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KING FAISAL UNIVERSITY
College Of Engineering

DEPARTMENT OF CHEMICAL ENGINEERING

CHE406: Chemical Engineering lab III

"Lab Manual"

Major Topics covered and schedule in weeks:

Topic	Week #	Courses Covered
Introduction and Lab safety.	1	
Continuous distillation	2	Separation processes 2
Cooling tower	3	Separation processes 3
Single effect evaporator	4	Separation processes 2
Liquid-liquid extraction	5	Separation processes 2
Gas absorption	6	Separation processes 2
Ion exchange	7	Separation processes 3
Mid Exam	8	
Flow meters characteristics	9	Process Dynamics and Control
Thermocouples characteristics	10	Process Dynamics and Control
On/Off and continues control	11	Process Dynamics and Control
Feedforward and cascade control	12	Process Dynamics and Control
Controller tuning	13	Process Dynamics and Control
Ratio control	14	Process Dynamics and Control
Final Exam	15	

Specific Outcomes of Instruction (Lab Learning Outcomes):

1. Conduct experiments in the areas of unit operations and process control and Apply safety rules and guidelines inside the lab and while conducting experiments
2. Practice different types of mass transfer operations and process control
3. Analyze and interpret experimental data with theories learned in previous courses
4. Write organized and cohesive technical reports
5. Work effectively in a team environment

Student Outcomes (SO) Addressed by the Lab:

z	Outcome Description General Engineering Student Outcomes	Contribution
1.	an ability to identify, formulate, and solve complex engineering problems by applying principles of engineering, science, and mathematics	H
2.	an ability to apply engineering design to produce solutions that meet specified needs with consideration of public health, safety, and welfare, as well as global, cultural, social, environmental, and economic factors	
3.	an ability to communicate effectively with a range of audiences	M
4.	an ability to recognize ethical and professional responsibilities in engineering situations and make informed judgments, which must consider the impact of engineering solutions in global, economic, environmental, and societal contexts	**L**
5.	an ability to function effectively on a team whose members together provide leadership, create a collaborative and inclusive environment, establish goals, plan tasks, and meet objectives	L
6.	an ability to develop and conduct appropriate experimentation, analyze and interpret data, and use engineering judgment to draw conclusions	M
7.	an ability to acquire and apply new knowledge as needed, using appropriate learning strategies	

Table of Content

Sn	Experiment Name	Page Number
i	LABORATORY SAFETY	4
ii	General Guidelines	9
iii	Laboratory Safety Rules	10
iv	EXPERIMENTS	11
v	Cooling Tower	14
vi	Single Effect Evaporator	15
1	Continuous Distillation	18
2	Gas Absorption	21
3	Liquid – Liquid Extraction	24
4	Ion Exchange	27
5	Level Control	30
6	Flow Control	34

LABORATORY SAFETY

Safety in the laboratory is everyone's responsibility, Experiments conducted in a safe and conscientious manner will protect you and your partner in the laboratory. The following guidelines are for your own protection and you are responsible for compliance with all safety regulations set forth in this manual.

GENERAL GUIDELINES

- Laboratory coat must be worn all the time when working in the laboratory.
- Only closed toe shoes are allowed in the laboratory. Do not wear sandals, slippers and high heel shoes inside the laboratory.
- Students with long hair must get their hair tied up tidily when doing laboratory work.
- Bags and other belongings must be kept at the designated places.
- Foods, drinks and smoking are strictly prohibited inside the laboratory.
- Noise must be kept to the minimum as a courtesy to respect others.
- Students are not allowed to work alone without the supervision of laboratory instructor. There must be at least 2 persons present in the laboratory at the same time.
- Students are not allowed to bring any outsiders (non-registered parties) into the laboratory.
- Any unauthorized experiment without the knowledge of the laboratory instructor is prohibited.
- All instruments and equipment must be handled with care.
- Workspace has to be cleaned and tidied up after the experiment completed. Instrument and equipment must be returned orderly after use.
- Students are strictly prohibited to take any equipment or any technical manuals out from the laboratory without the permission of laboratory instructor.
- Students are required to instill an instinctive awareness towards property value of laboratory equipment and to be responsible when using it. Any damages can cause to jeopardize the success of not only the individual work but also to the university.
- Do not attempt to remove and dismantle any parts of the equipment from its original design without permission.
- Students shall be liable for damages of equipment caused by individual negligence. If damages occurred, an investigation will take place to identify the causes and the names of the involved students and will be recorded for faculty attention.
- Disciplinary action shall be taken against those students who failed to abide the rules and regulations.

LABORATORY SAFETY RULES

- It is always a good practice and the responsibility of an individual to keep a tidy working condition in laboratory.

- Follow the procedures given by the laboratory instructor when conducting laboratory experiment.
- Before any experiment starts, students must study the information / precaution steps and understand the procedures mentioned in the given experiment sheet.
- Students should report immediately to the laboratory instructor if the laboratory equipment is suspected to be malfunctioning or faulty.
- Student should report immediately to the laboratory instructor if any damages on equipment or any hazardous situation are noticed.
- Students should report immediately to the laboratory instructor if any injury occur.
- If there is a tingling feel when working with electrical devices, stop and switch off the devices immediately. Place a warning note before reporting to the laboratory instructor and wait for further instructions.
- Do not work with electricity under wet condition in laboratory. Electric shock is a serious fatal hazard.
- Students are required to wear goggles, gloves, apron and mask when handling corrosive or active chemical agents.
- Hazardous chemical agents must be properly stored and labeled in a designated place. Students must acquire and read the material safety data sheet of a particular chemical agent before using it.
- Familiarize yourself with the safety equipment in the laboratory such as fire extinguishers, eye wash station and shower station.

Experiment 1: COOLING TOWER

I. Objective:

- To illustrate the practical operation of a small cooling tower under a range of process conditions.
- To observe the water flow pattern and distribution with various water and air flow rates.
- Reinforce the concepts of the psychometric chart and mass and energy balances.
- To estimate the minimum gas flow rate necessary.
- To calculate the height of transfer units, the number of transfer units and the height of packing for evaporative cooling.

II. Test Standard

No standards

III. Theory:

The following terms are commonly used when referring to the performance of a cooling tower:

Approach Temperature difference between the temperature of the condenser water leaving the tower and the wet-bulb temperature of the air entering the tower.

Blow down Water discharged to the drain periodically to avoid buildup of dissolved solids.

Fill The structure that forms the heat-transfer surface within the tower. Water from the condenser or coil is distributed along the flow passages of the fill down to water basin.

Makeup Water added to the circulating water to compensate for the loss of water due to evaporation, drift, and blow down.

Range Temperature difference between the temperature of condenser water entering the tower and the temperature leaving the cooling tower Ttl , both in.

Tower coefficient

Baker and Shryock (1961) developed the following theory for the thermal analysis of the cooling tower. Because the makeup water in a typical cooling tower is usually about 2 percent of the circulating condenser water, if the energy difference between the makeup water and the blow down and the drift losses are ignored, and if the enthalpy increase of the water from the addition of liquid water is ignored, then the energy balance between condenser water and air can be calculated as:

$$m_w Cp_w dT_w = m_a dh_a$$

Where:

m_a, m_w: air and water mass flow rates

Cp_w: water specific heat

T_w: water temperature

h_a: air enthalpy

If the thermal resistance of the saturated air film that separates the condenser water and the airstream is ignored, the combined heat and mass transfer from the air-water interface (the saturated air film that surrounds the condenser water droplets) to the bulk air stream can be evaluated as:

$$m_a dH_a = K_m (h_s - h_a) dA$$

Where:

K_m: mass transfer coefficient

h_s: enthalpy of saturated air film

A: Surface are at water – air interface

Consider a cooling tower with a fill volume V and contact surface area $A = aV$. Here a is the surface area of fill per unit volume. Also let $K = K_m/Cp_w$. Then combining equation we get:

$$\frac{KaV}{m_w} = \int_{T_{w1}}^{T_{w2}} dT/(h_s - h_a)$$

The integrated value of the above equation is generally known as the tower coefficient, or the number of transfer units **NTU**, of the cooling tower.

