Chemical Engineering Laboratory III

Ch.E. 424.2

Laboratory Manual

W2012-T2
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COURSE INFORMATION

Course objectives
In this course, experiments are chosen from the fields of process dynamics and control, reactor design, heat and mass transfer. Students will collect and analyze the experimental data using the theoretical principles of heat and mass transfer, reaction engineering and process control. 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. Chemical Reactors
2. Cooling Tower
3. Solid Leaching
4. Process Identification and Control of a Liquid Surge Tank
5. Biodiesel - Transesterification

Laboratory reports
Each partner in the group is to submit one formal report, one brief technical report and two technical memos 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. The guidelines for preparation of reports can be found in the laboratory manual, as well as on the course website. 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!

This year in addition to submission of a printed copy, students are expected to email an electronic copy of their report to the instructor. Dr. Niu will be marking Cooling Tower, Solid Leaching and Biodiesel (section ), Dr. Kennell will be marking Chemical Reactors, Process Control and Biodiesel (section )

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/dept/cen/safety_toc.html). Each student must hand in a signed safety release form to the Laboratory Coordinator before they will be permitted to perform experiments in ChE 424. The form is available on the safety website. 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 0.5% per day for any late report will be deducted from the full marks of this course.* In this course, each student will be given 7 “free” late hand-in days 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.*
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 Honesty/Dishonesty 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" [1]. Proper technical report writing is nicely described in the book “Technically Write” [2]. 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

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 date, 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 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 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 procedure. The procedure 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.
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 procedures used 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 or a graph could be included to support the conclusions. The text should not exceed 
two double-spaced typewritten pages. See the Chapter “Informal Reports Describing Facts and 
Events” in Blicq.

Cited references

   (Library Call No. T 10.5, E27).

2. Blicq, R.S. Technically write, Communicating in a technological era. Prentice-Hall Canada. 

Other references

Jeter, S. M. Writing style and standards in undergraduate reports. Glen Allen, Va.: College Pub., 
EXPERIMENTS

1 - Chemical Reactors

The mathematical models proposed for ideal plug-flow and continuous stirred-tank reactors do not include the effects of the flow pattern on conversion. In a PFR the velocity profile is assumed to be flat and the dispersion of reactants and products by turbulence and diffusion is neglected. In a CSTR all reactants and products are presumed to be perfectly mixed and the reactor contents as soon as they enter the vessel. These requirements for the idealized models are usually not met in practice.

The back-mixing created by turbulence and diffusion in a tubular reactor produces a conversion which is lower than predicted by the plug-flow model. Since back-mixing is not as extensive as in a stirred tank, conversion in a tubular reactor is often higher than conversion in a CSTR.

The deviation of an actual tubular reactor from the plug-flow model is a function of the bulk velocity. In a laminar flow, where velocity profile is parabolic, the deviation is largest. Under conditions of fully developed turbulence ($Re \approx 10,000$) the velocity profile is more flat and the plug-flow model is more closely approximated.

In this experiment, the deviation of experimental reactors from idealized models will be illustrated. The use of a batch reactor to generate data for the determination of kinetic parameters will also be considered.

Theory

For this experiment, the reaction performed is the saponification of ethyl acetate. This reaction is elementary and second-order. The reaction equation is:

$$\text{NaOH} + \text{EtOAc} \rightarrow \text{NaOAc} + \text{EtOH}$$

$$A + B \rightarrow C + D$$

(A) Batch Reactor

For a constant volume isothermal batch reactor, the design equation is:

$$\frac{dC_A}{dt} = r_A$$

For a bimolecular second order reaction, the rate equation is:

$$-r_A = k \cdot C_A \cdot C_B$$

In the case where equal amounts of A and B are charged to the reactor (i.e. $C_A = C_B$), the following stoichiometry table applies:
<table>
<thead>
<tr>
<th>SPECIES</th>
<th>INITIAL</th>
<th>CHANGE</th>
<th>FINAL</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>$N_{Ao}$</td>
<td>$-XN_{Ao}$</td>
<td>$N_{Ao}(1-X)$</td>
</tr>
<tr>
<td>B</td>
<td>$N_{Bo} = N_{Ao}$</td>
<td>$-XN_{Ao}$</td>
<td>$N_{Ao}(1-X)$</td>
</tr>
<tr>
<td>C</td>
<td>$N_{Co} = 0$</td>
<td>$+XN_{Ao}$</td>
<td>$N_{Ao}X$</td>
</tr>
<tr>
<td>D</td>
<td>$N_{Do} = 0$</td>
<td>$+XN_{Ao}$</td>
<td>$N_{Ao}X$</td>
</tr>
<tr>
<td></td>
<td>$N_{To}$</td>
<td>0</td>
<td>$N_{To} = N_T$</td>
</tr>
</tbody>
</table>

For this stoichiometric table, $X$ represents the fractional conversion of species $A$.

PLEASE NOTE THAT THE INITIAL CONCENTRATIONS WILL NOT BE THE SAME. YOU WILL BE REQUIRED TO ADJUST THE EQUATIONS AND YOUR ANALYSES ACCORDINGLY.

The design and rate equations are written in terms of concentrations. Thus, we need to use the relations:

$$C_A = C_{Ao} (1 - X)$$
$$C_B = C_{Bo} (1 - X)$$

If we combine the above equations, we see that:

$$\frac{dC_A}{dt} = r_A = -kC_{Ao}^2 (1 - X)^2$$

As well,

$$\frac{dC_A}{dt} = -C_{Ao} \frac{dX}{dt}$$

Therefore,

$$\frac{dX}{dt} = kC_{Ao} (1 - X)^2$$

If both sides of this equation are integrated...

$$\int_0^X \frac{dX}{(1 - X)^2} = kC_{Ao} \int_0^t dt$$

...we see that:

$$\frac{X}{1 - X} = ktC_{Ao} \quad (1)$$
Conductivity Measurements

The conductivity probe produces a voltage which is related to the concentration of ions in the solution.

\[ V_t = K \sum_i C_i \ell_i \]

Where \( V_t \) is the voltage at time \( t \), \( C_i \) is the concentration of \( i \) (gmol/L), \( \ell_i \) is the conductance of species of \( i \) (S·cm\(^2\)/gmol), and \( K \) is a constant of proportionality.

There are three ions present in the system: \( \text{OH}^- \), \( \text{CH}_3\text{COO}^- \), and \( \text{Na}^+ \). By defining each concentration in relation to the initial concentration of NaOH introduced to the system, the concentration of each species is defined as follows:

\[
\begin{align*}
C_{\text{OH}^-} &= C_A = C_{\text{Ao}} (1 - X) \\
C_{\text{CH}_3\text{COO}^-} &= C_C = C_{\text{Ao}} X \\
C_{\text{Na}^+} &= C_A + C_C = C_{\text{Ao}}
\end{align*}
\]

Therefore, the total voltage that is read by the probe is defined as:

\[
\begin{align*}
V_t &= K \left[ C_{\text{Ao}} (1 - X) \ell_{\text{OH}^-} + C_{\text{Ao}} X \ell_{\text{CH}_3\text{COO}^-} + C_{\text{Ao}} \ell_{\text{Na}^+} \right] \\
V_t &= K C_{\text{Ao}} \left[ (1 - X) \ell_{\text{OH}^-} + X \ell_{\text{CH}_3\text{COO}^-} + \ell_{\text{Na}^+} \right]
\end{align*}
\]

At the start of the experiment, \( X = 0 \) and the voltage can be defined as \( V_t = V_o \).

\[
V_o = K C_{\text{Ao}} \left[ \ell_{\text{OH}^-} + \ell_{\text{Na}^+} \right]
\]

