - 5.5	0 - 4.5
Exceptional	Excellent	Very Good	Good	Satisfactory	Passable	Fail

EXPERIMENTS

1. FERMENTATION: KINETICS OF YEAST GROWTH

Objective

To study the biological process of yeast growth and to determine the kinetics of the process.

Introduction

Fermentation involves the growth of microorganisms on an organic substrate. The end products of fermentation include the microorganisms as well as reduced or oxidized organic compounds. Sugars are a good substrate for microorganisms to grow on because they yield both oxidizable and reducible intermediates. The end products of fermentation are determined by the type of microorganism, type of substrate and environmental factors such as temperature and pH.

The lactic acid and alcoholic fermentation processes are two routes commonly used by cells to metabolize sugars. Yeasts use the alcoholic pathway. A hexose sugar produces 2 moles of CO₂ and 2 moles of ethanol (92 g) per mole of sugar (180 g of glucose) utilized. The theoretical yield of ethanol, therefore, is 51.1% by weight. The process generates 2 moles of adenosine triphosphate (ATP) per mole of sugar utilized, which can be regarded as "energy" for the microorganism to use for growth and reproduction.

Some microorganisms use molecular oxygen in their metabolism and grow aerobically. This process is known as respiration as opposed to fermentation. Respiration is much more efficient than fermentation with 38 moles of ATP being produced per mole of sugar utilized. The sole carbon product in this case (besides microbial mass) is carbon dioxide. In recent years, however, the word "fermentation" is being used to describe any culture of microorganisms.

Saccharomyces cerevisiae are capable of growing either by a fermentation or respiration pathway (facultative). If sufficient O₂ is available, they will prefer to use the respiratory pathway, reducing the sugar up take and spoiling ethanol production. However, there is a greater production of cell mass per unit mass of substrate consumed.

S. cerevisiae is a high poundage product. In North America over 500 million pounds are produced annually.

Experiment

In this experiment you will be growing a strain ("baking" version) of *S. cerevisiae* commonly used to produce bread. You will use a combination of one of the following conditions (ask your instructor).

Initial Glucose Conc. (g/L): 40, 80, 120

Yeast Inoculation (g/L): 3.0, 4.0, 5.0, 6.0

Temperature (°C): 20, 22, 24, 26, 28, 30

The reaction is under batch conditions. You must take a pre-inoculation sample; post inoculation sample; after the lag phase is completed sample every 10 minutes for the first hour; every 30 minutes for the next two hours; every hour for the next two hours; and one final sample the next morning. You should determine the following parameters:

- Kinetic growth constants
- Cell mass yield
- Ethanol yield

Fermenter Apparatus

The culture vessel is an "Omni-Culture Plus", Virtis Company, bench-top fermenter – chemostat (similar to that shown below)

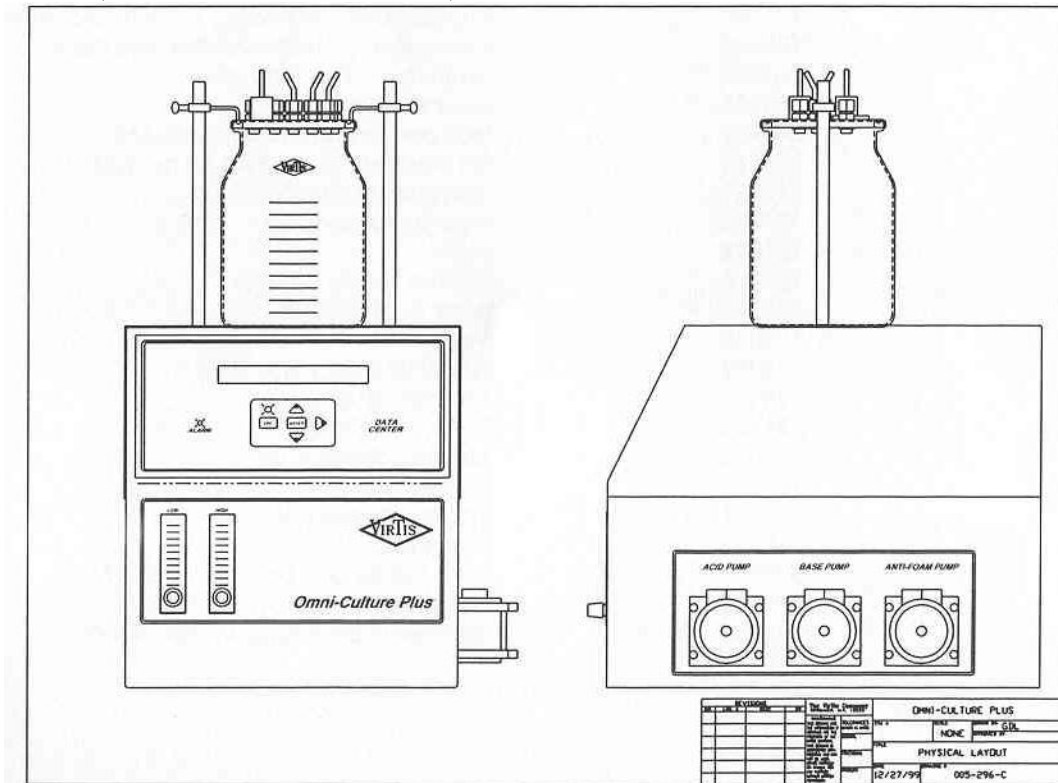


Figure 1. Schematic of Omni-Culture Plus fermenter.

This equipment can be used to perform continuous or batch experiments. You will be using only the batch mode. The pH will be monitored but not controlled.

The vessel is a 2500 mL total capacity glass jar, having a working volume of 2000 mL in the batch mode. Fittings are provided for temperature measurement and control, inoculation, and sampling.

Temperature control is assisted by the use of a water bath. Mechanical agitation is provided by a six-blade impeller coupled magnetically to a variable speed motor (from 200 – 1000 rpm) Panel readout alternately provides the current and set-point values for the RPM, temperature, air, pH, DOX, foam and time. The readings for air, DOX and foam are irrelevant for this experiment and may be ignored.

Method

The media is already prepared for you in the fermentation flask. The flask contains 1500mL. Besides glucose, the broth includes other nutrients including: yeast extract (8.5 g/L), NH_4Cl (1.32 g/L), $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (0.11 g/L), $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (0.08 g/L), K_2HPO_4 (2.0 g/L).

You will grow the yeast under low oxygen conditions, so little air has to be provided. You must bring the media to the required temperature and mix the broth at 500 RPM. Yeast will be provided to you in a freeze-dried state. Carefully weigh out the mass of yeast that you need for inoculation (ask demonstrator). Before inoculation, take a 4 or 5 mL sample. When the broth has reached the required temperature, begin the fermentation by inoculating the yeast through the inoculation port. Do this quickly to prevent contamination by bacteria. Record the time and immediately take your second sample.

All samples will be analyzed for glucose after filtering, cell mass and ethanol concentration (after filtering) as discussed in the Appendix. Cell mass should be done right away. Ethanol can be done after the three hour lag phase has been completed. Filtered samples can be stored in the department's refrigerator for later analysis.

The yeast can be viewed under the microscope according to the procedures outlined in the Appendix. Pictures are to be taken under the direction of the lab demonstrator.

You must compare your results to the theoretical curves available for microorganism growth, and evaluate your kinetic constants and yield factors. In addition, some scale-up evaluation would be of interest.

Theoretical Background

When a microorganism is first introduced to a new growth media, it takes a certain amount of time before it becomes accustomed to its new conditions. This is known as the "lag phase" and little extra cell mass is generated here. Once growth begins to occur it generally follows a sigmoidal type of curve during which growth is accelerating quickly (log phase) at first but then de-accelerates as substrate (glucose in this case) is consumed. A general curve is as shown in Figure 2:

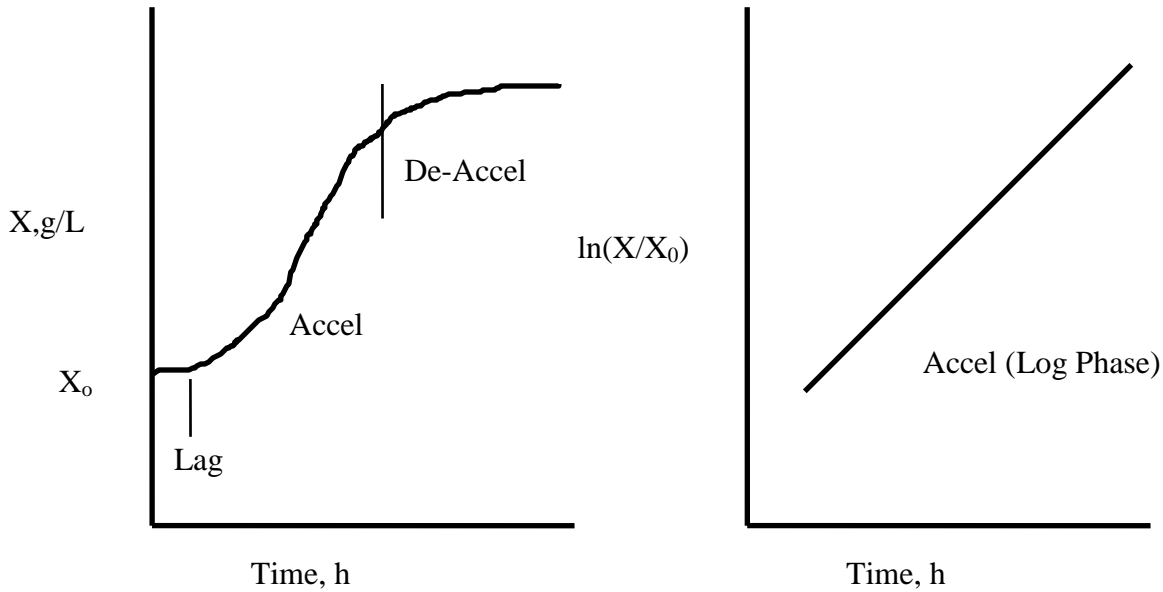


Figure 2. Typical growth curves of yeast.