Heat and mass transfer process for counter flow cooling tower

Figure 1shows the heat and mass transfer process between condenser water and air in a counter flow cooling tower with a water air ratio mw/ma = 1.2. In Figure 1 (a), water is cooled from the entering temperature Twe = 85°F (29.4°C) to the leaving temperature Twl, = 75°F (23.9°C) at the exit. The temperature of the saturated air film corresponding to the condenser water temperature drops along the saturation curve and can be represented by the section we–wl.

Air enters the counter flow tower at a dry bulb Tae = 80°F (26.7°C) and a wet bulb T'ae= 68°F (20°C). Because the temperature of the entering air is higher than the temperature of leaving water, that is, Tae > Twl, first air is evaporative cooled until the air temperature approaches the water temperature. After that, air is humidified and heated because the water temperature is higher. Air is essentially saturated at the top exit of the tower. The humidifying and heating process of air is illustrated by the curve ae–al shown in Figure 1 (a). The driving potential of heat and mass transfer at the condenser water inlet is indicated by the difference in enthalpy between the saturated air film and the air, or hs,we– hal. At the condenser water outlet just above water basin it is indicated by hs,wl - hae.

In Figure1 (b), for the same counter flow cooling tower, air enters at Tae = 72°F (22.2°C) and at

T'ae = 70°F (21.1°C); water may be cooled from 86.5 to 76.5°F (30.3 to 24.7°C). Because Twl > Tae air is humidified and heated along a curve nearly parallel to the saturation curve. The driving potential is still the difference in enthalpy between a saturated air film and air. Air is also approximately saturated at the top exit and approaches point al on the saturation curve. Under this condition, the sensible heat transfer is about 23% of the total heat transfer. This value indicates that even on rainy days, when air enters the tower in a near saturated condition, there is still latent and sensible heat transfer between water and air. The driving potential is mainly due to the higher enthalpy of the saturated air film surrounding the condenser water droplets, which are at a higher temperature than the contacted air.

Tower Capacity, Size, and Coefficient

The energy balance equation can also be expressed as:

$$m_w Cp_w(T_{we} - T_{wl}) = K_m aV(h_s - h_a)dA$$

The total heat removed from the condenser water $m_w Cp_w (T_{we}\text{-}T_{wl})$ represents cooling tower capacity. Tower capacity must meet total heat rejection at the water cooled condenser.

Tower size is indicated mainly by the volume of the fill *V*, which includes both the cross sectional area and the depth of the fill. Strictly speaking, heat and mass transfer occurs in the space between the fill and the water basin as well as between the fill and the water spraying section, For simplicity, these can be considered as being included in the volume of fill.

The tower coefficient KaV/m_w, actually indicates the heat-transfer unit or size of the fill. It is the primary factor that influences the effectiveness of the cooling tower. An increase of tower capacity may be attributed to a larger tower size or a higher tower coefficient or both. For a fixed water circulation rate, an increase in tower coefficient may be caused by a larger tower size or a better fill configuration.

By using the numerical integration method, the tower coefficient for a counter flow cooling tower can be calculated as:

$$\frac{KaV}{m_w} = (T_{w2} - T_{w1})/(h_s - h_a)$$

Factors affecting cooling tower performance

The criteria for selecting a cooling tower for a water cooled condenser are the effective remove of total heat rejected at the condenser and the minimization of the sum of power consumption in compressors, condenser fans, and condenser water pumps. Proper selection of tower range, water-air ratio, approach, fill configuration, and water distribution system directly affects the performance of a cooling tower.

Range and water circulating rate

Many cooling towers are custom made; i.e., the manufacturer varies the fill configuration, water circulation rate, and air flow rate to meet the operating characteristics required by the customer. The range T_{we} — T_{wl}, and the corresponding water circulating rate m_w, are the primary parameters that should be specified during design. The range depends on the heat rejected from a water cooled condenser Q_{rej}, and the circulation rate of condenser water m_w. Their relationship can be expressed as:

$$(T_{we} - T_{wl}) = \frac{Q_{rej}}{Cp_w m_w}$$

The greater the water circulation rate, the smaller the range. In contrast, a smaller m_w results in a greater range. A greater range and a smaller m_w result in a higher condensing pressure, a lower pumping energy, a smaller tower size, and a lower airflow rate.

Tower coefficient and water-air ratio

Cooling tower performance is often determined from field tests and presented in the form of an empirical correlation as:

$$\frac{KaV}{m_w} = C(m_w/m_a)^n Z^{-m}$$

Where:

C = constant

Z = depth of the fill,

The average value of exponent n varies from -0.4 to -0.65. According to Webb (1984a, 1984b), the exponent m varies between 0.7 and 1.0.

Approach

Approach determines the temperature of condenser water leaving the tower T_{wl} corresponding to a local outdoor design wet bulb T'_o. If the range and Q$_{rej}$ are fixed values, a closer approach always means a lower T_w , and a lower condensing pressure and temperature. Approach is closely related to the tower coefficient and the size of the tower. A closer approach always means a larger tower, and a greater approach results in a smaller tower.

Outdoor wet bulb temperature

Because heat and mass transfer at the fill is based on the difference in enthalpy between the that affects the performance of the cooling tower. If two different cooling tower systems have the same range and approach at different locations, the one with a higher design outdoor wet bulb temperature will have a smaller tower size than the system with a lower outdoor wet bulb temperature.

Fill configuration and water distribution

Fill configuration has a direct effect on the tower coefficient. An efficient fill has a greater surface area per unit volume *a*, which means more contact surfaces, a longer contact time, and intimate contact between air and water. Two types of fill are widely used in cooling towers systems: splash bars, as shown in Figure 2 (a), and the more recently developed cellular film, shown in Figure 2 (b). Splash bars have the advantage of not being easily plugged by dirt contained in water; the splashing action can also redistribute water at various levels. However, field practice has shown that when wooden splash bars are replaced by polyvinyl chloride (PVC) cellular film fill, the temperature of condenser water leaving the tower drops 2°F (1.1°C) compared to the value before retrofit. Contaminated water must be strained or treated before it is cooled in a PVC cellular film fill. An even water distribution system is very important to tower performance, especially for counter flow towers.

IV. Apparatus:

General Description

A schematic diagram of the set-up is shown in Figure 3. The water cooling tower unit comprises a ten plate column, the tower base with air circulation system, the water reservoir with heater and make-up tank and the control console with recirculation pump and instrumentation.

Principles of Operation:

Water Circuit Water, which is regulated by a flowmeter, is pumped from the loading tank up to the distribution cap where the temperature is taken. Water is evenly distributed over the packing. The packing breaks the water into thin films or droplets resulting in a large surface area in contact with air stream and thereby assist the cooling process. The water is cooled largely by evaporation and, to a small extent, by direct contact cooling. At the bottom of the tower the water falls past a thermometer and into the loading tank where it can be reheated and recirculated to the column. Due to the evaporation of some of the water into air stream, a continuous supply of fresh ‘make-up’ water is added to the system. The make-up tank is equipped with a float valve that will open and transfer water into the loading tank as required.

Air Circuit Air, which can be preheated, is pulled from the atmosphere using a fan placed at the base of the tower and passes upwards through the packing. The air then passes through a droplet arrestor before exiting to the atmosphere through an orifice. Fan speed adjuster can be adjusted to change the flow rate of air and the pressure drop through the orifice can be used to estimate the air flow rate. The dry and wet bulb temperatures of inlet and outlet air are measured.

Temperature selector switch and display The temperature selector switch is used for selecting the sensor and measuring the certain point. There are 7 measurement points provided into the system for measuring air and water temperatures. The temperature display will display the temperature of selected measurement point. The measurement points are explained as follow:

T1 = Air inlet temperature (dry bulb)
T2 = Air inlet temperature (wet bulb)
T3 = Air outlet temperature (dry bulb)
T4 = Air outlet temperature (wet bulb)
T5 = Cooling tower water inlet temperature

T6 = Cooling tower water outlet temperature
T7 = Make up water temperature

V. Procedure:

Pre-operation procedure

1. Place the trainer in a room with sufficient area.
2. Prepare the equipment required for the experiment.
3. Ensure that all valves are closed.
4. Connect the cooling tower trainer to a suitable power supply
5. Prepare the cooling tower trainer in the area with enough space, where you can get fresh water supply and water drain.
6. Fill the system with distilled water trough the manual valve (MV7) until the water at the basin reaches for about 14 liter.
7. Connect MV4 to water supply. Fill the Make-up Water Tank by open MV4 and MV3 then close MV4 when it is full.
8. Ensure all switches in the instrument and control panel are off.
9. Open MV2 on Cooling Tower Trainer.
10. Turn on the ELCB, MCB and the main power switch of Cooling Tower Trainer.
11. Set the water thermostat to 60^{o}C position.
12. Use MV1 and MV2 to adjust the water flow.
13. The trainer is ready to be used for the experiments after all of the steps above have been completed.