At the point where conversion is complete (PROVIDED SPECIES A IS THE LIMITING REAGENT), \( X = 1 \) and the voltage can be defined as \( V_t = V_c \).

\[
V_c = K C_{\text{Ao}} \left[ \ell_{\text{CH}_3\text{COO}^-} + \ell_{\text{Na}^+} \right]
\]

Thus, at any time during the experiment:

\[
V_o - V_t = K C_{\text{Ao}} \left[ X \ell_{\text{OH}^-} - X \ell_{\text{CH}_3\text{COO}^-} \right] = K C_{\text{Ao}} X \left[ \ell_{\text{OH}^-} - \ell_{\text{CH}_3\text{COO}^-} \right]
\]

For the unique case of complete \( A \) conversion:
Therefore, at any time $t$:

$$\frac{V_o - V_t}{V_o - V_c} = X = \frac{C_{Ao} - C_A}{C_{Ao}}$$

(2)

If we substitute equation (2) into equation (1), the resulting equation is:

$$\frac{X}{1 - X} = \frac{V_o - V_t}{V_o - V_c} = C_{Ao} k t$$

Finally, when the term $V_t$ is isolated, the following linear equation is produced:

$$V_t = \left(\frac{V_o - V_t}{t}\right) \left(\frac{1}{k C_{Ao}}\right) + V_c$$

(3)

From a plot of $V_t$ against $(V_o - V_t)/t$, the result is a straight line with a slope of $1/k C_{Ao}$. From the value for this slope, the rate constant for the reaction can be determined.

**Experimental Procedure**

**(A) Batch Reactor**

DO THIS SECTION LAST. BE SURE TO SAVE ENOUGH FEED SOLUTION IN THE PROVIDED PAILS.

In this section, the conversion in a batch reactor is to be determined as a function of time. The suggested procedure for this section is outlined below.

**Procedure**

1) Drain approximately 2.0 gallons of each reagent from the feed tanks and store in the pails provided until the end of the experiment. Weigh out equal volumes of solution so that volume ratios are preserved. BE SURE TO NOTE WHICH REACTANT WILL BE THE LIMITING REAGENT AND CONSIDER HOW THIS WILL AFFECT ASSUMPTIONS MADE IN YOUR ANALYSES.

2) Mount the dip-type conductivity probe within the batch vessel.
3) Pour the sodium hydroxide solution into the reactor.

4) Start the electric stir rod.

5) Pour the ethyl acetate solution into the reactor and begin digitally recording voltage data for the batch reaction.

6) Record the voltage readings at 10-second intervals during the initial minutes of the reaction and at longer time intervals as the reaction progresses. Collect batch data for approximately 40-45 minutes before allowing the reaction to proceed overnight.

7) Record one final voltage reading the following morning. It can be assumed at this time that the maximum potential reaction conversion has been achieved.

8) Drain the reactor contents and clean the vessel.

(B) **Continuous Stirred-Tank Reactors (CSTRs)**

In this section, the conversion in a CSTR system is compared to the conversion in a batch reactor. Two 180-liter tanks of prepared solution should be ready for you in the laboratory. The concentration of NaOH and ethyl acetate will be supplied by the TA. The reagents can be reacted at either equivalent volumetric flows or at equivalent rotameter readings.

**Procedure**

1) Start the pumps and set the rotameter reading at 70 for either one or both of the rotameters in order to operate at either equivalent volumetric flows or rotameter readings.

2) Once both tanks are filled with solution, begin recording the transient conductivity readings in both of the CSTRs at consistent time intervals. Do this until it is observed that the conductivity readings for both tanks have reached steady-state.

3) Change the flow rate setting from 70 to 60 on either one or both of the rotameters (depending on your procedure). Record the transient conductivities in both CSTRs and determine the steady-state conductivities in the two reactors. Observe any potential volume changes in either reactor.

4) Repeat step 3) for at least 2 lower and one higher flow rate settings. Ensure that your flowrate settings include 10 and 100.

5) Manually record the temperatures in each CSTR at each steady-state conversion.

6) Once the feed tanks are empty or near empty, shut off the pumps, close the valves at the base of the tanks, and fill both of the tanks with tap water.
(C) **Tubular Reactor**

In this section the deviation of flow patterns in a tubular reactor from ideal plug-flow behavior will be qualitatively observed. A suggested procedure is outlined below.

1) Open the valves at the base of the tanks and begin pumping water through the PFR.

2) Set the flow rate at a reading of 50 for both rotameters.

3) Inject approximately 5 mL of concentrated KMnO$_4$ solution at the entrance `T` of the reactor using the syringe provided.

4) Estimate the length of the colored region at the entrance, middle, and exit of the reactor.

5) Repeat steps 2) and 3) for rotameter readings of 10 and 100. Qualitatively compare the lengths of the colored regions between the three flow rates.

**Report**

1) Compare the conversion in a batch reactor with the conversion from the CSTRs in series for the same reaction time.

2) Determine the reaction rate constant from the batch reaction results. Compare the steady-state conversions measured in the CSTRs to those predicted by applying the determined rate constant. Perform an error analysis.

3) Formulate a model for the in-series CSTRs capable of predicting the TRANSIENT conversion in each CSTR due to a step-change in reactant flow rate. i.e. \( \frac{dX_1}{dt} = f[X_1, k, C_{Ao} \ldots \text{ etc.}] \); \( \frac{dX_2}{dt} = f[X_1, X_2, k, C_{Ao} \ldots \text{ etc.}] \)

4) Compare the experimental transient response with the model prediction. Discuss possible explanations for the discrepancy between each profile, if any.

5) Comment on the differences in flow pattern that exist at various flow rates within the tubular reactor. Discuss how these various flow patterns could affect the assumption made about a PFR.

**References**


Figure 3.1: Tubular Reactor

Na OH

Et OAc

CONDUCTIVITY CELL

FLOW TUBE
LENGTH = 60.5 ft.

TUBE DIAMETER = 1.01 in.

TO DRAIN

Tobular Reactor
2 Cooling Tower

**Object:** To measure the ability of air to cool warm water.

**Introduction:** The Chemical Process Industries (CPI) pump vast quantities of water through heat exchangers in order to cool hot processing fluids. The cool, source water leaves these heat exchangers at elevated temperatures. In an attempt to reduce the discharge of hot water into the aquatic environment, the CPI utilizes cooling towers to cool and recycle the water. Cooling towers utilize ambient air as the medium to remove thermal energy from the warm water. Most of that energy is transferred by evaporation. And although this seems to violate the laws of thermodynamics, the air often cools the water to temperatures below the temperature of the incoming ambient air (especially in dry climates such as Saskatchewan). This is because the water can be cooled to the wet bulb temperature of the incoming air.

**Industrial Cooling Towers:** Many people relate cooling towers to the nuclear power industry where huge, natural draft chimneys often dominate the skyline:

![Natural Draft Chimneys Above Cooling Towers](http://www.ocrwm.doe.gov/youth/problem.shtml)

The actual cooling process occurs below the chimneys, and for fan assisted cooling towers these chimneys do not exist:

![Fan Assisted Cooling Tower](http://www.coolingtowersystems.com/towergallery.htm)

However the air is moved through the cooling towers, there are two types of flow patterns that can occur, counter current and cross current as demonstrated in the sketch below:
Although chemical engineers know that counter current flow provides optimum energy transfer capabilities, the majority of modern, industrial cooling towers operate with a cross current flow pattern due to mechanical considerations. But in this lab, students will study the performance of the P.A. Hilton Model H892 cooling tower system which utilizes counter current flow technology.