During the acceleration (log phase) and de-acceleration phases of the growth curve, the following transient biomass equation defines the specific growth rate (μ) of the microorganism:

$$\frac{dX}{dt} = \mu X \quad (1)$$

The specific growth rate, μ , is a function of the substrate concentration, often modeled according to the Monod equation

$$\mu = \frac{\mu_M S}{K_S + S} \quad (2)$$

...where t = time (h)
 μ = specific growth rate (h^{-1})

μ_M	=	maximum specific growth rate (h^{-1})
K_s	=	Monod or saturation constant (g/L)
X	=	cell mass concentration (g/L)
S	=	substrate concentration (g/L)
S_o	=	substrate concentration at time 0 (g/L)

If K_s is quite small (say 0.2 g/L) relative to S_o (10 to 50 g/L), the specific growth rate will be relatively constant near μ_m for most of the batch fermentation as μ is not affected by S until S is quite small, which occurs very near the end of the run. Therefore, during the acceleration phase, equation (1) can be integrated to give:

$$\ln\left(\frac{X}{X_o}\right) = \mu_M t \quad (3)$$

...where X_o = cell concentration at time $t = 0$ (inoculum)

A plot of $\ln(X/X_o)$ versus t over the acceleration phase of growth should give a straight line with a slope of μ_M - hence the reason why it is called the log phase of growth.

The production of product (P) and cells ($X - X_o$) is also a function of the decrease of substrate:

$$\frac{dP}{dt} = -Y_P \frac{dS}{dt} \quad (4)$$

and
$$\frac{dX}{dt} = -Y_X \frac{dS}{dt} \quad (5)$$

...where P = product concentration (g/L)
 Y_P = product yield factor (g product/g substrate used)
 Y_X = cell yield factor (g cells produced/g S used)

If both product and cell yields are constant over the batch run, they can then be determined at any time, t , from the following equations:

$$Y_P = \frac{P}{(S_o - S)} \quad (6)$$

and
$$Y_X = \frac{(X - X_o)}{(S_o - S)} \quad (7)$$

Report

(a) **Results:** Present a semi-log plot of cell growth with time and fit equation (3) to the data and determine μ_m . Tabulate the values of product and cell yields over the run as well as the average. Comment on the difficulties (impossibility?) of obtaining K_S from your data (you are not required to determine K_S) and discuss experiments which could be conducted to determine K_S .

(b) **Design:** Design a fermenter to handle 1000 litres of medium containing 90 g/L of glucose. Estimate the time for the fermentation to be complete (assume a yeast inoculation of 1.0 g/L), the concentration of ethanol produced and the amount of ethanol produced. If 7 hours are required to empty, clean and fill the tank, how much ethanol could be produced in one year assuming an operating time of 50 weeks. State any other assumptions used in the analysis.

NOTE: This experiment will take about 5 hours to complete as growth is quite slow at the beginning. It is important, therefore, that you start the lab before lunch if possible. You should make arrangements with your demonstrator to inoculate the fermenter some time during the morning (mid to late, between classes).

Additional information

A. Optical Density

The amount of light scattered by a microbial suspension can be proportional to its mass or number concentration. The spectrophotometer will measure the amount of light, which passes through a given distance of your suspension. This is recorded as:

$$T = 100 \left(\frac{I}{I_o} \right)$$

...where: T = % Transmittance
I = light incident on detector
I_o = light incident on detector with no microbes

The concentration of cells in the solution is related to the ratio of detected light by the Beer-Lambert law.

$$\log_{10} \left(\frac{I_o}{I} \right) = eCL = OD$$

...where: e = extinction coefficient (depends on wavelength)
C = cell concentration

L = path length
OD = optical density
or OD = $2 - \log_{10} T$

You will measure OD at 640 nm. If necessary your samples must be diluted so that the OD to fall within 0 to 0.3 OD units.

Sample Preparation: Using a clean 1 mL pipette, transfer 1 mL of your sample from the fermentor sample bottle to a cuvette containing either 50 or 100 mL of deionized water. Mix well and measure OD using deionized water as blank. The corresponding cell mass, x, can be found from the empirical equations:

$$x = A * \text{O.D.} \quad \dots \text{ for 50 mL dilution}$$

$$x = B * \text{O.D.} \quad \dots \text{ for 100 mL dilution}$$

...where A and B are correlation constants. Consult the demonstrator for their values.

B. Microscopic Examination

Visually examine the microbial mass using 45x lens on microscope by the following procedure:

- a. Take 1 drop from your sample bottle and place on a clean microscope slide. Smear to a thin layer.
- b. Place a "slip cover" over the wet smear.
- c. Investigate under the microscope to determine shape and size, take a picture for your report.

C. Ethanol Analysis

Your samples must first be quickly centrifuged and decanted to remove the yeast and stop fermentation.

The ethanol will be determined by GLPC (gas-liquid partition chromatography). The instrument is a Shimadzu GC- 2014 gas chromatograph, the column is a HP Plot U, 30m x 0.530 mm.

The chromatograph is computer controlled; operation of the program will be discussed by the laboratory demonstrator at the time of the experiment.

D. Glucose Analysis Procedure

Your samples must first be quickly centrifuged and decanted to remove the yeast and stop fermentation.

The instrument is a YSE 2700 Select Biochemistry Analyzer. The SOP is available in the laboratory; operation of the instrument will be discussed by the demonstrator.

Reference:

Shuler, M.L. and Kargi, F. Bioprocess Engineering, Basic Concepts. 2nd Ed. 2002. Prentice-Hall Inc.

Bailey, J.E. and Ollis, D.F. Biochemical Engineering Fundamentals. 2nd Ed. 1986. McGraw-Hill Book Company.

2. FILTRATION: Plate and frame separation of calcium carbonate and water

Background

The plate and frame filter, or filter press, is one of the most widely employed filter designs. It consists of two end filter plates to which the filter medium is attached. Between the two end plates are frame plates and wash plates. Feed slurry is pumped into the frame plates and filtered solids collect there. Wash plates have a filter medium on both faces and have a particular port arrangement to enable slurry washing procedures. The number of frame plates and wash plates can be altered so that the separation area and solids capacity of the filter meets a range of demands. All of the plates are held pressed together to form a seal which enables the filter unit to be operated at pressure, this pressure being the driving force for the filtration or washing/drying processes. The plate and frame filter is batch operation and usually requires dismantling for the recovery of solids at the end of the filtration cycle. It also allows washing of the filter cake in-situ.

The Armfield UOP12 Filtration Unit uses a press made from 5 plates, two end plates, two frame plates and a wash plate. The filter medium has nominal pore size of 63 μm . While this size allows reasonable filtration rates, it also necessitates the use of precoat/filter aid to recover particles much smaller than 63 μm .

Theory

The general equation for filtration is developed from first principles for flow through porous media. The theoretical development is performed excellently by Bennett and Myers (3rd Edition, pages 224-244). In brief, for an incompressible filter cake (possibly weak assumption for these conditions), the filtration equation giving the instantaneous relationship between upstream filter pressure and flowrate is:

$$P = (K_1 \times V + K_2) \times Q \quad (1)$$

....where P = pressure upstream of filter unit, Pa
 V = volume of filtrate collected, m^3
 Q = flowrate of filtrate, m^3/s

... and K_1 is a constant which is strongly dependent on the characteristics of the cake building up in the filter according to:

$$K_1 = \frac{s \times \rho \times \mu \times \alpha}{(1 - m \times s) \times A^2} \quad (2)$$

... where: s = mass fraction of solid in original slurry
 ρ = liquid density, kg/m³
 μ = liquid viscosity, kg/m-s
 α = constant specific cake resistance, m/kg
 m = ratio of wet cake to dry cake
 A = filtration area, m²

... and K_2 is a constant that is strongly dependent on the characteristics of the filter medium:

$$K_2 = \frac{R_M \times \mu}{A} \quad (3)$$

... where: R_M = filter medium resistance, m⁻¹

The equation for volume of filtrate collected relative to the flowrate of filtrate is:

$$Q = \frac{dV}{dt} \quad (4)$$

where ... t = time, s

For constant flowrate filtration ($Q=Q_0$), we can easily see that $V=Q_0 \cdot t$ and putting that in the filtration equation (Equation 1) we get:

$$P = K_1 \times Q^2 \times t + K_2 \times Q \quad (5)$$

Thus a plot of P vs t can be used to determine K_1 and K_2 from the slope and intercept of the hopefully straight line. Note that if there is no slurry and therefore no cake building up in the filter, then the flowrate should be proportional directly to the upstream pressure.

After filtration is ceased, in practise, wash water is pumped through the cake to wash out the mother liquor. Finally, some time is needed to open the filtration press, drain and dump the cake, and then reassemble the apparatus. Thus the total filtration time is the sum of the filtration, wash and empty/reassemble times.

Apparatus and Procedures

Note: This apparatus uses a progressive cavity pump that is good for pumping a slurry. This type of pump must be primed before use. **Never run this pump dry, pull out the top steel pipe to ensure liquid is in the pump before you start these experiments.** The first time you use the pump, disconnect the tube to the filter press and pump the initial water into the drain tank. This is to prevent sediment (rust) in the pump from entering the filter press. Also, continuously monitor and observe the pressure at the outlet

of the pump, it **MUST NOT exceed 1.5 bar**. Immediately stop the pump if this condition occurs.

A PDF manual on the website (ArmfieldFilter.pdf) includes pages from the manufacturer's Teaching manual. Read these pages so that you are clear on all parts of the apparatus, safety precautions and proper operating procedures.

Before you start pumping any fluid through the filter press, make sure the data acquisition system is recording the time, turbidity, flows and pressures in the system. Make sure this software has the correct slurry concentration and solid particle density. Also, make sure the turbidity meter reads zero for pure water running through the pipe. At all times, fluids leaving the filtration unit will be sent to the drains, do not recycle fluids to the mixing tank.

In these experiments, we will only be performing constant flowrate operation, that is we will not operate the automatic pressure control option of this apparatus. Each team will perform Exercises A and modified versions of Exercises B and C. Before pumping slurries through the filter unit at some known constant flowrate (see Table below), you will pump pure water through the filter medium at 20, 40, 60, 80 and 100 L/h, measuring the pressure drop at each flowrate. You will pump two slurries through the filter press, the first one will be 0.5% CaCO₃. The second one will be 0.5% CaCO₃ but will also include some filter air (perlite) as shown in the table below. Run the whole volume of the mixing tank (about 10 L) through the filter press for each of your three runs (the pump will automatically shut off when the tank nears empty).