Experiment

1. Prepare the unit as explained above.
2. Ensure the water flow control is fully opened by turning the stem counter clockwise.
3. Switch ON the water pump switch. Make sure the water pump is working normally.

You will see the water is flowing inside the cooling tower through the water distributor.

4. Adjust the control valve until the water flow quantity reaches the maximum level.
5. Ensure thermostat is adjusted at 60°C.
6. Switch ON both water heaters (heater 1 and heater 2 switches).
7. While observing the fan anemometer display, switch ON the fan switch and adjust the fan speed adjuster to "maximum" position by turning the adjuster clockwise.
8. Allow the system to reach steady state (about 10 minutes). Use the temperature selector switch to display the temperatures and record the temperatures on the raw data sheet. Measure the time required of the makeup water to add 50 ml to the basin
9. Reduce the water flow rate to 10 l/min.
10. Allow the system to reach steady state (about 10 minutes). Use the temperature selector switch to display the temperatures and record the temperatures on the raw data sheet.
11. Increase water flow rate to maximum (the same flow rate as in step 4).

12. Reduce the fan speed to approximately 50% of the maximum speed.
13. Allow the system to reach steady state (about 10 minutes). Use the temperature selector switch to display the temperatures and record the temperatures on the raw data sheet. Measure the time required of the makeup water to add 50 ml to the basin.
14. Reduce the water flow rate to 10 l/min (the same flow rate as in step 9).
15. Allow the system to reach steady state (about 10 minutes). Use the temperature selector switch to display the temperatures and record the temperatures on the raw data sheet.
16. Switch OFF the water pump, air fan and the heaters.
17. Remove the top cover of the tower and remove half of the packing.
18. Switch ON the water pump, air fan and heaters
19. Adjust the control valve until the water flow quantity reaches the maximum level (the same flow rate as in step 4).
20. Ensure thermostat is adjusted at 60°C.
21. Adjust the fan speed adjuster to "maximum" position by turning the adjuster clockwise (the same speed as in step 7).
22. Allow the system to reach steady state (about 10 minutes). Use the temperature selector switch to display the temperatures and record the temperatures on the raw data sheet.
23. Reduce the water flow rate to 10 l/min (the same flow rate as in step 9).
24. Allow the system to reach steady state (about 10 minutes). Use the temperature selector switch to display the temperatures and record the temperatures on the raw data sheet.
25. Increase water flow rate to maximum (the same flow rate as in step 4).
26. Reduce the fan speed to approximately 50% of the maximum speed.
27. Allow the system to reach steady state (about 10 minutes). Use the temperature selector switch to display the temperatures and record the temperatures on the raw data sheet.
28. Reduce the water flow rate to 10 l/min (the same flow rate as in step 9).
29. Allow the system to reach steady state (about 10 minutes). Use the temperature selector switch to display the temperatures and record the temperatures on the raw data sheet.

VI. Experimental Work:

At each operating condition of the experiment calculate the following:

1. From the psychometric chart find:

Enthalpy of air entering and leaving the cooling tower (h_{ae}, h_{al})

Specific volume of air entering and leaving the cooling tower

Absolute humidity of air entering and leaving the cooling tower

Enthalpy of water entering and leaving the cooling tower ($h_{s, we}$, $h_{s, wl}$)

2. Calculate the deference in air enthalpy of air entering and leaving the cooling tower
3. Calculate the driving force at the top and bottom of the cooling tower

At the top: $h_{s, we} - h_{al}$

At the bottom: $h_{s, wl} - h_{ae}$

4. Calculate the energy absorbed by air.
5. Calculate the energy released by water (range)

6. Is there any difference between the energies calculated in step 4 and 5? Explain.
7. What is the effect of air speed on water temperature and energy absorbed by air
8. Calculate the makeup water flow rate. Explain the relation between air speed and water evaporation.
9. Using data from full packing and 50% of the packing calculate the approach (the difference between water leaving the cooling tower and the wet bulb temperature of air entering the tower). In your own words what are the factors affecting the approach.
10. Calculate the tower coefficient at the following conditions:

- Full packing, maximum air speed and maximum water flow rate.
- Full packing, maximum air speed and 10 l/min water flow rate.
- %50 packing, maximum air speed and maximum water flow rate.
- 50% packing, maximum air speed and 10 l/min water flow rate.

From your results explain the effect of the above variables on tower coefficient.

To calculate the tower coefficient follow the following procedure of the numerical integration of the equation (also refer to table 1):

$$\frac{KaV}{m_w} = \int_{T_{w1}}^{T_{w2}} dT/\,(h_s - h_a)$$

- Divide the water temperature into intervals of 0.5°C starting from water outlet temperature. Fill the temperatures in the first column.
- From the psychometric chart find the enthalpy of saturated air h_s at each temperature. Fill the data in the second column.
- From the psychometric chart find the enthalpy of air entering the cooling tower h_a at the entering wet bulb temperature T'_{ae}. Put this value at the top of the third column
- The next value of h_a is calculated by difference using the following formula

$$h_{a,i+1} = h_{a,i} + \frac{m_w}{m_a} Cp_w (T_{w,i+1} - T_{w,i})$$

Fill the data in the third column.

- Calculate the rest of the table from the first three column.
- The tower coefficient is:

$$Tower\ coefficient = \frac{KaV}{m_w} = \sum \frac{\Delta T_w}{(h_s - h_a)}$$

Table 1: Numerical integration for the tower coefficient

T_w	h_s	h_a	$h_s - h_a$	$1/(h_s - h_a)$	$\Delta T_w/(h_s - h_a)$

Constants:

Air density: 1.2 kg/m^3
Area of air flow: 0.00636 m^2
Water density: 1000 kg/m^3
Specific heat of water: 4.187 kJ/kg°C

References:

1. C.J. Geankoplis “Transport Processes and Separation Process Principles”, 4th Ed., Prentice Hall, NJ (2003).
2. W.L. McCabe, J.C. Smith and P. Harriot, “Unit Operations of Chemical Engineering” 7th Ed., McGraw-Hill, New York (2005).
3. R.E. Treybal, “Mass-Transfer Operations”, 3rd Ed., McGraw-Hill, New York (1981).

Experiment 2: SINGLE EFFECT EVAPORATOR

I. Objective:

- The objective of this experiment is to study the performance of a single effect evaporator. Perform mass and energy balances to determine steam economy and heat transfer coefficients.

II. Test Standard

No standards

III. Theory:

Steam economy, which is defined as the mass of water vapor generated per mass of steam is the process parameter most significant in evaluating the performance of evaporators. The other important parameter upon which both the evaporation and condensation process depend is the heat transfer coefficient.

Steam economy*:

$$E = m_v/m_s$$

Heat transfer rate

$$q = UA\Delta T$$

Where

E: steam economy.
m_v: vapor flow rate.
m_s: steam flow rate.
q: heat transfer rate.
U: overall heat transfer coefficient.
A: heat transfer area.
ΔT: temperature difference.

**The above steam economy equation is valid only when steam is used as heating medium* —

Mass and energy balances

Assuming that the stream entering the effect is saturated and that the condensate resulting from the steam is not sub cooled, then the total heat transfer in the effect is obtained as:

$$q = m_s\lambda_s$$

λ_s is the latent heat of vaporization of steam.

The heat given up by the condensing steam is absorbed by the process fluid and used for partial vaporization. From mass and enthalpy balance:

$$q = (m_f - m_p)H_v + m_pH_p - m_fH_f$$

Where:

m_f, m_p: mass flow rates of feed and product
H_v, H_p, H_f: enthalpies of vapor, product and feed

IV. Apparatus:

General description

A schematic diagram of the set-up is shown in Figure 1. Hot water is supplied to the evaporator's reboiler where heat transfer takes place causing partial evaporation of. The vapor generated passes through a cyclone then to a condenser where it is condensed and collected in the condensate tank.