**Theory for Counter Current Flow Cooling Towers** (see Bennett and Myers: “Momentum, Heat and Mass Transfer”, 3rd Edition, McGraw-Hill, 1982, 644 - 648): The following summarizes the important equations that need to be solved to model the performance of a cooling tower. More information is available from Bennett and Myers. The course website also has an Excel spreadsheet that provides a solution of the theory noted below:

Considering a thin, cross sectional slice taken perpendicular to the flow, a mass and energy balance can be written as:

\[
\frac{dY_A}{dz} = \frac{Y_A - Y_{AS}}{H_{GB}}
\]

(1)

\[
\frac{dT_Y}{dz} = \frac{T_Y - T_S}{H_{GB}}
\]

(2)

.. where \(H_{GB}\) is the height of a mass transfer unit based on the gas phase and is the ratio of the gas flux \((G_B)\) divided by the volumetric mass transfer coefficient \((k_ya)\), \(Y_A\) represents humidity \((kg \text{ H}_2\text{O} / kg \text{ dry air})\), \(Y_{AS}\) is saturated humidity at \(T_Y\), \(z\) is position up the tower, \(T_Y\) is air temperature and \(T_S\) is the adiabatic saturated air temperature corresponding to \(T_Y\) and \(Y_A\). In these equations it has been assumed the height of a thermal transfer unit is the same as that for mass, which is approximately true for moist air. Besides writing balances in terms of driving
forces, the energy lost in the water phase must equal the energy gained in the air phase expressed as:

\[ -L_c \times C_{px} \times \frac{dT_x}{dz} = G_b \times C_{ph} \times \frac{dT_x}{dz} + G_b \times \lambda_r \times \frac{dY_A}{dz} \]  \hspace{1cm} (3)

… where L_c is the water flux in the column, C_{px} is the heat capacity of the water, T_x is the water temperature, C_{ph} is the heat capacity of moist air and \( \lambda_r \) is the energy of vaporization of water at 0 °C.

Substituting Equations 1 and 2 into Equation 3 gives:

\[ -L_c \times C_{px} \times \frac{dT_x}{dz} = \frac{G_b}{H_{gb}} \times [C_{ph} \times (T_y - T_s) + \lambda_r \times (Y_A - Y_A)] \]  \hspace{1cm} (3B)

The term in brackets on the RHS of the above equation is just the difference in the enthalpy of air from saturated conditions, so we can rewrite Equation 3B as:

\[ -L_c \times C_{px} \times \frac{dT_x}{dz} = \frac{G_b}{H_{gb}} \times [H_y - H_{ys}] \]  \hspace{1cm} (3C)

We can now introduce another energy balance over dz, equating liquid enthalpy change to gas enthalpy change:

\[ -L_c \times C_{px} \times \frac{dT_x}{dz} = G_b \times \frac{dH_y}{dz} \]  \hspace{1cm} (4)

or:

\[ \frac{dH_y}{dT_x} = \frac{L_c}{G_b} \times C_{px} \]  \hspace{1cm} (5)

Equation 5 represents the operating line equation for a cooling tower. It is linear and can be written in the form:

\[ H_y = H_{y0} + \frac{C_{px} \times L_c}{G_b} \times (T_x - T_{x0}) \]  \hspace{1cm} (5A)

.. where “0” represents the bottom of the column and “1” represents the top of the column. Since we know the condition of the inlet air, H_{y0}, and the desired outlet water temperature at the bottom of the tower, T_{x0}, Equation 5A gives us the value of H_y at any water temperature, T_x, up the column until we reach the inlet water temperature, T_{x1}, which will give us the enthalpy of the air exiting the column, H_{y1}. In addition, there is an “equilibrium” correlation (Eq. 6), which can be used to calculate the values of the saturated air enthalpy at each water temperature T_x

\[ H_{ys} = 0.00229 \times T_x^3 - 0.0767 \times T_x^2 + 3.63 \times T_x - 2.385 \]  \hspace{1cm} (6)
.. where in Equation 6, $H_{YS}$ is in kJ/kg and $T_X$ is in °C. In order to calculate the height of packing, you need to combine Equations 3c and 4 to give:

$$- \frac{dH_Y}{dz} = \frac{H_Y - H_{YS}}{H_{GB}}$$  \hspace{1cm} (4A)$$

Equation 4A can then be solved to find the total height of the cooling tower, $Z$:

$$Z = H_{GB} \times \int_{H_{Y0}}^{H_{Y1}} \frac{dH_Y}{H_{YS} - H_Y}$$  \hspace{1cm} (7)$$

The integral in Equation 7 represents the number of transfer units, $n_{OG}$. In this experiment, you know the value of $Z$ and since the column is so short, you can approximate the integral in the above equation from:

$$n_{OG} = 0.5 \times \left( \frac{1}{H_{YS,1} - H_{Y1}} + \frac{1}{H_{YS,0} - H_{Y0}} \right) \times (H_{Y1} - H_{Y0})$$  \hspace{1cm} (8)$$

Since the enthalpy values at each end of the column can be determined from the experimental measurements, Equation 8 can be solved which then allows you to use Equation 7 to calculate $H_{GB}$ for each operating condition you use.

Another important line to put on the enthalpy versus temperature graph is $H_Y$ vs $T_Y$ from a relationship generated by combining Equations 2 and 4A:

$$\frac{dH_Y}{H_{YS} - H_Y} = \frac{dT_Y}{T_S - T_Y}$$  \hspace{1cm} (9)$$

If a plot of $H_Y$ vs $T_Y$ crosses the $H_{YS}$ vs $T_X$ line, the cooling tower will fog and you will see a water vapor stream coming from the top of the cooling tower, or else the mist eliminator will be soaked with water.

To get an estimate of $H_{GB}$ at each operating condition, you can try the correlation of Sherwood and Holloway for wooden slats:

$$k_ya = 0.00105 \times G_b^{0.66} \times L_c^{0.61}$$  \hspace{1cm} (10)$$

and then:

$$H_{GB} = \frac{\bar{G}_b}{k_ya}$$  \hspace{1cm} (11)$$

… where in Equations 10 and 11, $G_B$ and $L_C$ are in units of lb mol/h-ft^2, $k_ya$ is in lb moles/h-ft^3 and $\bar{G}_b$ is in units of lb moles/h-ft^2.
Apparatus and Methods:

The sketch below shows the experimental apparatus used in this investigation:

The Hilton Model H892 Cooling Tower System

The system consists of a column packed with slats, a water reservoir with heaters to heat the water, a centrifugal pump to circulate the water and an air fan to push air up the column. Thermocouples exist to measure temperatures as well as pressure taps to measure air pressure drop and air flowrate; and a rotameter to measure water flowrate. The system should have been filled with distilled water (make up tank filled near the top) and the centrifugal pump inside the steel housing should have been bled of air prior to the lab schedule. You should see water to a high level in the Make Up tank. The level of water in the Make Up tank is an important variable in this study as well as the air and water flowrates, temperatures of both the air (wet and dry bulb) and the water at the top \( (t_3, t_4, t_5) \) and the bottom \( (t_1, t_2, t_6) \) of the column.

Procedures:

1. Begin by setting the air flowrate to maximum (slide fan damper wide open) and water flowrate to maximum (rotameter valve wide open) for about 5 minutes in order to fully wet the slats inside the column.
2. The water flowrate will then be set to four random values between 10 to 50 g/s at each of two air flowrates, that will be set at two random values between 3 mm and 25 mm water pressure drop as measured at the top column pressure port. The lab instructor will tell you what air and water flowrate values your team will use and what thermal power input your team will use (one of 0.5, 1.0 or 1.5 kW). Note the electrical power input represents the heat exchangers of a chemical process.
3. At each combination of flowrates (a total of 8 combinations) you will wait until the column achieves steady state, likely about 15 minutes. Steady state will be assumed to have been achieved when the temperatures of the air and water streams do not change over a period of 2 minutes.
4. At each steady state, record the water and air flowrates, the temperatures (six values), the pressure drop across the column (measure pressure right above and below the packing) and the rate of water consumption (due to evaporation) from the make up tank (for this latter measurement, determine the weight of water loss in the Make Up tank after a
measured time interval by pouring a measured mass of water into the Make Up tank to bring it back to a previous observed level).