All calibrations have been done beforehand for you and are shown in this document. Check the concentration of particles in the feed tank prior to running the slurries through the filter press, using the sample tube and the turbidity detector in the loop, the instructor will show you how to do that.

One of the purposes of your study is to compare the filtration of CaCO₃ slurry both with and without filter aid (perlite) in the mixing tank. After pumping each slurry through the filter press, run wash water through the cakes for five minutes (the wash-water pressure should be controlled at 0.5 bar). During wash, the outlet valve V3 must be open and the outlet valve V4 should be shut, but both V3 and V4 are open during slurry filtration. After shutting off the slurry pump and the wash water, close all four valves (V1, V2, V3, V4) thereby isolating the filter unit for removal from the housing.

The filter press will be carefully removed and dismantled, using the instructions of the demonstrator. You will carefully (with the soft flesh of your finger) scrape some cake onto a pre-weighed stopglass to determine the wet and dry weights of some of the cake (not all of it, need only about 5 grams). When you remove the cake, do not scratch the membranes because it is a major problem to replace them and this will mean you have to redo the experiment. After you have sampled some of the cake, rinse the remaining cake off the membranes with a slow stream of cold water. Carefully reassemble the filter press ensuring the gaskets are properly fit into their slots and the frame and wash plates are

placed back together in the right orientation (see the PDF file, the demonstrator should assist you with the reassembly). Tighten the filter press clamp wing nuts when the filter press is back in the housing, and tighten in rotation to maintain uniform clamping pressure. Do not over-tighten, as the apparatus is plastic.

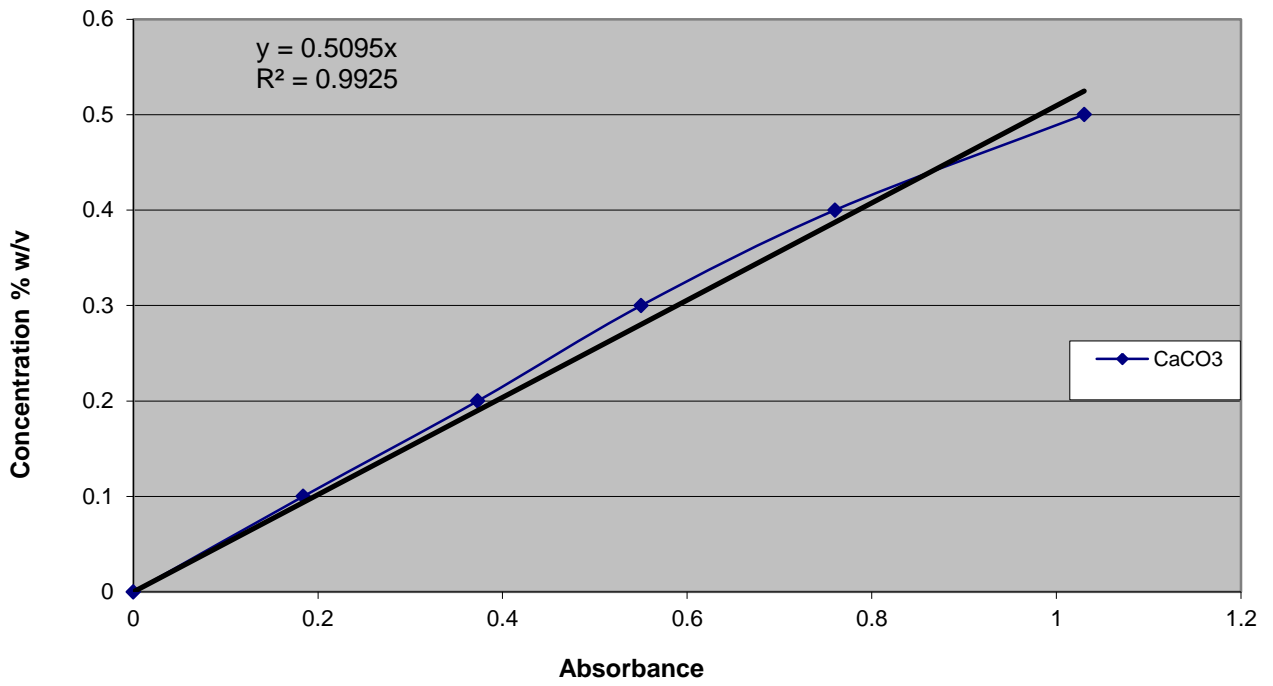
Table 1. Operating Conditions of the Filtration Unit

TEAM	SLURRY FLOWRATE (L/H)	FILTER AID (%W/V)
A	40	0.125
B	50	0.125
C	60	0.125
D	70	0.125
E	40	0.25
F	50	0.25
G	60	0.25
H	70	0.25
I	40	0.30
J	50	0.30
K	60	0.30
L	70	0.30
M	40	0.375
N	50	0.375
O	60	0.375
P	70	0.375
Q	40	0.50
R	50	0.50
S	60	0.50
T	70	0.50

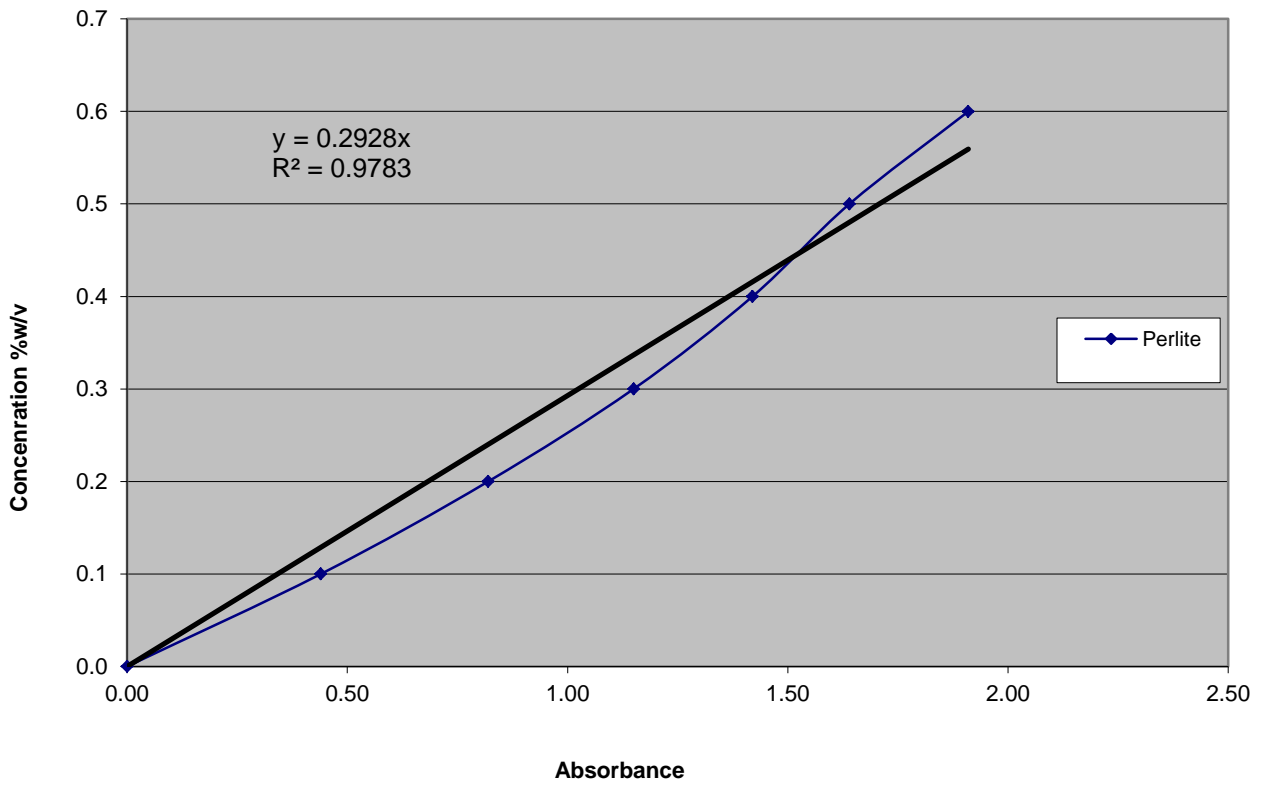
Calculations

Determine as many equation constants of the system as possible for both the case of pure CaCO_3 filtration and for the mixture of CaCO_3 and filter aid. Check that the mass of particles collected by the computer agrees with the mass of particles you placed in the mixing tank. Discuss the effect of filter aid on the operation, especially on the losses of CaCO_3 through the filter medium. Design a filtration press that will clarify an average of $5 \text{ m}^3/\text{h}$ of 0.5 % CaCO_3 solution, first at constant flowrate operating conditions; and second at constant pressure operating conditions; using similar filtration characteristics and flow per unit area conditions as were used in these experiments. Assume that after filtration, washing is carried out at a constant flowrate using water with a volume equal to 10% of the filtrate collected; and a dismantling/cleaning and reassembly time of 5 hours is required. Discuss as fully as possible how you would determine if filter aid is an economical additive to remove the CaCO_3 particles.