Principles of Operation:

Feed Circuit Two feed tanks with the unit, the larger one is used for potassium chloride solution and the other one is used for deionized water that is used at startup and shutdown. The feed is preheated in a plate heat exchanger then passed to the evaporator where water is evaporated. Water vapor is condensed in a water cooled condenser and collected in the condensate vessel. The concentrated product is collected in the product vessel.

Vacuum Pump The system can operated under reduced pressure using the vacuum pump that is connected to the top of the condensate vessel. The amount of vacuum can be controlled by a needle valve which allows varying amounts of air into the suction of the pump.

V1	Feed selector valve for Potassium Chloride and deionized water tanks.
V2	Evaporator input valve.
V4	Product steam (first position) to condenser.
V5	Selector valve directing product steam to condenser.
V6	Condensate tank and product tank connection valve.
V8	Vacuum control valve.
V9	Product tank drain valve.
V10	Condensate tank drain valve.
V12	Evaporator tube hot water outlet valve.
V14	Evaporator tube hot water inlet valve.
V16	Cooling water control valve.
V17	Selector valve directing condensate either to condensate tank or sample port.
V18	Product flow control valve.
V21	Product tank bleed valve.

T1	Feed temperature after preheating.
T2	Product temperature.
T4	Feed temperature prior to entry into evaporator tube.
T6	Product steam temperature.
T8	Condensate temperature.
T9	Cooling water inlet temperature.
T10	Cooling water outlet temperature.
T11	Heating water temperature (Outlet from jacket at top of evaporator).
T12	Heating water temperature (Inlet to jacket at bottom of evaporator).
C1	Feed conductivity after preheating.
C2	Product conductivity.
WF1	Cooling water flow rate sensor

V. Procedure:

Pre-operation procedure

1. Connect the unit to a suitable power supply.
2. Make sure that all power breakers are set to ON.
3. Make sure all potentiometers are set to zero.
4. Connect the condenser water inlet to a suitable cold water supply and connect the outlet to drain and make sure V16 is open and start the cooling water to the unit.
5. Adjust the set point of the pressurized hot water temperature controller to 125^{o}C, and then turn ON the hot water pump. (wait until the water temperature reaches 90^{o}C before starting to pump the feed solution to the evaporator.
6. Start the feed pump by turning the feed potentiometer to 3.0.
7. Adjust the set point of the feed pre-heater temperature controller to 70^{o}C, and turn ON the pre-heater water circulation pump.

Shut down procedure

1. Switch valve (V1) to feed deionized water from tank to the evaporator.

2. Pump deionized water through the equipment until all Potassium Chloride has been flushed from the system.
3. If the vacuum pump is running, slowly open needle valve V8 to let air into the system. When valve V8 is fully open and the pressure reading has stabilized, switch OFF the vacuum pump.
4. Switch OFF the pump and heater elements of the pressurized hot water.
5. Switch OFF the Pre-heater pump.
6. Wait until the temperature of the feed entering the first evaporator module is less than 65°C, and then turn OFF the potentiometers.
7. Shut OFF the cold water supply and then close valve V16.
8. Turn OFF the mains electricity supply.

Experiment

1. Make sure that valves V2, V4, V6, V8, V12, V14, V16 and V18 are OPEN.
2. Make sure that valves V7, V9, V10, V11, V13, V15, V20 and V21 are CLOSED.
3. Make sure that three way valve V1 is arranged to feed from the deionized water tank.
4. Make sure the three way valve V17 is arranged to direct the condensate to the condensate tank
5. Make sure that the three way valve V5 is arranged to direct the steam to the condenser.
6. Fill the feed tank with 20 liters of 0.5% potassium chloride solution and the deionized water tank with 10 liters of deionized water.
7. Start the unit as explained above.
8. When water starts to boil (T6 will read approximately 100°C) adjust cooling water flow rate to the condenser to be 0.5 liter / min. Increase it if necessary.
9. Adjust valve V18 to keep the cyclone approximately half full of liquid.
10. Once the system has stabilized switch valve V1 to feed from the feed tank.
11. Allow the system to reach steady state (approximately 15 minutes) before taking any readings.
8. For reduced pressure operation; start the vacuum pump then close valve V8 slowly. Make only small adjustments to the valve and always allow a short time period after any adjustment for the system to reach steady state again. ***Do not set the system pressure below 500 mbar***

VI. Experimental Work:

At each operating mode of the evaporator, perform the following:

1. Make material balance (over all and potassium chloride balances).

Overall balance:

$$m_F = m_P + m_V$$

Solute balance:

$$m_F X_F = m_P X_P$$

m_F: Feed mass flow rate.

m_P: Product mass flow rate.

m_V: Vapor mass flow rate.

X_F: Potassium chloride concentration in the feed.

X_P: Potassium chloride concentration in the product.

2. Make energy balance and calculate heat losses.

$$m_F h_F + m_{HW} h_{HW,in} = m_P h_P + m_{HW} h_{HW,out} + m_V h_V + Q_{Loss}$$

m_F: Feed mass flow rate.

m_{HW}: Hot water mass flow rate

h_F: Feed specific enthalpy

h_{HW}: Hot water specific enthalpy

h_P: Product specific enthalpy

h_V: Vapor specific enthalpy

Q_{Loss}: Heat losses

3. Calculate the economy of the evaporator.

$$E = \frac{m_V h_V}{m_{HW}\left(h_{HW,in} - h_{HW,out}\right)}$$

References:

1. C.J. Geankoplis "Transport Processes and Separation Process Principles", 4th Ed., Prentice Hall, NJ (2003).

2. W.L. McCabe, J.C. Smith and P. Harriot, "Unit Operations of Chemical Engineering" 7th Ed., McGraw-Hill, New York (2005).

3. R.E. Treybal, "Mass-Transfer Operations", 3rd Ed., McGraw-Hill, New York (1981).

Experiment 3: CONTINUOUS DISTILLATION

I. Objective:

- The objective of this experiment is to study the principles included in the process of continuous distillation in a tray column under steady state conditions. Also to determine the operating lines on McCabe – Thiele diagram and the number of theoretical plates and the column efficiency

II. Test Standard

No standards

III. Theory:

Definitions

Weeping point Refers to a condition when the vapor velocity through the perforations of a tray is so low that the liquid can't be held out of the perforations and instead of overflowing over the downcomer, the liquid "weeps" through the perforations of a tray. The vapor at this point loses contact with the liquid. This definition does not apply to packed columns.

Flooding point Any countercurrent separation device has a capacity limit. This limit in distillation is known as flooding and is a result of high vapor velocity. At high vapor velocities, the pressure exerted by the vapor from below balances the gravity head of the liquid, as a result liquid starts building up in the column. This condition is reflected by sudden increase in the pressure drop.

Column efficiency The overall column efficiency in a distillation column is defined as the ratio of total number of ideal equilibrium stages to the number of actual trays in the column. An ideal distillation stage is one in which the outgoing liquid and vapor phase establish an equilibrium with each other. High column efficiency indicates a thorough contact between gas and liquid.

Reflux The portion of the vapor that is condensed and then returned to the distillation column.

McCabe – Thiele Method The simplest and perhaps most instructive method for analysis of binary distillation is the graphical approach presented by McCabe and Thiele in 1925. This method is based on the constant molar overflow assumption, which implies constant molar vapor flow rate and constant molar liquid flow rate leaving all stages in a section. Hence in each section of a distillation column, the operating line can be considered a straight line.

McCabe – Thiele Method

When using this method one has to work with mole fractions not mass fractions. From simple material balance the operating lines can be derived. These lines gives a mathematical relation between the composition of the vapor phase and the liquid phase in the column. For the stripping section:

$$y_{m+1} = \frac{L_m}{V_{m+1}} x_m - \frac{W}{V_{m+1}} x_m$$

And for the rectifying section:

$$y_{n+1} = \frac{L_n}{V_{n+1}} x_n - \frac{D}{V_{n+1}} x_D = \frac{R}{R+1} x_n + \frac{x_D}{R+1}$$

The reflux ratio is defined as:

$$R = \frac{L_n}{D}$$

Where:
y: vapor mole fraction.
x: liquid mole fraction.
L: liquid flow rate.
V: vapor flow rate.
W: bottoms flow rate.
D: distillate flow rate.
R: reflux ratio.