5. Once all your data has been collected, shut off the heaters and the power main. Do not forget to have your lab record book initialed and dated by the lab instructor before you leave.

Data Analysis: In this experiment you know the packing height and need to use your data to determine the experimental overall height of a transfer unit, $H_{GB}$, for each operating condition (use Equations 7 and 8 above); and compare that to the “expected” values of $H_{GB}$ (Equations 10 and 11 but watch your units). Then plot and determine how $H_{GB}$ changes as a function of both air and water flowrates through the column. In order to do those analyses, you will have to compute air enthalpies in and out of the column. You should also do a mass and energy balance check for each operating condition. You know how much energy you put into the water from the electric heaters (add in 0.1 kW from the centrifugal pump) and then see if all that energy is captured by the air. You also know the rate of water loss in the make-up tank, and check to see if all that water is exiting in the air. Do these checks at all operating conditions. The air flowrate is determined by the pressure drop ($\Delta P$) across an orifice measured in mm H$_2$O and the use of an empirical equation:

$$m_{\text{AIR}} = 0.0137 \times \frac{\Delta P}{\sqrt{V_1 \times (1 + Y_{\text{AIR}})}}$$  \hspace{1cm} (12)

… where $V_1$ is the specific volume of the dry air at the exit of the column in m$^3$/kg and $m_{\text{AIR}}$ is the mass flowrate of dry air in kg/s, which is constant through the column.

Design Project: Assume that you will build a large scale cooling tower for a new heavy oil upgrader to be located in Saskatoon using the same slat material and therefore the same $H_{GB}$ values as you determined experimentally. Design the tower (area and height) to cool 20000 m$^3$/day of warm water at 37 °C to 22 °C (use the average July max temperature of 25 °C air dry bulb temperature and average relative afternoon humidity of 47% as your inlet air condition).
3 – Solid Leaching

The objective of this experiment is to examine leaching, a process of extracting valued chemicals from solids into liquids. Although chemical engineers mostly process gases and liquids, many of the chemicals needed by society are found inside solid particles. Examples include uranium in ore, oil in canola seed and sugar in sugar cane. Chemical engineers often begin the processing steps by first moving the desired chemical from the solid into a liquid; this is called leaching.

Theory

Counter Current Flow Leaching:

This analysis assumes the presence of three components: 1) an inert solid material called the raffinate, Phase C; 2) The chemical in the solid we want to extract called the solute, Species A; and 3) the extraction liquid used to extract Species A called the solvent, Phase B. In this analysis, we assume that Phase C is not soluble in Phase B, but that Phase B can remain to some extent with Phase C as it exits from each stage of the extraction process. Species A leaves each stage of the extractor mixed with Phase B and is at the same concentration in Phase B in both the overflow (the liquid effluent from each stage which is assumed to have no Phase C) and in the liquid in the underflow (solid effluent from each stage). A schematic of the extraction model for a single stage extractor is shown below:

![Figure 1. Schematic of a single stage extractor.](image)

The amount of solid per unit liquid in the underflow, \( X_C \) (kg solid/kg liquid), is assumed to be a known “equilibrium” function of the concentration of Species A in the liquid, \( x_A \) (mass fraction). IT IS ASSUMED FOR THE PURPOSE OF PARAMETER \( X_C \) THAT SPECIES A IS ALWAYS REGARDED AS A LIQUID AND NOT A SOLID. A typical equilibrium relationship is shown in the following figure as the underflow line:
Since Species A is at the same concentration in the underflow and overflow, and there are only two species in the liquid:

\[ x_{A1} = y_{A1} \]  
\[ x_{A1} + x_{B1} = 1.0 \]

Mass balances around Figure 1 yield the following three relations (Phase C is not included in \( L \), the underflow, and \( V \), the overflow):

\[ L_0 + V_2 = L_1 + V_1 \]  
\[ (L_0 \cdot x_{A0}) + (V_2 \cdot y_{A2}) = (L_1 \cdot x_{A1}) + (V_1 \cdot y_{A1}) \]  
\[ L_0 \cdot X_{C0} = L_1 \cdot X_{C1} \]

Figure 2 will produce an equation of the type:

\[ X_C = f(x_A) \]

Assuming the feed flow rates, feed concentrations, and equation (6) function are known, then equations (1) and (3)-(6) are five equations with five unknowns \((x_{A1}, y_{A1}, L_1, V_1, \text{ and } X_{C1})\). These can be solved fairly easily for a single-stage operation, but if we run several stages in the counterflow fashion (counterflow is the most optimum mass transfer strategy for the same reason as counterflow heat transfer is the most optimum energy transfer strategy), there becomes too many equations and unknowns to solve in this fashion. However, a much more convenient way to solve continuous, counterflow leaching is graphically. Consider the general system sketched below with a total of \( N \) extraction stages:
We let the sum of the feed flows be equal to \( M \), and by a mass balance:

\[
M = L_0 + V_{N+1} = L_N + V_i
\]  

Equation (7)

Similarly, for a mass balance of Species A:

\[
(L_0 \cdot x_{A0}) + (V_{N+1} \cdot y_{AN+1}) = M \cdot z_{AM} = (L_N \cdot x_{AN}) + (V_1 \cdot y_{A1})
\]  

Equation (8)

Equations 7 and 8 allow calculation of the mixing flow rate, \( M \), and the mixed concentration, \( z_{AM} \), so they are both known quantities. From the following, \( X_{CM} \) (the mixing solids fraction) is defined by:

\[
L_0 \cdot X_{C0} = M \cdot X_{CM} = L_N \cdot X_{CN}
\]  

Equation (9)

We define the delta point, \( \Delta \), as the difference in flow of streams flowing past each other:

\[
\Delta = L_0 - V_1 = L_N - V_{N+1} = L_i - V_{i+1}
\]  

Equation (10)

This value is a constant. The graphical solution of multistage countercurrent extractions is performed on a graph like Figure 2 with known underflow and overflow lines. The coordinates of \( L_0 \) are known: \((x_{A0}, X_{C0})\). The coordinates of \( V_{N+1} \) are known: \((y_{AN+1}, 0)\). The coordinates of \( M \) are known: \((x_{AM}, X_{CM})\). We can place these three points on the graph and will find that \( M \) lies on a straight line joining \( L_0 \) and \( V_{N+1} \). Finally, the coordinates of the delta point, \( \Delta \), on this graph are:

\[
x_{A\Delta} = \frac{(L_0 \cdot x_{A0}) - (V_1 \cdot y_{A1})}{L_0 - V_1} = \frac{(L_N \cdot x_{AN}) - (V_{N+1} \cdot y_{AN+1})}{L_N - V_{N+1}}
\]  

Equation (11)
$X_{CA} = \frac{X_{C0} \cdot L_0}{L_0 - V_1}$

(12)

Unlike the positions of $L_0$, $V_{N+1}$, and $M$, we can’t locate the delta point since the values of $V_I$ and $L_N$ are not known. However, we need to find the coordinates of this point in order to complete the graphical solution.