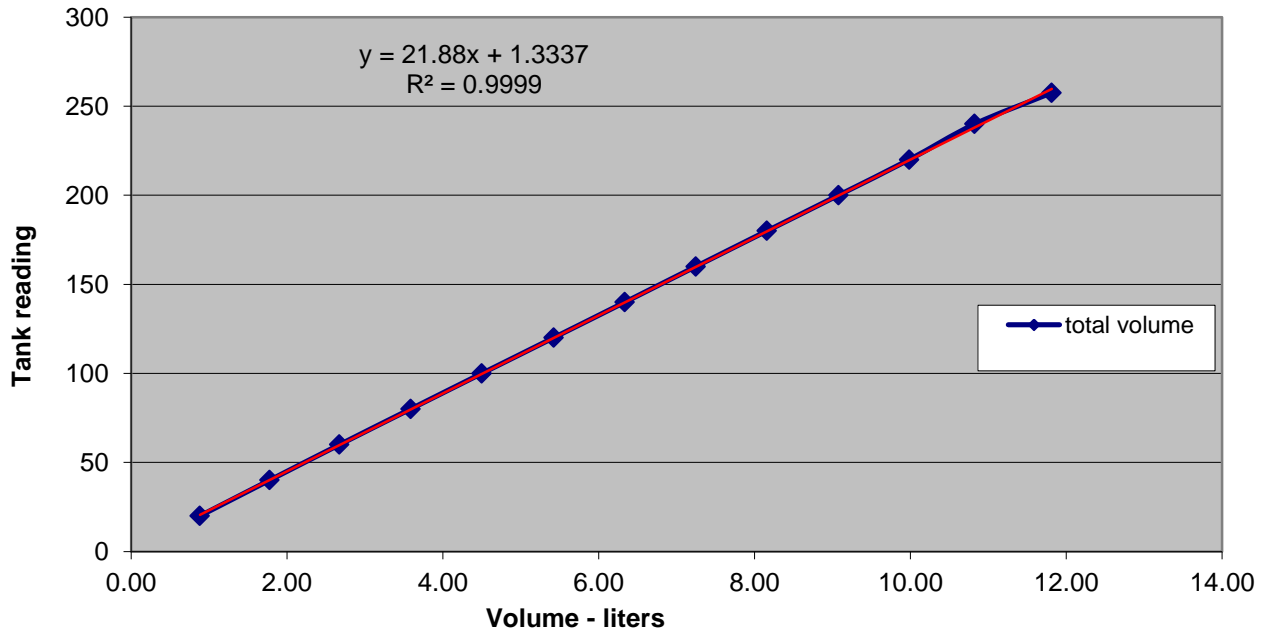
Calcium Carbonate Calibration



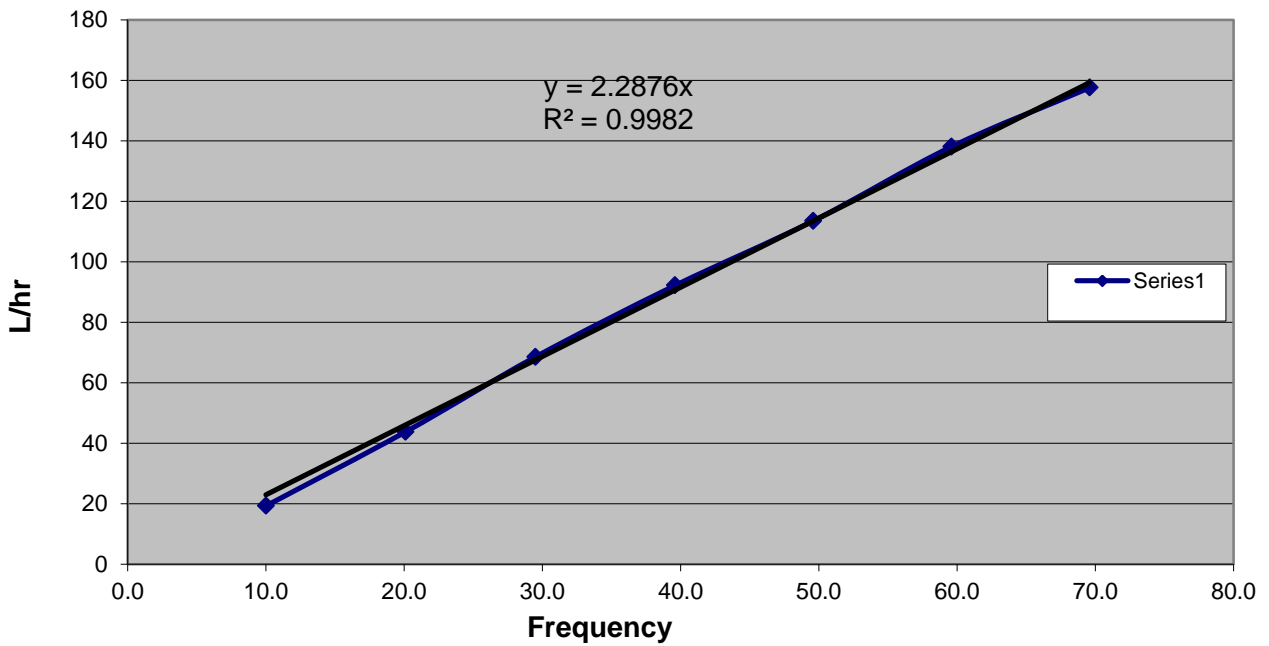
Perlite Calibration



Filtration Tank Calibration



Pump Flow Calibration



3. DISTILLATION: Batch separation of Methanol and Water

Introduction

You have learned the principles of batch distillation in ChE 315, Mass Transfer I. Unlike continuous operation of a distillation column in which the system is operated continuously under steady state conditions, in batch distillation we deal with a dynamic situation. The process starts by charging the still pot with feed (serves as a reboiler) and is heated. Product accumulates in the collection vessel and is removed at the end of the operation. This process is suitable for situations where the facility is used for multiple purposes, the product is demanded seasonally, the upstream process is batch-wise, research-and-development, or the system is fouling and regular cleaning is necessary. Batch distillation could be single stage or multi-stage. In this lab, you will run a batch distillation system for separation of methanol from water under a constant reflux ratio.

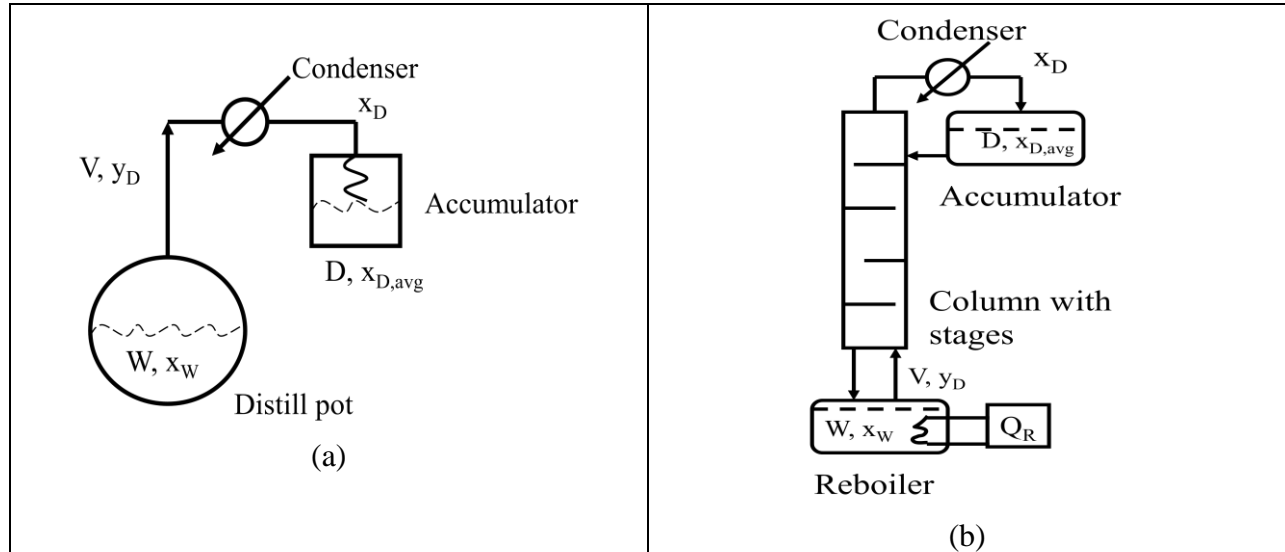


Figure 1. Illustration of batch distillation: (a) single stage, and (b) multistage.

Experimental system

Schematic of the batch distillation system is shown in Figure 2. The packed column placed above the still pot has an inner diameter of 100mm and a height of 1930 mm (packing height: 1220mm). It is packed with knitted wire packing (structured wire packing), referred to by the manufacturer as “Stainless Steel Opti-pak 925” packing. The packing is produced by knitting multiple fine metallic filaments that are then crimped and either spiral wound or folded and layered into elements for insertion into the tower. The structure of the packing creates multiple twisting paths for the vapor to travel while providing a high void fraction that minimizes pressure drop. At the same time, the inherent capillary action of the fine wires causes the liquid to spread as thin layers (films) that continuously combine and divide for optimum mixing and contact with the vapor throughout the packing material. The packing used in our column has a specific surface area of $550 \text{ m}^2/\text{m}^3$.