For the notations refer to Treybal (1981).

The relative amount of liquid and vapor are in the rectifying and stripping sections are dependent on the feed conditions. The feed can be liquid, vapor or a vapor liquid mixture. The feed quality is defined as:

$$q = \frac{H_V - H_F}{H_V - H_L}$$

The quality line or q-line is given by:

$$y = \frac{q}{q-1}x - \frac{x_F}{q-1}$$

Where:
H: enthalpy.
q: quality line.

The q-line passes through the point of intersection of the operating line of the stripping section and the rectifying section as will as through the point where $x = x_F = y$.

For $q = 0$ the feed is saturated vapor, for $q = 1$ the feed is saturated liquid, for $q > 1$ the feed is sub cooled liquid and for $0 < q < 1$ the feed is vapor liquid mixture.

The minimum reflux ratio is obtained when the operating lines intersects on the equilibrium line resulting in an infinite number of stages. Typically; distillation column must be run at 1.3 – 1.5 of the minimum reflux ratio.

In designing a distillation column; the general procedure is first to determine the reflux ratio, the preferred feed tray location, the number of stages, the duty of the reboiler and the duty of the condenser for a given feed and product specifications. The theoretical number of trays is then adjusted to account for the fact that equilibrium is not achieved completely at each tray. Parameters affecting the tray efficiency include; vapor and liquid flow rates, physical properties, tray design and tray spacing. The overall efficiency and tray efficiency are determined experimentally. The over all efficiency is defined as:

$$E_o = \frac{Number\ of\ theoritical\ trays}{Number\ of\ actual\ trays}$$

IV. Apparatus:

General description

The distillation unit (Figure 1) is designed to operate as either a batch distillation unit or continuous distillation unit. It can be operated at either atmospheric pressure or under vacuum.

Principles of operation

Distillation column The 50 mm diameter column is made up of two glass sections four sieve plates. The columns are separated by a central feed section and arranged vertically for counter-current vapor / liquid flow. The column is insulated to minimize heat loss.

Reboiler The reboiler situated at the base of the column is manufactured from 316 stainless steel and has a heating element. Either batch or continuous distillation can be carried out using this reboiler. In continuous operation, valve V1 is open and bottom product flows from the reboiler through the bottom product cooler to the bottom product tank. It is possible to preheat the feed to the column by directing the feed through a spiral coil in the bottom product cooler.

Condenser Vapor from the top of the column passes to a water-cooled, coil-in-shell condenser. Cooling water enters the condenser at a regulated rate through a variable area flowmeter FI1 and the flow rate is controlled by diaphragm valve V5.

Decanter Condensate is collected in a glass decanter (phase separator) which is by-passed for normal distillation experiments by opening valve V10. When the decanter is in use (separation of two immiscible liquids as condensate), valve V10 is closed.

Manometer The total pressure drop across the column is indicated on a U-tube manometer ΔP1 via appropriate tapings in the column fitted with isolating valves V6 and V7.

Product receiver All of the vessels in the system are connected to a common vent on the top product receiver. This vent is normally connected through a 4.0m length of tubing to a fume cupboard or safe atmospheric vent outlet.

Control consol The console is attached to the process unit by an umbilical cable. Refer to figure 2 for a description of the console controls and connections.

T1	Top tray temperature
T2	2nd tray temperature
T3	3rd tray temperature
T4	4th tray temperature
T5	5th tray temperature
T6	6th tray temperature
T7	7th tray temperature
T8	8th tray temperature
T9	Temperature of liquid in reboiler
V1	Continuous feed valve
V2	Reboiler sample valve
V3	Condenser sample valve
V4	Top product receiver sample valve
V5	Cooling water control valve
V6	Lower lead line of the manometer connection
T10	Temperature of vapor leaving the column above tray 1
T11	Temperature of cooling water entering condenser
T12	Temperature of cooling water leaving condenser
T13	Temp of condensate as reflux / top product
T14	Temperature of feed from feed tank
V7	Lower lead line of the manometer
V8	connection
V10	Dosing vessel flow control valve
V11	Decanter bypass valve
V12	Drain valve
V13	Product recycle valve
V14	Ejector water supply valve
	Ejector flow control valve

Sampling points Samples for analysis can be taken from pertinent points in the system as follows:

Feed liquid: From feed tank
Liquid in reboiler: V2 (WARNING Liquid at boiling point)
Condensate from condenser: V3 (reflux / top product)
Top product receiver: V4
Bottom product receiver: V11

V. Procedure:

Pre-operation procedure

1. Prepare 10 liter solution of 60% (mole basis) water and 40% (mole basis) methanol by mixing exactly 4.005 litter of water and 5.995 liter methanol and charge them to the reboiler.
2. Prepare another 5 liters of the same solution above and charge them to the feeding vessels.
3. Connect the unit to a suitable power supply.
4. Make sure that all power breakers are set to ON.
5. Connect the condenser water inlet to a suitable cold water supply and connect the outlet to drain and make sure V5 is open and start the cooling water to the condenser until the cooling water flow rate FI1 to the condenser is approximately 3 liters / min (adjust if needed).

Experiment

1. On the control consol, switch ON the reboiler's heating element.
2. Adjust the power controller until the wattmeter reads approximately 1.75 kW.
3. Adjust the reflux ratio to total reflux on the control consol (ask the lab supervisor for directions).
4. Wait for the system to reach steady state (15 – 20 minutes).
5. Measure the pressure drop across the column by opening valves V6 then V7.
6. Take sample from the reboiler and the condensate and measure the refractive index.
7. Use the temperature selector switch to display the temperatures and record the temperatures and flow rates on the raw data sheet.
8. Adjust the feed flow rate to be approximately 7.2 liters / hr.
9. Repeat the procedure for a reflux ratio of 5:1.

VI. Experimental Work:

What is the time needed to reach steady state? Comment on your result

What is the effect of reflux ration the operation of the column?

Perform the following calculations for 5:1 reflux ratio:

1. Calculate heat removed by the condenser Q_C.
2. Calculate heat loss from the column

$$Q_L = Q_R - Q_C$$

3. Determine the number of theoretical plates using McCabe – Thiele method.

Calculate the column efficiency

References:

1. C. J. Geankoplis “Transport Processes and Separation Process Principles”, 4th Ed., Prentice Hall, NJ (2003).
2. W. L. McCabe, J.C. Smith and P. Harriot, “Unit Operations of Chemical Engineering” 7th Ed., McGraw-Hill, New York (2005).
3. R. E. Treybal, “Mass-Transfer Operations”, 3rd Ed., McGraw-Hill, New York (1981).
4. J. D. Seader and E.J. Henley, “Separation Process Principles”, Wiley, New York, (1998).
5. R. H. Perry and D. W. Green, "Perry’s Chemical Engineers’ Handbook", 8th ed., McGraw-Hill, USA (2008).

Experiment 4: GAS ABSORPTION

I. Objective:

- The first objective of this experiment is to study the column dynamics under different gas and liquid flow rates and their effect on the pressure drop across the column. The second objective is to demonstrate the absorption of CO2 from air stream by a solution of NaOH and to calculate the overall mass transfer coefficient using gas analysis

II. Test Standard

No standards

III. Theory:

characteristics of the column and, the height is found from mass transfer characteristics of the packing and the gas-liquid system.

Flooding depends on pressure drop across the column, it being higher at the bottom and lower at the top to allow the gas to flow upward. The gas flow is usually turbulent and in a dry column, the pressure drop raises with gas flow with an exponent of 1.8 to 2.0; a typical feature of turbulent flow.

$$\Delta P \alpha V_G$$

Where:

ΔP: the pressure drop across the column.