Rather than go through the theory of the graphical method, the following is an example which can help demonstrate the technique. In the example, we are using leaching to remove oil (the solute, Species A) from a crushed and previously mechanically extracted seed, Phase C. The seed/oil feed mixture flows at 100 kg/h and is 14% oil by mass ($x_{A0} = 1.0$, $X_{C0} = 86/14 = 6.143$). We wish to determine the number of countercurrent flow stages we will need to get the oil loss in the final raffinate to 1.0 kg/h for the 14 kg/h of oil we fed to the start of the leaching process. This is not like most practical problems. Usually, you know the number of stages and want to compute the loss of solute in the raffinate coming from the last stage. But you still have to use the same graphical approach as shown here. You have to guess the final solute loss, and then graphically solve to see if the number of stages you get equals the known number of stages. If not, make a new estimate of the solute loss in the final raffinate, solve graphically, etc.

**Example**

The seed mixture (oil and seed) defined in the above paragraph flow at a 100 kg/h into the first stage ($L_0 = 14$ kg/h) and a pure hexane flow of 80 kg/h is used to extract the oil (Phase B: $y_{AN+1} = 0$, $V_{N+1} = 80$). Each stage of our extractor reaches equilibrium and the relationship between Species A weight fraction the solid-to-liquid ratio is:

$$X_C = 1.5 - (0.2 \cdot x_A)$$

We begin by producing a graph and plotting the above equation and the points $L_0$, $V_{N+1}$ and $M$ as shown in Figure 4. We note that $M$ falls on the straight line connecting $L_0$ and $V_{N+1}$, as it should. The coordinates of $M$ come from equation (7) to get the value of $M$ ($14 + 80 = 94$), equation (8) to get the value of $z_{AM}$ ($14/94 = 0.149$) and equation (9) to get the value of $X_{CM}$ ($14 \times 6.143/94 = 0.915$).
It turns out that we can also locate the point $L_N$ on this graph using a trick. We know that $L_N$ must lie on the underflow equilibrium line for $X_C$, but what is $X_{AN}$? Consider the ratio:

$$\frac{X_{CN}}{x_{AN}} = \frac{(kg \cdot solids)/(kg \cdot liquid)}{(kg \cdot oil)/(kg \cdot liquid)} = \frac{kg \cdot solids}{kg \cdot oil}$$

But we know what the ratio of $(kg \cdot solids)/(kg \cdot oil)$ coming out the underflow of the last stage is, since all 86 kg/h of solids is there and we have 1 kg/h of oil we are losing, so the ratio is 86. But we also know $X_{CN} = 1.5 - (0.2 \times x_{AN})$. So we have two equations and two unknowns from which we compute $X_{CN} = 1.4965$ and $x_{AN} = 0.0174$. Now we can place the LN point on our graph (see Figure 4).

$V_1$ is the other exit stream from the extraction stages and it must lie on a straight line with both $L_N$ and $M$. It will also lie on the overflow line (i.e., the axis, where $X_C$ is zero). This gives the value of $y_{A1} = 0.36$ on Figure 4. Note that all $V$ points lie on the overflow line (since no solids are in the liquid streams) and all $L$ points (except $L_0$) lie on the underflow line.

Now we can calculate the values of $L_N$ and $V_1$ using total mass and species A balances around the whole extractions system:

$$L_N + V_1 = M = 94$$

$$(L_N \cdot x_{AN}) + (V_1 \cdot y_{A1}) = L_0 \cdot x_{A0} = 14 \quad \Rightarrow \quad (L_N \times 0.0174) + (V_1 \times 0.36) = 14$$

This leaves two equations with two unknowns. Solving gives the results of $L_N = 57.9$ kg/h and $V_1 = 36.1$ kg/h. With knowledge of these variables, we can compute the location of the delta point.
\[ x_{AA} = \frac{(57.9 \times 0.0174) - 0}{57.9 - 80} = -0.0456 \]

\[ X_{CA} = \frac{86}{14 - 36.1} = -3.89 \]

Now we can step off the stage by stage extraction calculations starting with Figure 5 that contains the points \( L_0, V_1, \Delta, \text{ and } L_N \). Note that \( L_0, V_1, \text{ and } \Delta \) lie on a straight line since they are streams passing each other in the extraction system. But since \( x_{AI} = y_{AI} \), we locate the point \( L_1 \) on the underflow line vertically above the point \( V_1 \). Now \( L_1 \) and \( V_2 \) lie on a straight line with \( \Delta \) since they are streams passing each other, and also \( V_2 \) is on the overflow line (axes), so we get the point \( V_2 \). We continue until the last vertical line hits \( L_N \), which in this case is at the 7\(^{th} \) stage as shown in Figure 5. So for this example, it would take 7 countercurrent leaching stages to get the oil down to the desired loss level in the overflow.

**Figure 5.** Graphical solution of countercurrent leaching.

**Apparatus and Methods**

On the following page is a schematic of the experimental apparatus used in this investigation:
NOTE THAT THE ARMFIELD MANUALS CONTAIN DETAILED INFORMATION FOR YOU TO STUDY. THE SYSTEM IS COMPUTER CONTROLLED. AS WITH ALL EXPERIMENTS, SAFETY ISSUES AND PROPER SHUT DOWN PROCEDURES ARE VERY IMPORTANT. BE SURE YOU READ AND UNDERSTAND THESE ISSUES AND PROCEDURES.

Figure 6. The Armfield UOP4 Mk II Solid/Liquid Leaching Unit
Procedure

A. Determination of solid to liquid ratio of KHCO₃ (Species A) solutions (this procedure can be carried out while you are running the leaching apparatus. See step B below).

1) In a beaker, mix an equal volume of water with a known weight of dry beads that contains no KHCO₃. Drain the beads on a screen, similar to the screen in the Armstrong Leaching Apparatus, for several minutes. Weigh the wet beads, the increase in weight of the beads is the water attached to the "raffinate" at $x_A = 0$.

2) Place a known weight of dry beads containing KHCO₃ in a dry beaker. Add a known volume of water (approximately 25% of the volume of the beads) and thoroughly mix the beads and water. Allow for at least 10 minutes for the beads to become thoroughly washed with the water. Drain the water from the beads through a screen for two minutes, measure the KHCO₃ concentration of the water (use the calibrated conductivity probe) and the weight of the wet beads. Repeat this step, gathering $x_A$ and $X_C$ data, for at least four different mixtures where you increase the volume of water using approximately the same initial mass of dry, KHCO₃ containing beads. This will generate your underflow line.

B. Operating the counterflow leaching apparatus.

1) Fill the solids hopper with a known weight of dry beads containing KHCO₃ (if the hopper begins to run out of pellets during operation, be sure to weigh any quantities you add to the solids hopper). Ensure the solvent holding tank is full of RO water. Ask the TA what feed rate of solids, how many spray (leaching) stages you will use (you will complete two runs, either 1 & 3 stages, 1 & 2 stages, or 2 & 3 stages), as well as the temperature and water flow rate you will use in your experiment. These values will not change for the duration of your study. Note that when performing multiple stage extraction, all pumps will operate at the same flow rates and all water will be at the same temperature). The TA will provide you with the initial concentration of KHCO₃ in the beads.

2) Start the rotor and operate it at its maximum speed, approximately 4 rpm. The TA will advise what setting to use in order to achieve this speed and what value that rotation speed actually is. Turn the solids feeder on and measure the time the feeder runs.

3) Just before the solids reach the first spray stage that you will use, turn on the water and ensure it is set at the flow rate and temperature you have been advised to use.

4) Measure the KHCO₃ concentrations in the overflow from each stage as a function of time until steady-state is reached (conductivity probes which have been calibrated are used). Each run should be allowed approximately 30 minutes to achieve steady-state.