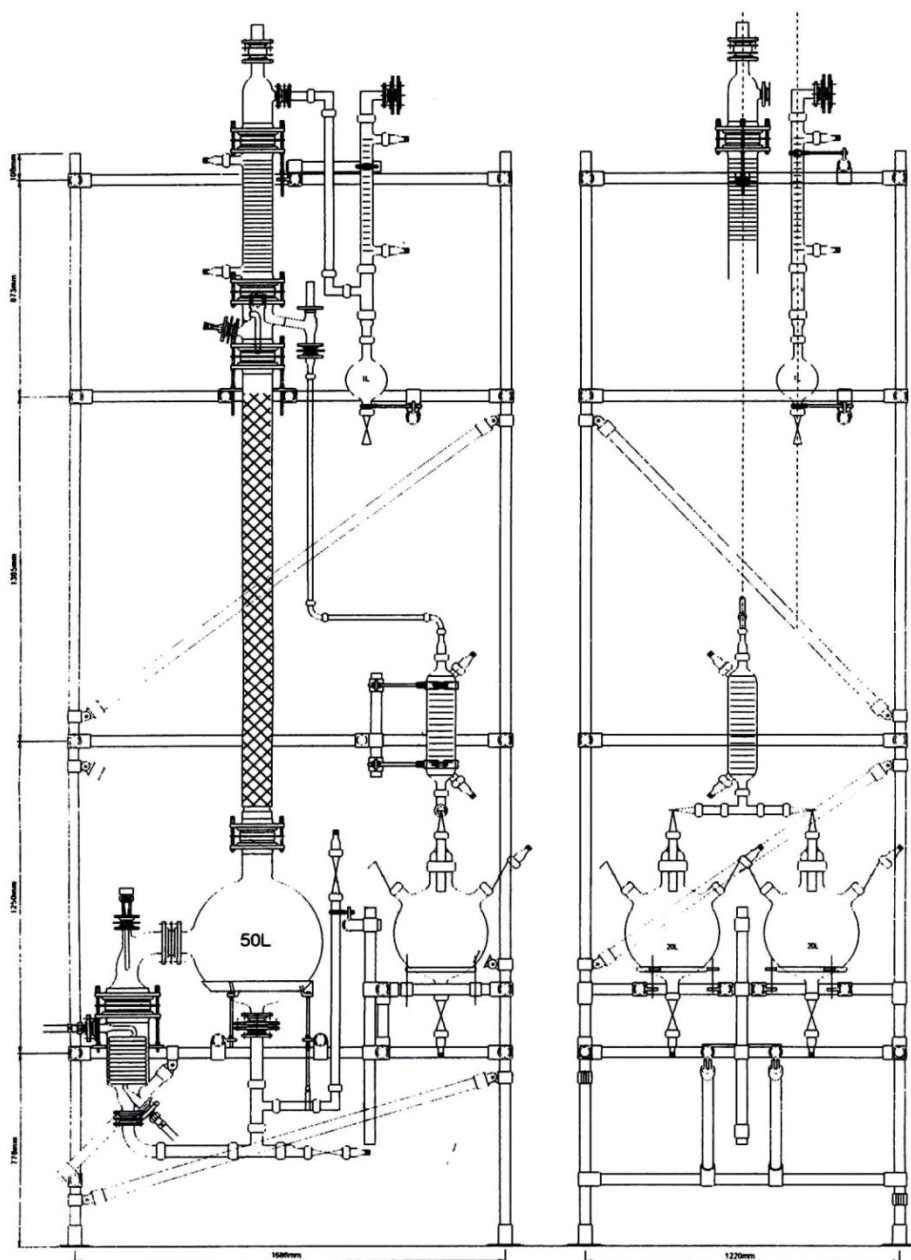


Figure 2. Schematic of the batch distillation system used in this experiment.

Method:

The still pot contains 30 – 50 liters of a mixture of methanol and water of unknown concentration. The volume can be determined by use of the graduations on the still pot, all methanol concentrations will be determined by use of the Agilent 6890N Gas Chromatograph. When you arrive in the laboratory the feed may or may not be preheated for you, in either case you will be required to heat the feed to boiling. Once the condensate has reached the top of the column the system should be run under full reflux for at least 15 minutes before any product is taken, this is done to sufficiently heat the upper sections of the column. After this initial heating period the reflux can be set to a rate determined by your TA.

Process parameters and temperatures are set and observed on the process computer in the pilot plant control room.

Sampling of the condensate can begin immediately and should continue until a sufficient amount of condensate has been collected and a noticeable change in methanol concentration has been observed in the condensate stream.

Final volume and methanol concentration should be determined for the feed (residue) tank and the condensate (product) tank. Be sure to sufficiently mix the contents of each vessel before sampling

Batch Distillation Calculations

Similar to other mass transfer operations mass balance and phase equilibrium are used to analyze the performance of a batch distillation column. However, mass balances can be written for the whole period of the operation, i.e., from the beginning to the end:

$$F_{\text{final}} = W_{\text{final}} + D_{\text{total}} \quad (1)$$

$$F_{\text{final}}z = W_{\text{final}}x_W + D_{\text{total}}X_{D,\text{avg}} \quad (2)$$

or at any moment:

$$x_D dD = d(Wx_W) = W dx_W + x_W dW \quad (3)$$

During operation, the mass is transferred from the distill pot (or the reboiler) into the accumulator, i.e.,

$$dD = dW \quad (4)$$

Combining equations (2) and (4):

$$(x_D - x_W)dW = W dx_W \quad (5)$$

Integrating equation (5) leads to what is called the Reyleigh equation:

$$\int_F^{W_{\text{final}}} \frac{1}{W} dW = \int_z^{x_{x,\text{final}}} \frac{1}{x_D - x_W} dx_W \quad (6)$$

The key to solving the Reyleigh equation is to find the mathematical relationship between X_W and X_D . Once the relationship is available, the equation can be solved numerically, graphically, or from an empirical equation. Please see the reference given at the end for definition of various terms and further information on batch distillation.

As indicated earlier in this lab you will investigate batch distillation of a mixture of methanol-water under constant reflux ratio. Samples from still pot (Residue), condenser (condensed vapor) and accumulator (Distillate) will be collected on regular intervals and will be analyzed by the gas chromatograph to determine X_W , X_D , $X_{D,\text{avg}}$, respectively. You would also determine the approximate volumes of the distillate and waste at the end of the experiment. You will observe how the composition and temperatures change in the still pot as the experiment proceeds.

Questions to answer in your report:

1. The experimental setup has a packed column above the reboiler. Do you treat this as single stage batch distillation or a multistage batch distillation?
2. You observed changes in the reboiler (still pot) temperature during the operation. Do the observed changes make sense? Justify your answer.
3. Using the collected data determine the number of theoretical stages and the HETP for the applied reflux ratio?
4. Using the experimental value of $X_{D,avg}$ for the final distillate, calculate W and X_w and D . Compare these values with those determined experimentally. Discuss the discrepancies and the factors contributed to these differences.

Reference:

Wankat P.C. Separation Process Engineering. 2nd Ed., 2007. Prentice Hall.

4. LIQUID-LIQUID EXTRACTION: Partially miscible liquids

Introduction

You have learned the principles of liquid-liquid extraction, especially, the partially-miscible liquid-liquid extraction, in ChE 315, Mass Transfer I. Simply speaking, liquid-liquid extraction is a mixing-separation process where two phases of liquids are mixed and then one or more desired components are transferred or separated from one liquid phase to the other to some extent due to the difference of their solubility in the two phases. This separation process is widely used in industry. The following photo shows a liquid-liquid extraction unit used in Uranium mill.



(Photo taken by Hui Wang at Cameco's Rabbit Lake Mill, 2007)

Extraction Devices

Due to the wide diversity of application, the industrial devices of liquid-liquid extraction vary greatly. They include mixer-settlers, spray columns, packed columns, plate columns and columns with mechanically assisted agitation. Regardless of the type of equipment, the calculation of the equipment design maintains the same if extraction is considered as an equilibrium-staged process. The number of stages of an extraction system is determined by equilibrium calculation, and the size of devices is obtained using the HETS, or height equivalent to a theoretical stage.

Principles of Liquid-Liquid Extraction

Let's recall the fundamental principles of the liquid-liquid extraction in brief. The task of separation is to transfer one or more components from one liquid phase to the other to a desired extent. The components to be removed or separated are called solute. The former liquid is called diluent and the latter is solvent. The two liquid phases are immiscible or partially miscible most of the time. At a condition where the solubility of a solute, A, in both phases, y_A and x_A , will be in equilibrium, the ratio of y_A to x_A , represented by K_d , is called distribution ratio.

The extraction can be regarded as one or more contacting stages. To simplify the extraction calculation, these stages are assumed as equilibrium ones, as shown in Fig. 1.

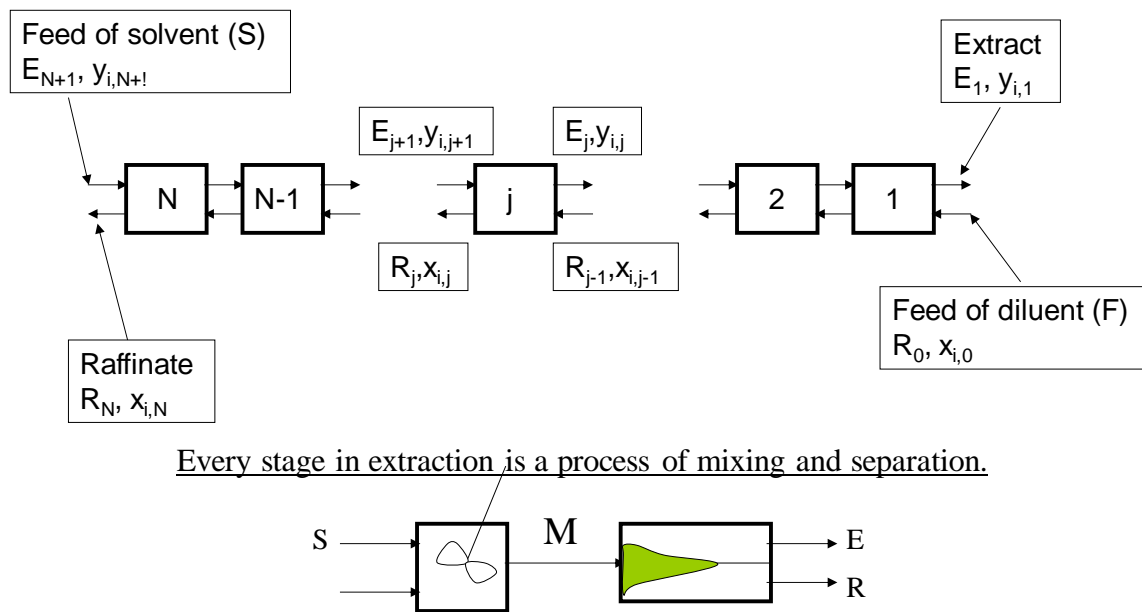


Figure 1. Equilibrium staged liquid-liquid extraction.

Extraction usually takes place as a counter-current flow regime as shown in Figure 1. The feed diluent becomes raffinate and the solvent becomes extract. The term “diluent” is also used to represent the component of the feed in which the solutes are dissolved.

The equilibrium data of an extraction system is generally transferred into a triangular graph as shown in Fig. 2. The two known corresponding equilibrium points in the two phases are connected by lines called tie line. From a set of known compositions of one phase, you can determine the compositions of the other phase if you know how to draw the tie line from a known composition point. This is an important skill to learn when you want to determine the number of stages of an extraction contactor or device.