V_G: gas velocity

n: ranges between 1.8 – 2.0

The pressure drop is largely independent of the tower diameter, but it depends significantly on the nature of the packing. Different packing have different free cross sectional areas. The free cross sectional area, which is related to the voidage (ε), increases with packing size for single type of packing, but differs between packing of the same nominal size, but different forms. The introduction of liquid into the tower increases the frictional resistance and viscous drag force affecting gas flow and so the pressure drop in the wet tower is greater than in the dry tower. If the gas flow rate remains constant, the pressure drop will increase with an increasing the liquid flow rate. This occurs because as the liquid fills the voids in the column, the cross sectional area available for gas flow is reduced. Up to the loading point, the pressure drop follows the same relation as in dry run. Beyond the loading point, the pressure drop rises rapidly with gas flow and the liquid hold up in the column also rises. This continues until gas can no longer pass freely up the column, or the liquid can pass freely down it. The frictional drag force becomes so great that the downward flow of liquid is substantially impeded and the column is to flood. Eventually, at the flooding point, the pressure drop rises drastically and the liquid may splash back from the column. The gas velocity corresponding to the flooding point is called flooding velocity and the column is operated at some fraction of this velocity, say 60%.

Gas absorption

Gas absorption is a mass transfer operation in which a gas mixture is contacted with a liquid to preferentially absorb one or more of the components of the stream. This operation is found in many industries for the recovery of valuable products and cleaning of exhausts or vent streams.

In this case there is a little change in the flow rates of the gas and liquid streams entering and leaving the absorber and they can be assumed to be constant. Also it can be assumed that the mass transfer coefficient is constant.

The quantity of the absorbed material can be calculated from the change in composition of the gas or liquid phases passing through the column.

Mole balance on the gas phase gives

$$N = y_i G_i - y_o G_o = d(Gy)$$

Where:

N: rate of absorption

G_i: gas molar flow rate entering the column.

G_o: gas molar flow rate leaving the column.

y_i: mole fraction of the component in the gas entering the column.

y_o: mole fraction of the component in the gas leaving the column.

For the case of diluted systems $G_i = G_o$.

Mole balance on the liquid phase gives:

$$N = x_i L_i - x_o L_o = d(Lx)$$

Where:

L_i: liquid molar flow rate entering the column.

L_o: liquid molar flow rate leaving the column.

x_i: mole fraction of the component in the liquid entering the column.

x_o: mole fraction of the component in the liquid leaving the column.

For the case of diluted systems $L_i = L_o$.

Performing material balance on a small element of the column with cross sectional area A and a height of dz gives:

$$Nadz = K_{og} aA(y - y^*)dz = d(Gy)$$

Where:

a: effective interfacial area per unit packing.

A: cross sectional area of the column.

K_{og}: overall mass transfer coefficient based on the gas phase.

y^*: equilibrium mole fraction of the component in the gas phase.

Rearranging and integrating the above equation to get height of transfer unit equation of a packed bed:

$$Z = \frac{G}{K_{og} aA} \int \frac{dy}{y^* - y}$$

For diluted component concentrations in an inert gas stream, the above equation can be simplified by assuming that the driving force over the column is the logarithmic mean of the concentration. Another simplification is to assume that the gas phase is an ideal gas, therefore, the concentrations are simply the partial pressures of the component at the top and bottom conditions of the column:

$$K_{og} = \frac{N}{aAZ} \left(\frac{ln(\frac{\bar{P}_i}{\bar{P}_o})}{\bar{P}_i - \bar{P}_o} \right)$$

Where:

$\bar{P}_i$: partial pressure of the component at the bottom of the column

$\bar{P}_o$: partial pressure of the component at the top of the column

IV. Apparatus:

Figure 1 is a schematic diagram of the equipment used in this experiment. The equipment consists of a packed absorption column having a diameter of 0.075 m and a height of 1.4 m and packed with 9 mm glass rashing rings. Pressure drop along the column can be measured by pressure tapings at the top, middle and bottom of the column and by using the manometers the pressure can be measured at any section of the column.

Liquid is pumped from a sump tank of 40 liters capacity by a centrifugal pump to the top of the column through a variable are flow meter. Air is supplied by a compressor and passes through a variable are flow meter. The gas to be absorbed (usually CO_2) is taken from a cylinder connected to the equipment and passes through another variablearea flow meter and mixed with air coming from the compressor and introduced at the base of the aborption column.

V. Procedure:

Pre-operation procedure

1. Fill the sump tank with 35 liters of tap water.
2. Connect the unit to a proper electrical supply.
3. Prime the water manometers with clean water until the meniscus in both tubes is at mid height (250 mm on the scale).
4. Fill the Hempl gas analysis apparatus 1.0 M NaOH up to the ZERO mark on the scale.
5. Connect a CO_2 gas cylinder, fitted with a regulator, to the inlet regulator on the equipment with a flexible tube, and set the cylinder regulator to minimum pressure.
6. Make sure that water and air inlet valves closed, also make sure that gas sampling valves are closed. Make sure that the valve at the discharge of the pump is open

Experiment

A. Column dynamics

NOTE: *This part is conducted using water only*

1. Set the valves V1, V2 and V3 as shown in figure 2.
2. Start the compressor and adjust the air flow rate to be 30 liters / min then read the pressure drop along the column. The total pressure drop is the summation of the pressure drop from the two manometers.
3. Increase air flow rate by 10 until you reach the maximum air flow rate and keep recording pressure drop for each flow rate.
4. Switch off the air compressor.
5. Start the water pump and adjust water flow rate to be 1 liter / min.
6. Start air compressor and adjust air flow rate to be 30 liters / min and record the pressure drop.
7. Increase air flow rate by 10 until you reach maximum air flow arte or the column floods and keep recording pressure drop.
8. Repeat steps 6 and 7 for water flow rates of 2 and 3 liter / min.

NOTE: *Once liquid holdup in the column is observed, take data at very tiny increments in gas flow rate in order to record flooding data. Patience is needed to obtain accurate steady-state readings especially in the holdup / flooding region. At a given liquid and gas flow rate in the vicinity of flooding, the pressure across the packed bed should rise slowly until the liquid holdup in the column reaches its maximum, but the pressure readings will* *probably oscillate. When visual flooding is observed for each run, take extra data points at air flow rates just prior to flooding and slightly higher if possible*

B. Gas absorption

1. Weigh accurately 280.00 g of sodium hydroxide pellets.
2. Add the pellets to the sump tank gradually and stir with a paddle until all the pellets are dissolved.

***WARNING:** Wear gloves and goggles.*

3. Start the pump and adjust the sodium hydroxide flow rate at 3 liters / min.
4. Start the air compressor and adjust air flow rate at 30 liters / min.

***WARNING:** The column must not be operated near the flooding point since the solution used is caustic. If any indication of flooding is noticed immediately close the air supply.*

5. Open the pressure regulator on the CO_2 cylinder and adjust the gas flow rate to 3 liters / min.
6. After the system reaches steady operation (approximately 15 – 25 minutes) take a sample from the air leaving the absorption column for analyzing by Hempel test apparatus. (refer to the analysis procedure in the following section).

C. Gas analysis

1. Flush the sample lines by repeated suction from the line using the gas piston and expelling the contents to the atmosphere. Note that the volume of the cylinder is about 100 ml, therefore; three volumes of the piston is sufficient.
2. With the absorption globe isolated and the vent to the atmosphere closed, fill the cylinder from the selected line by drawing the piston out slowly. Take a sample of about 20 ml. Wait a two minutes for the gas to come to the temperature of the cylinder. (V1 = 20 ml).
3. Isolate the cylinder from the column and the absorption globe and vent the cylinder to atmospheric pressure. Close the line to the atmosphere after about 10 seconds.
4. Connect the cylinder to the absorption globe. The liquid level should not change. If it changes open the vents shortly to atmosphere again then close it.
5. Wait until the level in the indicator tube is on ZERO this means that the pressure in the cylinder is atmosphere.
6. Slowly push the piston to empty the cylinder into the absorption globe, then slowly pull the piston out again
7. Repeat the step above several times until no change is observed on the volume reading.
8. Read the indicator tube marking; this represents the volume of the gas absorbed in the sample (V2).
9. Repeat this procedure for three samples and take the average volume (V2).

***NOTE:** Refer to figure 3 and figure 4 in the appendix for more details.*

VI. Experimental Work:

A. Column dynamics

1. Prepare A log-log plot of $\Delta P/H$ vs. V_G with liquid flow as a parameter where ΔP is the pressure drop across the top and bottom packing sections of the tower (Pa), V_g is the superficial gas velocity [volumetric flow rate (m^3 / sec) / tower cross sectional area (m^2)] and H is the packing height. The slope of the line is the exponent of the correlation:

$$\ln \Delta P/H = a + n \ln V_G$$

This equation is valid for two phase counter current flow in the packed tower from dry bed conditions to the loading point. The date for the dry beds should be on the same graph. Comment on the value of n obtained.