5) Use at least 2 weighed samples of the effluent beads to determine the amount of water left in the final effluent raffinate (weigh the beads before and after drying overnight). This will be done for both experimental runs performed.
6) Using 2 more weighed samples of the underflow product (wet effluent beads), add known masses of RO water to these beads and mix well. After allowing the mixtures to reach equilibrium (at least 10 minutes), filter the mixture and weigh both the filtered solution and the extracted beads for each sample. **Measure the conductivity of the filtered solution.** Perform an overall mass balance of these 4 recorded weights for each sample to ensure the procedure was performed accurately. If the percentage error is <10%, use the 4 recorded weights for each sample and the results from step 5) to calculate the amount of KHCO₃ left in the effluent underflow. This will be done for both experimental runs performed.

7) Measure the weight solids left in the hopper at the conclusion of both experimental runs to determine the solids feed rate.

**Report**

1) Plot the concentration of KHCO₃ in each liquid stream vs. time, where in this case time is measured from when the liquid flow first contacts the train of pellets.

2) Report on the accuracy of the overall mass balance of KHCO₃.

3) Determine if your steady-state concentration measurements from all the leaching experiments, combined with your measurements of the equilibrium solids to liquid ratio of the underflow, fall on the expected leaching theory explained above. *(i.e. – Compare the theoretical and experimental values of \( x_A, y_A, \) and \( X_C \) for the products of each run and conduct an error analysis)*

**Design Problem**

Assume that you need to build a large scale leaching facility to remove KHCO₃ from 500 kg/h of solid granules with the same underflow line and leaching data you measured in this experiment. The granules will have the same initial loading of KHCO₃ as for the experiment you have just completed. Determine what would be the minimum amount of water you could use to remove 99% of the KHCO₃ in a seven stage, countercurrent flow extraction system. Discuss factors that would influence the economic optimization for recovery of KHCO₃ in water.
4 - Dynamic Identification and Control of a Surge Tank

Objectives

1. To become familiar with a process, which can adequately be presented by a first order model.

2. To determine experimentally the model parameters.

3. To control the level of liquid in a surge tank by using:

   (a) a conventional controller; and
   (b) a micro computer.

Introduction

In this experiment, the step response technique is used to identify the model and the model parameters of a surge tank. Subsequently, the liquid level in the surge tank is controlled by an electronic analog controller and a digital controller.

1. Process Identification

Figure 1 shows the experimental setup. A block diagram representation of the feedback control system appears in Figure 2a. An equivalent block diagram is shown in Figure 2b.

Introduction of a step change in the inlet flow rate to the tank and monitoring the response in the liquid level results in the combined transfer function for the load and the measuring element. Assume negligible dynamics for the measuring element. Now, the load transfer function $G_L(s)$ can be obtained, since the measuring element gain can be obtained from the measuring element calibration curve.

Introduction of a step change in the control valve actuating pressure and observing the liquid level response results in the combined transfer functions for the valve, the process and the measuring element. The valve has negligible dynamics since the time constant of the valve is much smaller than the process time constant. The steady state gain for the valve is

$$K_v = \left(\frac{\Delta F}{\Delta P_c}\right)_{ss}$$

where $\Delta P_c$ is the step change in the valve’s actuating pressure and $\Delta F$ is the resulting change in the outlet flow rate at steady state. In this way, the process transfer function $G_p(s)$ may be obtained from the above step tests. Due to the nonlinear dependency of the outlet flow rate of the tank on the liquid level, it is important to carry out all the experiments around a fixed steady-state liquid level.
Figure 1: Piping and instrumentation diagram (P&ID) of the surge tank.

Figure 2: Two identical block diagrams of the surge tank.
2. Feedback Control

(a) Analog Controller:

The liquid level is measured by a Differential Pressure (DP) cell, converted to an analog signal (4-20 mA) which is directly transmitted to the controller. The controller is a conventional 3-mode electronic unit with Proportional, Integral and Derivative actions.

\[
P - \text{action:} \quad P_c = \bar{P}_c + K_c e(t) \\
I - \text{action:} \quad P_c = \bar{P}_c + K_c / \tau_i \int_{o}^{t} e(t) dt \\
D - \text{action:} \quad P_c = \bar{P}_c + K_c \tau_D (de(t) / dt)
\]

where
- \( P_c \) : valve’s actuating signal, controller’s output, psig
- \( \bar{P}_c \) : controller bias at \( e(t) = 0 \)
- \( e(t) \) : error signal = \( h_{sp} - h(t) \), psig
- \( K_c \) : controller gain = 100/PB, PB is the controller proportional band
- \( \tau_i \) : integral time, min/repeat
- \( \tau_D \) : derivative time, min

Different combinations of the above controller actions result in P, PI, PD and PID modes.

(b) Computer:

Direct Digital Control (DDC) of the liquid level is carried out by a computer running Labview. The signal from the DP-cell is first converted to an analog electronic signal (4-20mA). The analog signal is then sampled at prespecified intervals of time and converted to a digital signal with an A/D converter. Provided that the sampling interval is ‘short’, the finite difference approximation of eq. (1) can be used to give the discrete version of the PID-controller by:

\[
V_n = V_o + K_c \left[ e_n + \Delta t \sum_{k=1}^{n} \frac{e_k + \left( \frac{\tau_D}{\Delta t} \right) (e_n - e_{n-1})}{\tau_i} \right]
\]

where:
- \( V_n \) computer output at the nth sampling interval, Volts
- \( e_n \) error at the nth sampling interval, Volts
- \( V_o \) computer output when \( e_n = 0 \), Volts
Δt sampling interval, sec

Equation 2 is the “position” form of the discrete version of the PID - controller. It is obtained by approximating the integral term with the rectangular rule, and the derivative term with a first-order backward difference.

Implementation of equation 2 requires the knowledge of \( V_o \), the steady state value of computer output signal. For a nonlinear process, which includes most chemical engineering processes, this means updating \( V_o \) for each new set point. In order to circumvent this problem, the “velocity” form of the discrete version of the PID - controller is used. This is obtained by writing an equation similar to equation 2 for the \((n-1)st\) sampling interval and subtracting it from equation 2.

The velocity form of the discrete version of the PID - controller becomes

\[
\Delta V_n = K_c \left[ e_n - e_{n-1} + \left( \frac{\Delta t}{\tau_I} \right) e_n + \left( \frac{\tau_D}{\Delta t} \right) (e_n - 2e_{n-1}) + e_{n-2} \right]
\]  

(3)

where \( \Delta V_n \) is the change in the computer output signal at the \(n\)th sampling interval relative to its value at the \((n-1)st\) interval. So,

\[
V_n = \Delta V_n + V_{n-1}
\]  

(4)

The valve’s actuating signal calculated in your application program using equations 3 and 4 is first converted to an analog signal with a D/A converter. It is then held constant during the sampling intervals with an electronic zero-order hold device and finally converted to a pneumatic signal via an I/P transducer.
PROCEDURE

1. Controller Description

The controller is Omega CN3251 Temperature/Process Controller. This is a PID controller which is able to be set working as a P, PI, PD and PID controller. A picture of the controller is shown below:

The first line (red numbers) shows process variables in normal operation mode. The second line (green numbers) displays active set-point in auto mode or percent of valve closure in manual mode. The description of the other buttons is as follows:

a. Auxiliary button: Auto/Manual selector. When the controller is in manual, the AUX light turns on.
b. Up/Down arrows:
   1. Used to change the set point in auto mode. **Set point should be kept at 50% for all experiments.** This is equal to 20 in height of water which is half of the total height of the column (40 in) forcing the controller to keep the water level at 20 in when it is working in auto mode.
   2. Used to set the water level manually when working in manual mode.
c. Reset button: To reach the controller menu. Holding the reset button for 3 seconds allows entering or exiting the controller menu.