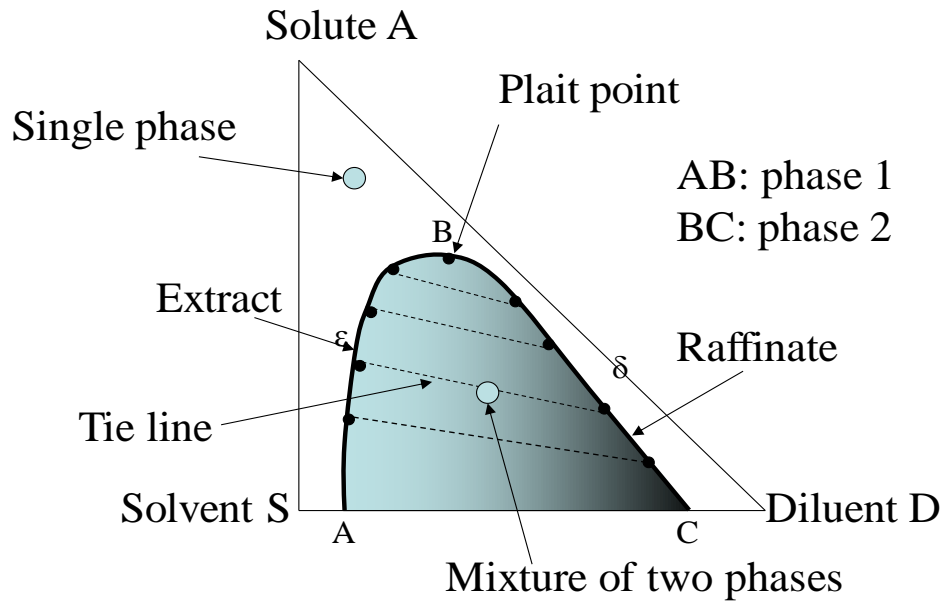


Figure 2. Triangular diagram of equilibrium for a partially miscible extraction system.

The streams of extract and raffinate between any two adjacent stages are called passing streams. Mass balance around an arbitrarily chosen stage shows that the difference in either total flow rates or component flow rates between a pair of passing streams is the same. That is, for total flow rates:

$$\Delta = E_0 - R_1 = \dots = E_j - R_{j+1} = \dots = E_N - R_{N+1}$$

and for component A:

$$\Delta x_{A,D} = E_0 y_{A,0} - R_1 x_{A,1} = \dots = E_j y_{A,j} - R_{j+1} x_{A,j+1} = \dots = E_N y_{A,N} - R_{N+1} x_{A,N+1}$$

and for component D:

$$\Delta x_{D,D} = E_0 y_{D,0} - R_1 x_{D,1} = \dots = E_j y_{D,j} - R_{j+1} x_{D,j+1} = \dots = E_N y_{D,N} - R_{N+1} x_{D,N+1}$$

These equations define a difference point or delta point in the graph shown in Fig. 3.

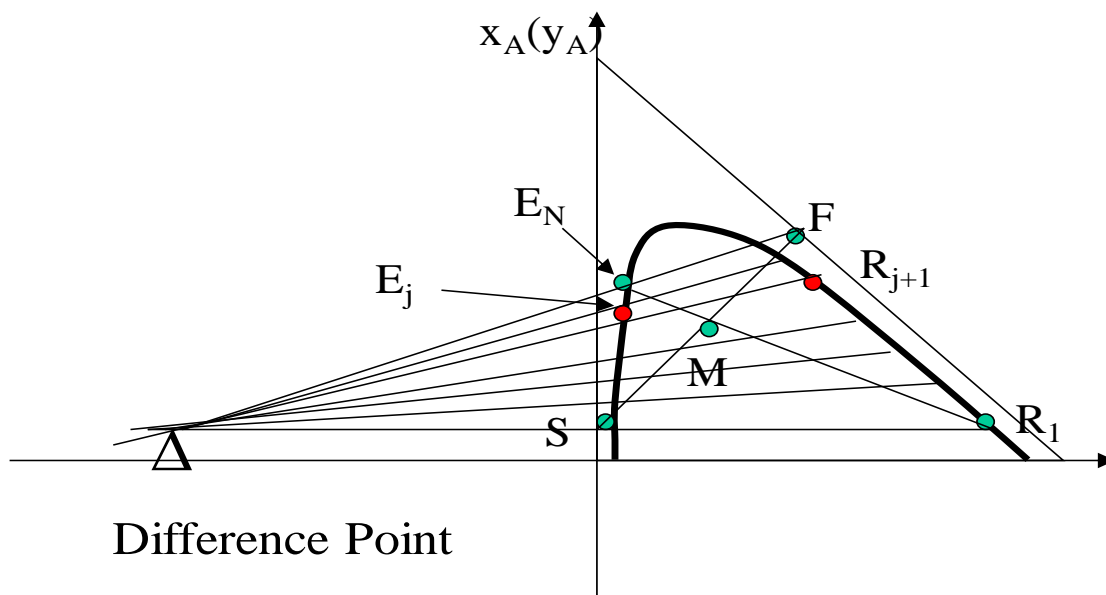


Figure 3. Difference point in the triangular diagram.

Now with the help of difference point and tie lines, you can determine the number of stages by stepping-off should you know the composition of two streams – diluent and solvent and a desired composition of either the outlet raffinate or the outlet extract.

(If you still have difficulty in understanding this part, go back to refer to your ChE 315 textbook or notes or other books on separation process principles.)

Objectives of the Lab

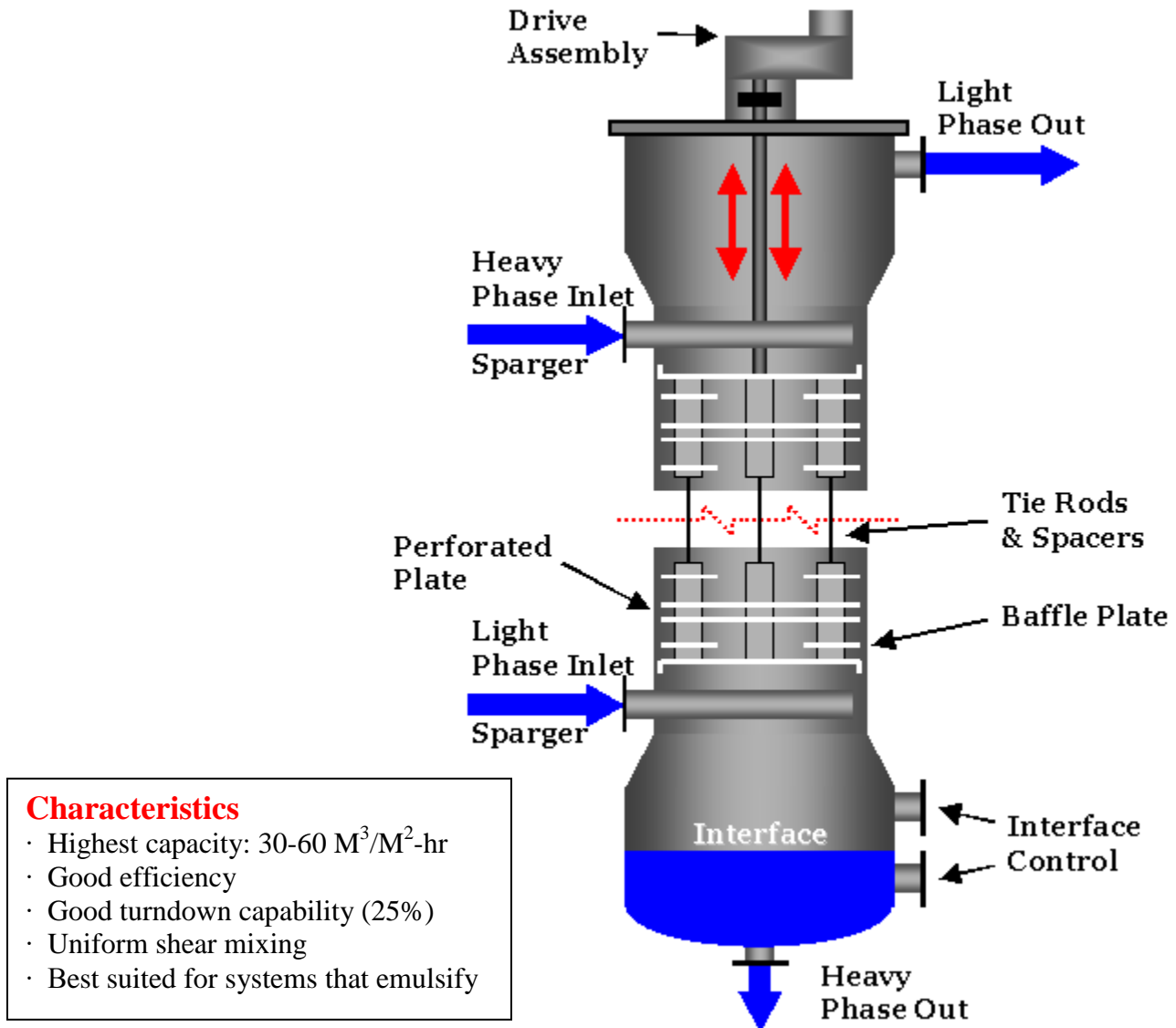
The objective of this experimental lab is to help you to enhance your understanding of the principles of the liquid-liquid extraction processes. You are to perform an experiment with the apparatus called Karr Reciprocating Plate Column, a column contactor with mechanically assisted agitation, to reduce the acetic acid concentration in Methyl-isobutyl ketone (MIBK) by extracting acetic acid into water which is used as the solvent.

The existing liquid-liquid extraction unit will allow the students to investigate the effects of parameters such as initial concentrations of acetic acid in MIBK, flow rates of diluent and solvent, or speed of agitation. However, due to the limited time this year we will focus on the effect of agitation speed.

Experiment Apparatus

The liquid extraction apparatus consists of four tanks, two pumps, a Karr Reciprocating column, flow control valves, and flowmeters. The system operates using an acetic acid rich Methyl Isobutyl Ketone (MIBK) as the Feed, and R.O. water as the Solvent. The water and MIBK can only interact while they are in the Reciprocating Plate column; otherwise they are physically separated in the apparatus.

Figure 1 - Karr Reciprocating Column (or Reciprocating Plate)



<http://www.liquid-extraction.com/karr-column.htm>
Koch Modular Process Systems

October 24, 2007 1: 09 pm

Experiment Procedure

Column Startup

A. Initial Set-up

Collect a 50 mL sample of the Feed and titrate it to determine the initial concentration. (See Titration procedure)

(If Required) Completely drain the column, dispose of azeotrope. The final volume in the column can be drained by opening the stainless steel plug in the middle of the column. Once the column has been drained, close the back flow preventer behind the raffinate flow meter. Fill the column with distilled water from the Solvent tank to the top of the column.