2. Prepare plots of ΔP versus G, where G is the mass velocity of the gas (kg / sec m^2) based on the empty tower cross section. The loading region and flooding point should be identified on the graphs. Liquid mass velocity, L, in kg/(sec.m^2) should be the parameter. Also include the dry bed data.
3. Choose a constant gas flow rate below the loading region for all liquid flow rates examined and prepare a log-log plot of ΔP vs. L, where L is the liquid mass velocity (kg / sec m^2) and comment on the shape of the graph.
4. Based on your results above which have more significant effect on the column pressure drop; gas or liquid flow rate. Explain your results

B. Gas absorption

1. Calculate the mole flow rate of the gas entering the column (CO_2 and air) assuming ideal gas. make use of the fact that the gas specific molar volume is 22.42 liter / mole at 1 atm and 273 K.
2. Calculate the mole fraction of CO_2 at the inlet of the column from material balance.
3. Calculate the mole fraction of CO_2 at the outlet of the column from the Hempel test by assuming ideal gas and using the fact that volume fractions are equal to mole fractions for ideal gases.
4. Calculate the overall gas mass transfer coefficient and comment on your result.

NOTE:

Column specifications:

Column height: 1.4 m

Column diameter: 0.075 m

Effective interfacial area per unit packing: 440 m^2/m^3

References:

1. C. J. Geankoplis "Transport Processes and Separation Process Principles", 4th Ed., Prentice Hall, NJ (2003).
2. W. L. McCabe, J.C. Smith and P. Harriot, "Unit Operations of Chemical Engineering" 7th Ed., McGraw-Hill, New York (2005).
3. R. E. Treybal, "Mass-Transfer Operations", 3rd Ed., McGraw-Hill, New York (1981).
4. J. D. Seader and E.J. Henley, "Separation Process Principles", Wiley, New York, (1998).
5. R. H. Perry and D. W. Green, "Perry's Chemical Engineers' Handbook", 8th ed., McGraw-Hill, USA (2008).

Experiment 5: LIQUID – LIQUID EXTRACTION

I. Objective:

- The objective of this experiment is to demonstrate the process of liquid – liquid extraction in a continuous counter current packed bed extractor. The equilibrium data of the system under study will be generated using batch experiments. In addition, the following will be determined: mass transfer coefficient, the number of transfer units and the height of transfer unit.

II. Test Standard

No standards

III. Theory:

Distribution coefficient

The distribution of a compound between two distinct phases is based on the affinity of the compound towards those phases. At equilibrium and at a given temperature, the ratio of concentrations of a compound in two distinct phases is a constant and defined as the distribution coefficient K_D:

$$K_D = \frac{y_A}{x_A}$$

Where:

transferred from one phase onto the other, if $K_D >> 100$. If $K_D < 100$, multiple extractions have to be carried out to transfer **A** from one phase to another.

McCabe – Thiele Method

This method requires the equilibrium curve and an operating line. This method assumes that the system is is0thermal, isobaric and the heat of mixing is negligible, this is true for diluted systems. Another simplification to the method is when dealing with immiscible liquids. In this case the mass fractions can be related to mass ratios as follows

$$X = \frac{x}{1-x}$$

$$Y = \frac{y}{1-y}$$

Where:

X: mass ratio of solute in raffinate.

Y: mass ratio of solute in extract.

Performing mass balance at the extractor will give the operating line equation which will be a straight line that passes through the points (X_0, Y_1) and (X_1, Y_0), the slope is"

$$Slope = \frac{F}{S}$$

Where:

X_0, X_1: mass ratio of the solute in the solvent at the bottom and top of the column.

Y_0, Y_1: mass ratio of the solute in the raffinate (feed) at the bottom and top of the column.

F: feed mass flow rate which equals the raffinate mass flow rate for diluted systems

S: solvent mass flow rate equals the extract mass flow rate for diluted systems

The number of transfer units can be determined using the equilibrium data plotted as mass ratios and by plotting the operating line with a slop of F/S that passes through the point (X_0,

Y_1). The composition of the raffinate leaving the column X_1 can also be determined from the plot.

The overall mass transfer coefficient is defined as:

$$K = \frac{Rate\ of\ mass\ transfer\ (N)}{Volume\ of\ packing\ \left(V_{Packing}\right) \times Mean\ driving\ force(MDF)}$$

The mean driving force between the top and bottom of the column can be defined in two ways; based on the extract phase and based on the raffinate phase. Therefore the mass transfer coefficient can be defined based on two ways; K_E (based on extract phase) K_R (based on raffinate phase):

Based on extract phase:

$$MDF = \frac{(Y - Y^*)_{Top} - (Y - Y^*)_{Bottom}}{\ln \frac{(Y - Y^*)_{Top}}{(Y - Y^*)_{Bottom}}}$$

Based on extract phase:

$$MDF = \frac{(X - X^*)_{Top} - (X - X^*)_{Bottom}}{\ln \frac{(X - X^*)_{Top}}{(X - X^*)_{Bottom}}}$$

Where:

Y^*: the mass ratio in extract phase that is in equilibrium with X.

X^*: the mass ratio in extract phase that is in equilibrium with Y.

The height of transfer unit is also can be defined based on the extract or the raffinate phases:

$$H_E = \frac{S}{SK_E}$$

$$H_R = \frac{F}{SK_R}$$

Where:

H_E: height of transfer unit based on extract phase.

H_R: height of transfer unit based on raffinate phase.

K_E: mass transfer coefficient based on extract phase.

K_R: mass transfer coefficient based on raffinate phase.

S: cross sectional are of the column.

IV. Apparatus:

he equipment consists of a packed extraction column, which is packed with raschig rings and equipped with feed tank, solvent tank, raffinate tank and extract tank. The feed and solvent tanks are connected to pumps and flow meters. The solvent is pumped to the top of the column (for this experiment) and the feed is pumped to through the bottom of the column. The two phases are allowed to contact over the packing, the raffinate phase leaves from the top to the raffinate tank or recycled to the feed tank and the extract phase leaves from the bottom of the column to the extract tank or recycled to the solvent tank. The equipment is also provided with a distillation unit that can be used to recover the solvent. Figure 1 is a schematic diagram of the apparatus.

1 Extraction column
2 Distillation unit
3 Extract tank
4 Feed and solvent flow meter
5 Control cabinet
6 Distillate vessel
7 Feed tank
8 Raffinate tank
9 Feed pump
10 Solvent pump
11 Solvent tank
12 Over pressure valve
13 Regulating valve
14 Frame

V. Procedure:

Pre-operation procedure

1. Fill the solvent tank with appropriate amount of distilled water.
2. Connect the unit to a proper power supply
3. Prepare the feed solution by dissolving 48 g of benzoic acid in 20 liters of petroleum ether then add the solution to the feed tank
4. Prepare a suitable quantity of separation flasks, beakers (250 ml), graduated cylinders (10 ml, 50 ml and 250 ml)

Experiment

A. Batch extraction

1. Dissolve a known weight of benzoic acid in 100 ml of water and add it to the separation vessel
1. Add 100 ml of petroleum ether to the separation flasks.
2. Shake the flask vigorously for 10 minutes.
3. Allow the contents of the funnel to separate.
4. Remove the aqueous layer to a beaker and measure its conductivity

NOTE: *You need at least 6 points in the concentration range of 0 – 1.2 g / liter*.

B. Continuous extraction

1. Close the regulating valve and start the feed pump and fill the column with the feed. Direct the raffinate to go back to the feed tank.
2. Start the solvent pump and direct the extract to the extract tank
3. When you see a clear phase interface between the feed and solvent in the clear space at of the column open the regulating valve.
4. Adjust the feed and solvent flow rates to maintain the phase interface at constant height.
5. Once the flow rates are stabilized direct the raffinate to the raffinate tank.
6. After the system reaches steady state take a sample from the extract and measure the conductivity.