Adjustable parameters needed to perform this experiment are proportional band (PB), $\tau_1$ and $\tau_D$. They are shown as Pb1 (%), Ar1 (repeat/min) and rAt1 (sec) for PB, $\tau_1$ and $\tau_D$ respectively.

To adjust the controller parameter, put the controller in auto mode and then hold the reset button for 3 seconds. Hit the reset button until you reach Pb1. Set the desired value by playing with arrow buttons and then hit the reset button to reach Ar1. Set $\tau_1$ and hit the reset button again to reach rAt1 to adjust $\tau_D$. Hit the reset button again and hold the reset button to exit the menu. It brings you back into the auto mode. You can now switch between auto and manual with auxiliary button.
2. **Process Identification**

1. Open up the supply air valve.

2. Isolate the analog controller loop from the DDC loop.

3. Switch the controller to MANUAL. In the MANUAL mode the controller acts as a remote manual valve stem positioner. This can be done by pushing the auxiliary button on the controller. When the auxiliary light is on, the controller is in manual mode.

4. Open up the inlet water valve and set it at approximately 3 gpm.

5. Adjust the valve stem position so that the liquid level in the tank is approximately 10 in. of water. Playing with up and down arrows allows controlling the level manually.

6. Wait until the liquid level comes to rest for 3 to 5 seconds.

7. Introduce a step change in the inlet water flowrate from 3 to 4 gpm while the flowrate is being recorded on the recorder. This can be done by switching the solenoid valve open.

8. Observe and record the resulting response in the liquid level on the recorder. Wait until new steady state is reached.

9. Set the inlet flowrate at its original value.

10. Wait until the liquid level comes to rest at its original value.

11. Introduce a small step change in the valve stem position and record the change in liquid level. To do this, set the water level at 10 in first and increase the closure percentage of the valve by 10%. For example if the second line of the controller (green numbers) shows 42% when the water level is at 10 in, increase is by 10% to 52%. DO NOT OPEN THE SOLENOID VALVE AT THIS STAGE.

12. Observe and record the resulting response in the liquid level on the recorder.

2. **Feedback Control**

(a) **Analog Controller**

(i) **P-Control**

1. Put the controller in auto.

2. Check the set point to be at 50%

3. Set the controller to be a P-Controller: $PB = 100\%, \ t_I = 0, \ t_D = 0$. The procedure to adjust the controller parameters is described in section one of this manual.
4. Switch the controller to manual and keep the water level at 10 in by playing with arrow buttons.

5. Switch the controller to AUTOMATIC. The device is now a feedback controller.

6. Push the data capturing button of the software on the screen. Observe and record the response.

7. Repeat steps 1-6 for PB ≈ 50% and PB ≈ 20%.

(ii) **PD-Control**

1. Put the controller in auto.

2. Check the set point to be at 50%.

3. Set the controller as a PD-Controller: PB ≈ 20%, t_I = 0, t_D = 2 seconds.

4. Repeat steps 4-6 in section i).

5. Repeat steps 1 - 4 in section ii) with t_D = 200 seconds and then t_D = 500 seconds

(iii) **PI-Control**

1. Put the controller in auto.

2. Check the set point to be at 50%.

3. Set the controller as a PD-Controller: PB ≈ 20%, t_I = 100 repeat/min, t_D = 0 seconds.

4. Repeat steps 4-6 in section i).

5. Repeat steps 1 - 4 in section ii) with t_I = 10 repeat/minutes and then t_I = 1 repeat/minutes

(iv) **PID-Controller**

The combination of the three controller modes when properly tuned should ensure a rapid, stable and accurate response.

An empirical method of finding the optimum controller settings was proposed by Cohen and Coon. The parameters are determined open-loop, so the controller is not hooked up to the system. A step-change is input into the controlling element (in this case the control valve at the bottom of the tank) and the response of the liquid level is recorded by the computer. This response should be sinusoidal in nature. The curve is known as the process reaction curve and contains the effects from the process, the valve and the measuring sensor.
According to Cohen and Coon, most processes will have a response to this change that may be approximated as a first-order system with dead time. This model is

\[ G_{PRC} \approx \frac{Ke^{-t_d s}}{\tau s + 1} \]

where: \( K = \) output (at steady state)/input (at steady state) - B/A; 
\( \tau = B/S, \) where S is the slope of the sigmoidal response at the point of inflection; 
\( \tau_d = \) time elapsed until the system responded.

Analysis of many systems by Cohen and Coon led to the following optimal settings for a PID-Controller:

\[ K_c = \frac{1}{K} \frac{\tau}{t_d} \left( \frac{4}{3} + \frac{t_d}{4\tau} \right) \]

\[ \tau_I = t_d \frac{32 + 6t_d / \tau}{13 + 8t_d / \tau} \]

\[ \tau_D = t_d \frac{4}{11 + 2t_d / \tau} \]

Because there is no time delay in the present system, you cannot use the Cohen-Coon formulas to calculate good values of \( K_c, \) \( T_I \) and \( T_D. \) Instead, calculate a value of \( K_c \) which with a proportional-only controller gives an offset of 2 inches of water. Then simply add some integral and derivative actions and see how the system response will change. You need to try 5 different trials. The two first parameters for the PID controller are given as follows:

1. PB=20%, \( \tau_I=10 \) repeat/min and \( \tau_D=2 \) seconds
2. PB= 100%, \( \tau_I=1 \) repeat/min and \( \tau_D=2 \) seconds

Try to improve the response of the above parameters in the next three trials. Change any parameters by your choice based on the expectation you have from changing that parameter to improve the controller response.

b) **Direct Digital Control (DDC):**

1. Take the best parameters from the analog controller to be used in the DDC. Convert the analog PID parameters to appropriate units for DDC. \( K_c \) in DDC is equal to 100/PB, I in DDC is equal to \( 1/\tau_I \) because I is in minutes/repeat in DDC and D in DDC is equal to \( \tau_D/60 \) because D is in minutes in DDC.

2. Set the set point at 20 in and the sampling time at 1 second.

3. Bring the head to the state of approximately 10-in.

4. Isolate the analog loop by switching to DDC and start capturing your data. Now the DDC controller is in place.
5. When done, switch to analog.

**NOTE:** The sampling interval and the controller integral time and derivative time must be in the same unit, for example in seconds.

4. Keep the set point at 20 in and repeat sections 3 to 5 for $\Delta t = 3$, 5 and 7 seconds.

**REPORT**

The report should be as brief as possible. Avoid repeating the theory which is explained in the handout.

1. **Process Identification**

Include the process open loop responses to a step change in the inlet flow-rate and in the valve actuating signal in your report.

What are the values for $K_p$ and $\tau_p$ (gain and time constant for the process), $K_v$ (gain for the valve), $K_m$ (gain for the measuring device), $K_L$ and $\tau_L$ (gain and time constant for the load)?

2. **Feedback Control**

a) **Analog Controller**

   (i) **P-Control**

Include the process closed-loop responses at $PB \approx 100\%$, 20\% and 5\% in your report. What is the effect of decreasing $PB$? Compare the theoretical and the experimental closed-loop response at $PB = 20\%$. In the evaluation of the theoretical response, use the transfer functions which were obtained experimentally. Use Simulink to calculate the theoretical response. Plot both the theoretical and experimental closed-loop responses on the same graph.

   (ii) **PD-Control**

Include the process closed-loop response at $PB \approx 5\%$ and $t_D \approx 0.02$ min, 0.2 min and 2 min in your report. What is the effect of increasing $t_D$?

   (iii) **PI Control**
Include the process closed-loop response at PB $\approx 20\%$, $t_l \approx 1$ min, 0.1 min and 0.01 min in your report. What is the effect of decreasing $t_l$?