B. Apparatus Start-up

Ask the instructor for the speed of the agitator and set speed on the side of column.

To start the column, first set the “FEED” rate of MIBK/acetic acid to the desired setting, and wait for the MIBK to fill the top of the column, and start to overflow. Once the MIBK is overflowing, set the “SOLVENT” flow rate to around 82 mL/min, and at the same time adjust the “EXTRACT” flow rate to the same setting to ensure column stability.

WARNING: *If the “SOLVENT” flow is higher than the “EXTRACT” flow rate, the interface level will rise until water overflows into the MIBK tank)*

The column is now running, but the water-MIBK interface must be watched consistently to ensure it is not moving up or down. If the “EXTRACT” rate is less than the “SOLVENT” rate, the interface level will drop.

C. Sampling

There are four possible things to sample on this apparatus: FEED, SOLVENT, EXTRACT, and RAFFINATE. The solvent and the feed can both be sampled from their respective tank’s drain valves. The EXTRACT can be sampled at the column drain valve located at the bottom of the column, and the RAFFINATE can be sampled from the small needle valve located on the bottom of the RAFFINATE flow meter.

D. Shutdown

At the end of each lab, the column should be properly shut down to ensure that there are no chemical spills. Turn off pumps. Close the Backflow Preventer.

E. Liquid Transfer: Tank to Tank

I. Opening Valves

To transfer liquid from one tank to the other, the Pump Feed valve must be opened on the tank you want to drain, and the Bypass valve opened on the tank you want to pump to. You must also ensure that the Bypass control valve is open to allow the liquid to bypass the column and flow from one tank to the other.

II. Transfer Solvent

To transfer rinse water from one tank to another you must open the “**SOLVENT**” valve on the tank you want to drain and open the “**PUMP BYPASS**” valve on the tank you want to fill.

III. Transfer Raffinate

To transfer MIBK/Acetic Acid, you must open the “**FEED**” valve on the tank to be drained, and open the “**PUMP BYPASS**” valve on the tank you want to fill.

Before Experiment

The equilibrium data of the ternary system of water-acetic acid-MIBK is taken from the Perry's Handbook of Chemical Engineering is given in Table 1.

Table 1 Water-Acetic Acid-Methyl Isobutyl Ketone (MIBK), 25°C

Weight % in water phase			Weight % in organic phase		
Water	Acetic Acid	MIBK	Water	Acetic Acid	MIBK
98.5	0	1.55	2.12	0	97.88
95.5	2.85	1.70	2.80	1.87	95.33
85.8	11.7	2.50	5.40	8.90	85.7
75.7	20.5	3.80	9.20	17.3	73.5
67.8	26.2	6.00	14.5	24.6	60.9
55.0	32.8	12.2	22.0	30.8	47.2
42.9	34.6	22.5	31.0	33.6	35.4

Before doing experiment transfer this data into a right triangle graph with conjugate line on it and make a few copies of this graph for later use.

After Experiment

Now you have outlet concentrations of acetic acid in raffinate and extract for the runs at different agitation speeds. With the assumption that your operations are performed under equilibrium, using one set of your experimental data to determine the number of stages using the graphic method just shown in the section of Principles. Now compare the performance of the extraction unit operated at different agitation speed. Comment on the observed difference in the results.

Acid/Base Titration for the Determination of Acetic Acid Extraction

Mettler Toledo DL28 with pH Probe

In this experiment, we will be stripping Glacial Acetic Acid from MIBK in a countercurrent extraction. The Solvent will be RO water and it will be an acidic solution as an Extract.

We will be using the Mettler Toledo DL28 with pH Probe for the titration of the extract, which is acetic acid and water. This unit will be using an internal endpoint method called "101". The DL 28 automatically and accurately titrates a weak acid with a strong base to a neutral end point. The $[H_3O^+]$ equivalence point is then calculated, based on the titration curve that the method plots.

A Mettler Toledo DL28 auto-titrator is used for the titrations. The main switch is in the back.

Procedure:

A. Titration of Extract

1. Take 50 mL of Extract
2. Place solution in Titrator sample container
3. Press "101" then "Start"
4. The titration will take 3-20 minutes depending on the initial starting pH and it will signal when done.
5. Dispose of all solutions in waste container provided.

B. Titration of Feed and Raffinate

1. Take 10 mL of Feed or Raffinate and add 40 mL of RO Water

2. Place pH probe in solution.
3. Press “101” then “Start”
4. The titration will take 3-20 minutes depending on the initial starting pH and it will signal when done.
5. Dispose of all solutions in waste container provided.

Materials to be used in Experiment

Methyl Isobutyl Ketone

Flash Point:	16 °C
LEL:	1.2 %
HEL:	8.0 %
Odour Threshold:	0.1 – 0.75 ppm
TWA:	50 ppm
STEL (15 min limit):	75 ppm
Appearance:	Clear, Colorless
Physical State:	Liquid
Molecular Weight:	100.2 g/mol
Relative Density:	0.8 @ 20°C
Viscosity:	0.7 cSt (0.7mm ² /sec) @ 20°C

Glacial Acetic Acid

Flash Point:	40 °C
LEL:	5.4 %
HEL:	16 %
TWA:	10 ppm
STEL (15 min limit):	15 ppm
Appearance:	Clear, Colorless
Physical State:	Liquid
Molecular Weight:	60.0268 g/mol
Relative Density:	1.04 @ 20°C
Viscosity:	1.221 cSt (1.221 mm ² /sec) @ 20°C

Water

Flash Point:	n/a °C
LEL:	n/a
HEL:	n/a
TWA:	n/a
STEL (15 min limit):	n/a
Appearance:	Clear, Colorless
Physical State:	Liquid

Molecular Weight:	18.02 g/mol
Relative Density:	1.00 @ 20°C
Viscosity:	1.00 cSt (1.00 mm ² /sec) @ 20°C

5. Boiling and Condensing Heat Transfer

A schematic diagram of this equipment is given in the Appendix. This experiment is concerned with measurement of heat transfer coefficients for boiling. It is also possible to make an evaluation of heat transfer processes which occur in a condenser. Visual observations of flow regimes play an important role in this experiment since there are substantial effects on heat transfer coefficient when the flow regime changes.

For the condenser, it is important to remember that this is a heat exchanger for which the wall temperature is unknown. Overall coefficients can be measured and they can also be predicted from the values of the individual (inside and outside) heat transfer coefficients.

The purpose of the experiment is to extract as much information as possible from the measurements, and to report this information.

Reading:

Fairly extensive (but not very difficult) reading will be necessary before you do the experiment. Read (if you have not already done so) the following sections in the book by Incropera et al.: 1.2.2, 6.1, 8.5, Introduction to Chapter 10, 10.2, 10.3, 10.6, 11.3.3, and equations 11.14, 11.15 and 11.17.

Procedures:

1. In this experiment, a copper element is heated in Forane R141b. You will be given a p-H diagram for this fluid. The heating occurs because of the passage of an electric current and is measured with a voltmeter and ammeter.
2. The power supply is controlled by an on/off switch and a Variac. The water flow rate is controlled by a valve and pre-calibrated rotameter. There are built-in safety mechanisms which shut the apparatus down if either the pressure or temperature gets too high (220 kN/m² or 160 °C). State in your note book whether this occurred.
3. Visual indications of convection heat transfer, nucleate boiling and film boiling will be observed and should be recorded along with the corresponding operating conditions.
4. A set of experiments is done at constant pressure. To do this the heater is turned on to some low value (5 to 50 watts) with no cooling water flowing. The pressure is allowed to come to steady state and conditions are recorded (V, I, P Cooling Water Temperatures, Cooling Water Flow rate, Temperature of Heater, Temperature of Refrigerant). The heat supply is then increased and the water supply is adjusted in order to maintain the same operating pressure. Conditions are recorded when steady state is reached. State how you know the steady state was attained. How much time was required? This procedure is repeated for six or seven conditions spanning nucleate to film boiling. Go back to two earlier conditions as a check.

5. Finally, the pressure is adjusted by varying the heating rate and cooling water flow rate and observing critical heat flux necessary to cause transition from nucleate to partial film boiling at various pressures.
6. Calculations should include heat fluxes and heat transfer coefficients as a function of temperature difference for both the heating element and condenser, critical heat flux vs pressure, and energy lost (or gained) from the system vs fluid temperature. The types of heat transfer observed should be described in your report.
7. The Software provided by P.A. Hilton contains six experiments. These experiments cannot be changed although when a data point is taken all the available data is recorded. The experiments to be performed are: Experiment 2, determination of heat flux and heat transfer coefficient at constant pressure; Experiment 3, investigation of the effect of pressure on the critical heat flux; Experiment 6, pressure—temperature relationship for a pure fluid. Experiment 1, visualization of the 3 modes of boiling is incorporated into the other experiments.
8. Note: The effect of air in a condenser is not to be done

References:

P.A. Hilton Limited. Experimental Operating and Maintenance Manual: Boiling Heat Transfer Unit H655. Hampshire, England: P.A. Hilton LTD. Dec. 1997.

6. FLUIDIZATION: Pressure drop and heat transfer in a Fluidized-Bed System

When a gas (or liquid) is passed upward through a bed of solid particles, and the fluid velocity is steadily increased, the particles eventually start to move and become suspended in the fluid. The term fluidization and fluidized-bed are used to describe the condition of fully suspended particles, since the suspension behaves as a dense fluid [1]. Due to the vigorous mixing and large contact area between gas and particles, a fully fluidized-bed has little temperature variation (bulk of the bed is practically at a uniform temperature), and gas usually has a temperature close to that of the bed. In addition, circulation of particles in the bed which is caused by rising bubbles results in large heat-transfer rates between the bed and immersed or containing heat transfer surfaces. Fluidized beds are used in a number of processes both catalytic (hydrocarbon cracking and re-forming) and non-catalytic (roasting of sulfide ores, coking of petroleum residues, incineration of sewage sludge and drying). Article by Botterill, available in the lab provides a brief review of gas-fluidized-bed systems and the relevant theory, please read the article before starting the experiment. The objectives of the present experiment are as follows:

- 1)- To investigate the relationship between bed pressure drop and air superficial velocity (upward air velocity) through a bed of granular material and to determine the minimum fluidization velocity.
- 2)- To investigate the effects of air superficial velocity, particle size of the solid material and depth of immersion on the surface heat transfer coefficient for a hot surface in an air fluidized-bed

Apparatus

Figure 3 shows a schematic diagram of fluidization and fluidized-bed heat transfer unit used in this experiment. A bed of a granular material (about 70 mm deep) is placed in a vertical glass column. A distribution chamber and an air distributor have been devised at the lower end of the column to ensure uniform air flow into the bed without causing excessive pressure drop. The air passes through the bed and leaves the system from the top after passing through a filter. The fluidized-bed system is equipped with probes for temperature and pressure measurement, and a horizontal cylindrical heating element. Probes and heating elements may be moved vertically to any level in the column. Compressed air is delivered through a filter/pressure regulator, an air flow meter fitted with a control valve and an orifice (to measure high flow rates) to the

distribution chamber. The heat transfer rate from the heating element is controlled by a variable transformer (voltage and current are displayed on the panel). Two thermocouples are embedded in the surface of element, one indicates the surface temperature and the other in conjunction with a controller, prevents the element temperature exceeding a set value. The temperature of element, supplied air and movable probe are displayed by a digital temperature indicator. The pressure of the air at any level in the bed is indicated by a liquid filled manometer. A second liquid manometer is used to determine the orifice differential pressure.

Pressure drop experiment

Procedure:

1. Ensure that all manometers have been zeroed.
2. Determine the constant for the high air flow orifice (Demonstrator will assist you with this).
3. Raise the heater to the highest position and turn its control to zero.
4. Remove the clamp and lower the thermocouple probe into the bed.
5. Pour a weighed mass (about 1.25 kg) of the chosen granular material into the bed chamber and reassemble all components.
6. Turn the air flow to a high value and allow the bed to mix thoroughly for two or three minutes.
7. Without changing the air flow, note
 - (i) The air inlet temperature t_3
 - (ii) The bed inlet temperature t_2
 - (iii) The bed height
 - (iv) The pressure drop across the bed (i.e. the difference between pressures indicated with the probe at its highest and lowest positions).
8. Reduce the air flow rate in steps and repeat the observations at each setting.
9. Having reduced the flow rate to zero, sharply tap the bed chamber with the knuckles until the bed reaches a minimum height.
10. Now repeat all the observations as the air flow is increased in similar steps.
11. At all stages make notes about the behaviour of the bed (size and behaviour of bubbles).

The test should be repeated with other sizes of particles.

Results:

1. For each material tested, plot the pressure drop (mm H₂O) versus superficial velocity (m/s) for:
 - (a) Velocity decreasing from a fluidized condition
 - (b) Velocity increasing from a compacted bed condition

Discuss the observed difference between the two curves.

2. Determine the minimum fluidization velocity for each material and discuss the effect of particle size on the minimum fluidization velocity.

Heat transfer experiment

Procedure:

1. Ensure that the manometers have been zeroed.
2. Use the clamp to position the probe thermocouple at the same level as the centre of the heater, but 15 to 20 mm to the side. (The clamp should be positioned so that in the lowest possible position, the heater centre is 20 mm above the distributor).
3. Pour a weighed mass (about 1.25 kg) of the chosen granular material into the bed chamber and reassemble all components.
4. Set the heater to a convenient height above the distributor (i.e. 40 mm).
5. Turn the air flow to a high value and allow the bed to mix thoroughly.
6. Adjust the variable transformer until the desired heater surface temperature (T_1) is attained (about 150°C is suitable).
7. Allow conditions to stabilize then record:
 - (i) Heater surface temperature T_1
 - (ii) Bed temperature T_2
 - (iii) Air temperature T_3
 - (iv) Heater voltage V
 - (v) Heater current A
 - (vi) Air flow rate, V_m (or orifice differential pressure, x)

9. Reduce the air flow rate, reset the variable transformer to obtain the desired value of T_1 , then repeat observations.
10. Repeat in convenient steps until the air control valve is closed.
11. The observations should be repeated with the heater at other heights above the distributor, (i.e. 20, 60, and 100 mm).

The test should be repeated with other sizes of particles.

Note: Provided the heater temperature of (T_1) is steady, the rate of heat transfer from the surface to the bed is the product of the voltage and current.

The bed has a large thermal inertia, and when fluidized, has little temperature variation. It is therefore not necessary to obtain complete stability of bed temperature.

Results:

1. For each material, plot the surface heat transfer coefficient (W/m^2K) versus superficial velocity (m/s) with heater height above the distributor as a parameter. Discuss the effects of superficial velocity and size of the particles on the heat transfer coefficient.
2. Calculate the maximum heat transfer coefficient for each material using Equation (3) given in the article by Botterill. Compare this with the value determined from the experiment.
3. Discuss the effect of heater height on the heat transfer coefficient.

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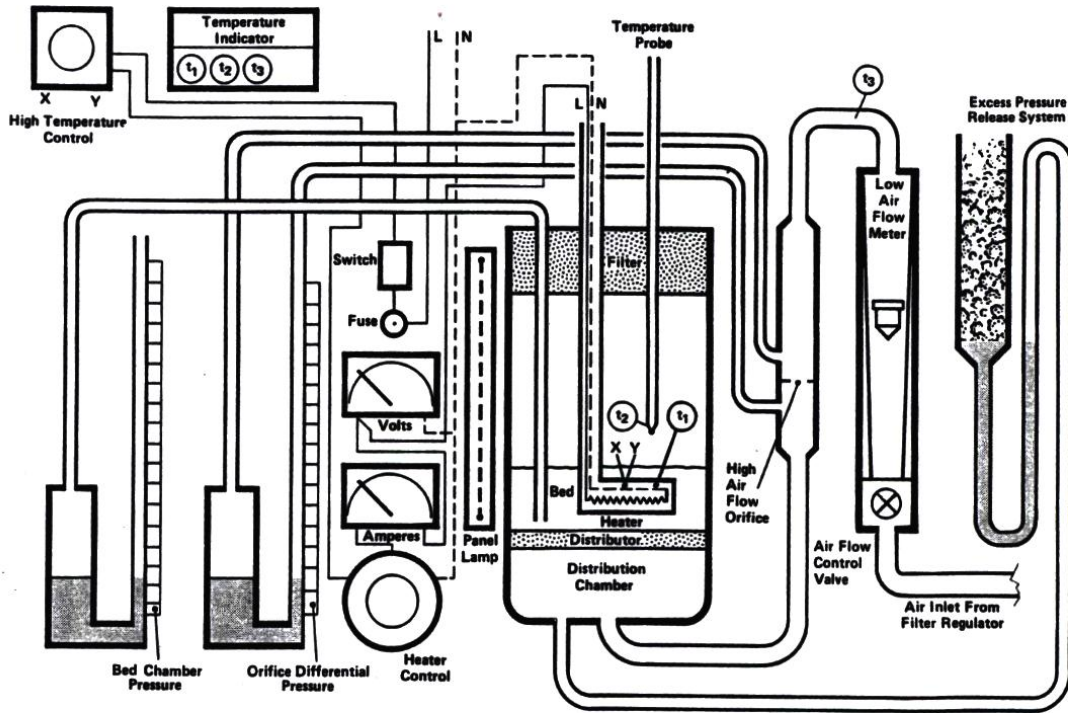


Figure 3 Schematic diagram of fluidization and fluidized-bed heat transfer unit

HILTON FLUIDIZATION AND FLUID BED HEAT TRANSFER UNIT

OBSERVATION SHEET (Pressure Drop)

Ref:

Date:

Bed Data: Material _____
 Mean particle size (dp) _____ μm
 Density of particle (solid) (Pp) _____ kg m^{-3}
 Cross sectional area (Sb) _____ m^2
 Mass of particles (M) _____ kg

Air Flow: INCREASING/DECREASING

	1	2	3	4	5	6	7	8	9	10
Orifice Differential Pressure $\frac{x}{\text{mm H}_2\text{O}}$										
Air Flow Rate (Metered) $\frac{V_m}{\text{litre s}^{-1}}$										
Air Inlet Temperature $\frac{t_3}{^\circ\text{C}}$										
Bed Temperature $\frac{t_2}{^\circ\text{C}}$										
Air Flow through Bed $\dot{V}_m \frac{T_2}{T_3} \frac{\dot{V}_b}{\text{litre s}^{-1}}$										
Superficial Velocity $\frac{10^{-3} \dot{V}_b}{S_b} \frac{U}{\text{m s}^{-1}}$										
Pressure Drop across bed $\frac{\Delta p}{\text{mm H}_2\text{O}}$										
Bed Height mm										

HILTON FLUIDIZATION AND FLUIDIZED- BED HEAT TRANSFER UNIT

Observation and Results Sheet (Heat Transfer)

Date:

Bed Data:	Material		
	Mean particle size	(d _p)	μm
	Density of particle (solid)	(ρ _p)	kg m ⁻³
	Cross sectional area	(S _b)	m ²
	Mass of particles	(M)	kg
	Initial height of bed (not compacted)	(h _i)	mm
	Surface area of heater	(A)	m ²

OBSERVATIONS

	1	2	3	4	5	6	7	8	9	10
Height of heater above distributor, h ₁ : mm										
Orifice difference pressure, x: mm H ₂ O										
Air flow rate metered, V _m : L s ⁻¹										
Heater surface temp., T ₁ : °C										
Bed temperature, T ₂ : °C										
Air inlet temp., T ₃ : °C										
Heater e.m.f., E: Volts										
Heater Current, I: Amps										

DERIVED RESULTS

Air flow through bed $V_b = V_m \frac{T_2}{T_3} \text{ L s}^{-1}$										
Superficial Velocity $U = \frac{10^{-3} V_b}{S_b} \text{ m s}^{-1}$										
Heat transfer rate, Q = E I W										
Heat transfer coefficient $h = \frac{Q}{A(t_1 - t_2)} \text{ W m}^{-2} \text{ K}^{-1}$										