(iv) **PID-Control**

Include the process closed-loop response in your report. What are the optimum controller settings obtained after tuning the PID controller? Use Simulink to compare the theoretical and experimental responses at the controller settings.

b) **DDC**

Why is it necessary to adjust the PID settings once you switch to computer control? Include the process response for sampling intervals $\Delta t \approx 3$, 5 and 7 seconds in your report. What is the effect of increasing $\Delta t$ and why?
5 - Biodiesel – Transesterification of Canola Oil

Introduction

The production of biodiesel fuel as an alternative source of energy for transportation has been very popular for the last few years. However, the drawback of biodiesel is its high production cost. More efficient, feasible and economical production methods need to be learned, researched and developed further in order to increase public demand on this environmentally friendly fuel.

This experiment is to provide a means to better understand biodiesel production process. A small-scale laboratory experiment is chosen for this study to duplicate industrial biodiesel production to a certain extent. The procedure to perform such laboratory experiment is illustrated in the diagram below.

This experiment is also aimed to accommodate students in performing several works. Firstly, to understand the concept or nature of transesterification reaction. Secondly, to differentiate between biodiesel and regular diesel by testing physical properties of the final biodiesel product. Finally, to make recommendations on improving biodiesel production by considering time and economical aspect.
Theory and Reaction

Biodiesel refers to diesel-equivalent processed fuel. Also known as mono alkyl ester of long chain fatty acids, the biodiesel is mostly produced through base catalysed transesterification. The base catalysed transesterification is a reaction of vegetable oil or animal fat with alcohol in the presence of a base catalyst to yield biodiesel and glycerol. Many industries today use the same type of reaction to reduce oil viscosity in biodiesel processing. Biodiesel production with base catalyzed reaction has several advantages. Firstly, the process requires low temperature and pressure, hence it is inexpensive. Secondly, the reaction produces high yield with minimum reaction time and least amount of reaction by-product. Lastly, the direct biodiesel conversion results in no intermediate compounds.

A. Mechanism of Transesterification Reaction

Vegetable oil and animal fat are mostly triglycerides, which are composed of three chains of fatty acids bound by a glycerol molecule. Transesterification or alcoholysis is a reversible reaction. Reversing the reaction backward can reproduce triglycerides. On the other hand, in the forward reaction, triglycerides are converted to glycerol (glycerine) and its corresponding fatty ester. One complete cycle conversion requires additional alcohol or removal reaction product (ester) to shift the equilibrium to the right side faster. A multi-step process or overall transesterification reaction is as follows:
R₁, R₂, and R₃ in the reaction represent fatty acid alkyl groups.

The overall transesterification reaction can be broken down into three individual steps. The first step is the conversion of triglyceride to diglyceride. By using methanol as alcohol and sodium hydroxide as catalyst, the first step transesterification reaction is shown below.

*Methoxide and water are formed by the reaction between sodium hydroxide and methanol when they are mixed together.* The second and the third step transesterification reaction produce monoglyceride and glycerol respectively. A complete transesterification reaction is summarised as follow:

Step 1: Triglyceride + MeOH + Catalyst $\leftrightarrow$ Ester + Diglyceride + Catalyst

Step 2: Diglyceride + MeOH + Catalyst $\leftrightarrow$ Ester + Monoglyceride + Catalyst
Step 3: Monoglyceride + MeOH + Catalyst $\leftrightarrow$ Ester + Glycerol + Catalyst

Overall: Triglyceride + 3 MeOH + Catalyst $\leftrightarrow$ 3 Ester + Glycerol + Catalyst

The principal reaction for each individual step is very similar. Organic group $R_1$, $R_2$ or $R_3$ of mono-, di- or tri-glyceride is exchanged with organic group (-CH$_3$) of an alcohol to produce different glyceride and ester. In our case, the alcohol is methanol. The organic group (-R) may change depending on the alcohol being used. To complete the whole conversion process, alcohol must be supplied in sufficient amount to replace $R_1$, $R_2$ or $R_3$ organic group of glyceride in each individual step. The stoichiometric ratio of transesterification reaction requires three moles of alcohol (methanol) and one mole of triglyceride to yield three moles of fatty acid ester and one mole of glycerol. Higher molar ratios result in larger amount of ester products. However, high molar ratio requires large amount of alcohol reactant, which also means high material cost. The acceptable molar ratios of alcohol to vegetable oil are 3:1 to 6:1.

**Experimental Procedure**

It should be noted that free fatty acid (in feedstock) and moisture content (in the reactant) also play important roles in producing higher level of biodiesel purity.

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1. Measure a known quantity of pure canola oil, anhydrous methanol and sodium hydroxide. Molecular weights of canola oil, methanol and sodium hydroxide are 882, 32 and 40 gram/mol respectively. When 6:1 molar ratio of alcohol to vegetable oil is used, 100 gram of canola oil requires at least 22 grams of methanol to meet this ratio. Oleic acid (61%) and linoleic acid (21%) are major compounds in the canola oil.

2. Transfer the canola oil into a silicon cup positioned inside a stainless steel vessel. Secure the vessel by clamping it into the bench top reactor. Set heater temperature to 70 ºC to heat the oil. (may be preset)

3. Weigh 1.03 gram of sodium hydroxide catalyst.

4. Place the methanol and sodium hydroxide into a beaker and swirl manually. Make sure all the catalyst is completely dissolved (cloudy mixture).

5. Pour methanol and sodium hydroxide mixture (methoxide catalyst) to the canola oil in the reaction vessel quickly. Extra caution is required during the pouring process, as the vessel temperature is very high and past the flash point of methanol. The objective is to maintain temperature for both canola oil and methanol-sodium hydroxide mixture at about the same in the beginning of reaction.

6. Attach the reaction vessel back to the reactor. Set the reaction temperature at 70 ºC. This temperature is maintained constant throughout the reaction by a thermostat inside the heating jacket during reaction. Set the RPM speed at 600 r/min (may be preset)

7. Boiling point of methanol is 64.7 ºC. During the reaction, methanol gets vaporized. To prevent any reactant loss, nitrogen gas is used to pressurize the alcohol vapor, converting it back into liquid phase in the reactor. For pressure setting, refer to Vapour Liquid Equilibrium (VLE) diagram of methanol. (14 psi)

8. Take small amount of sample from sampling valve every 10 minutes for the first half hour, every 15 minutes for the remaining half hour. Add tetrahydrofuran (THF) to each sample.
**THF is used to dilute the sample before analyzing in HPLC.** The dilution factor should be about 20:1 (THF to sample volume ratio, 50 µl to 1 ml).

9. Test the diluted samples for its methyl ester, monoglyceride, diglyceride, triglyceride and glycerol contents with High Pressure Liquid Chromatography (*Agilent HPLC 1100 series, Phenomenex column 300 x 7.8mm, THF mobile phase; Injection volume 5µl; flow 1ml/min, column thermostat 45°C, detector at 45°C)*.

10. Once the reaction is completed, cooling water is circulated to cool the remaining product in the vessel.

11. Centrifuge the remaining product at 3000 r/min for 15 minutes. Two phases are formed as a result of centrifuge. Upper layer consists of biodiesel, methanol and catalyst. Lower layer consists of glycerol, excess catalyst, methanol, and small trace of unreacted oil.

12. Weigh the upper and lower layer centrifuge products separately.

**References**

1. Allen, M. and Prateepchaikul, G. ‘The Modeling of the Biodiesel Reaction (online posting)’, Department of Mechanical Engineering, Faculty of Engineering, Prince of Songkla University, Hadyai, Thailand.

   <http://journeytoforever.org/biofuel_library/macromodx.html>