VI. Experimental Work:

A. Batch extraction

1. Calculate the concentration of benzoic acid in the extract phase from the conductivity data.

2. Calculate the concentration of benzoic acid in the raffinate phase by performing mass balance.
3. Construct the equilibrium curve of the system (X vs. Y).

B. Continuous extraction

1. Construct the operating line.
2. Calculate number of transfer units.
3. Calculate mass transfer coefficient.
4. Calculate height of transfer units.
5. Calculate the height of packing and compare your result with the actual height of packing of the column.

Specific gravity of petroleum ether: 0.76
Diameter of the column: 0.05 m
Height of packing: 1.5 m

References:

1. C. J. Geankoplis "Transport Processes and Separation Process Principles", 4th Ed., Prentice Hall, NJ (2003).
2. W. L. McCabe, J.C. Smith and P. Harriot, "Unit Operations of Chemical Engineering" 7th Ed., McGraw-Hill, New York (2005).
3. R. E. Treybal, "Mass-Transfer Operations", 3rd Ed., McGraw-Hill, New York (1981).
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5. R. H. Perry and D. W. Green, "Perry's Chemical Engineers' Handbook", 8th Ed., McGraw-Hill, USA (2008).
6. J. M. Coulson and J. F. Richardson, Chemical Engineering Volume 2", 5th Ed., Butterworth-Heinemann, England (2002).

Experiment 6: ION EXCHANGE

I. Objective:

- The objective of this experiment is to demonstrate the operation of ion exchange unit. The effect of cation and anion resins on conductivity will be examined. Also the combined effect of both resins on the total removal of ions will be examined

II. Test Standard

No standards

III. Theory:

The general principle of ion exchangers is that some ions are favored more than the ions present on the resin. The stronger ion removes the weaker from the resin. For example, Na^+ is removed by Ca^{2+} but in turn Ca^{2+} is removed by Al^{3+}. Therefore, unwanted ions in the solution are always attracted more strongly than the ions that are present in the resin, which are given up during the exchange process. For example, removing Ca^{2+} cations and HCO_3 anions from untreated water involves the following processes:

The H^+ ions and OH^- ions given from the resin combines with each other to produce water. Ion exchange is a reversible process, this means that for a resin that is exhausted it can be restored to its active state by removing the adsorbed ions by providing H^+ ions and OH^- ions. This is called the regeneration process and it is usually done by using solutions of strong acids and strong bases

Each type of resin has a maximum capacity which is the quantity of ions that can be removed from the solution and it is expressed as mmol / L or equivalents / L. In practice the theoretical total capacity can not be reached due to many reasons, therefore; the volumetric usable capacity **NVK** is used instead. The NVK depends on many factors such as: water composition, ratio of resin to water flow rate and temperature. The theoretical amount of water that can be treated is expressed as:

$$V_{H_2O} = \frac{NVK.V_T}{K_{H_2O}}$$

Where:

V_{H2O}: water volume

NVK: volumetric usable capacity

V_T: Volume of resin

K_{H2O}: concentration of ions in water

IV. Apparatus:

The ion exchange unit used in this experiment consists of two transparent plastic pipes filled with ion exchanger resin. A supply tank with four compartments for test water, distilled water, HCl solution and NaOH solution that will be used during the experiment. The effluent from the ion exchange unit passes to a measuring tank to measure its conductivity and then collected in the collecting tank. Figure 1 is a schematic diagram of the unit.

1 Pipes containing the resin
2 Flow meter
3 Measuring tank
4 Conductivity meter
5 Pump switch
6 Intake pipe
7 Pump
8 Supply tank
9 Collecting tank

V. Procedure:

Pre-operation procedure

1. Fill one of the pipes with acidic cation resin (about 40 – 60 mm level, approximately 14 – 20 ml).
2. Fill the other pipe with basic anion resin (about 40 – 60 mm level, approximately 14 – 20 ml).
3. Fill the suply tank as follows
    - One compartment with distilled water (for regeneration).
    - One compartment with 2% NaOH solution (for regeneration of basic anion resin).
    - One compartment with 5% HCl solution (for regeneration of acidic cation resin).
    - One compartment with test water or tab water
4. If fresh resin used it must be rinsed with distilled water

Regeneration

The regeneration should be always performed before starting the experiment and when you are instructed to do during the experiment. This is done to make sure that the resin is fresh.

1. Adjust the valves so that the regeneration solution enters at the top of the pipe and flows down.
2. Insert the intake pipe into the proper regeneration solution compartment.
    - HCl solution is used to regenerate the cation resin.
    - NaOH solution is used to regenerate the anion resin.
3. Switch ON the pump and adjust the flow rate to be about 40 ml / min for at least 4 minutes
4. Take the intake pipe and place it in the distilled water compartment and rinse the resin until the conductivity of the outlet water is the same as the distilled water.

Experiment

A. Exchanging cations

1. Place the intake pipe in the test water compartment.
2. Adjust the valves so the water flows to the top of the cation resin pipe.
3. Switch on the pump and adjust the flow rate to be about 80 ml / min.
4. At regular intervals of 1 minute record the conductivity of the outlet water for 20 minutes.

B. Exchanging anions

1. Switch OFF the pump and adjust the valves so that the water flows to the top of the anion resin pipe.
2. Switch ON the pump and adjust the flow rate to be about 80 ml / min.
3. At regular intervals of 1 minute record the conductivity of the outlet water for 20 minutes.

C. Total demineralization

1. Regenerate the resins as described in the regeneration section.
2. Adjust the valves so that the water flows to the top of the cation resin pip, the outlet is fed to the top of the anion resin pipe and flows to the measuring tank.

3. Switch ON the pump and adjust water flow rate to be about 80 ml / min.
4. At regular intervals of 1 minute record the conductivity of the outlet water until it is the same as the test water.
5. Regenerate the resins as described in the regeneration section (record the data in the raw data sheet for this regeneration only
6. Switch ON the pump and adjust water flow rate to be about 120 ml / min.
7. At regular intervals of 1 minute record the conductivity of the outlet water until it is the same as the test water.

VI. Experimental Work:

1. Calculate the minimum regeneration time for both the cation and anion resins and compare with the time you used in your experiment.
    - The maximum capacity of the resin is its NVK value times the volume of the bed
    - The minimum amount of ions required to regenerate the resin is equal to its maximum capacity.
    - From the preceding information the volume of the solution required for the regeneration and the time can be calculated.
2. Plot the conductivity versus time for all parts of the experiment and comment on your results.

References:

1. J. D. Seader and E.J. Henley, “Separation Process Principles”, Wiley, New York, (1998).
2. R. H. Perry and D. W. Green, "Perry’s Chemical Engineers’ Handbook", 8th ed., McGraw-Hill, USA (2008).
3. Kirk-Othmer Encyclopedia of Chemical Technology

Experiment 7: LEVEL CONTROL

I. Objective:

- The objective of this experiment is to demonstrate two points closed loop control and to test P, PI, PD, and PID controllers at different parameters to control the level in a tank

II. Test Standard

No standards

III. Theory:

Two point closed loop control system

Simply it is known as on – off feedback control system, it is cheap, easy and effective. In some cases they may represent good design choice. In this control system when the process variable PV drops below or increases more than a certain value that is set point SP; a signal is sent to the control element such as a pump, compressor, or valve to be closed or opened. In our case, the level inside the tank is controlled by switching the pump ON or OFF. If the level drops below the desired value the pump will turn ON to fill the tank and if the level increases the pump will turn OFF.

In most applications of on – off feedback control, some consideration needs to be given to other costs, such as wear and tear of control valves and perhaps other start-up costs when power is reapplied each time the PV drops. Therefore, practical on – off control systems are designed to include hysteresis: there is a dead band, a region around the set point value in which no control action occurs. The width of dead band may be adjustable or programmable

Continuous control system

Sometimes it is called linear control systems, it uses a linear negative feedback to produce a control signal mathematically based on other variables, with a view to maintain the controlled process within an acceptable operating range. The output from a linear control system into the controlled process may be in the form of a directly variable signal, such as a valve that may be 0 or 100% open or anywhere in between. Sometimes this is not feasible and so, after calculating the current required corrective signal, a linear control system may repeatedly switch an actuator, such as a pump, motor or heater, fully on and then fully off again.

PID control Is a control loop feedback mechanism (controller) widely used in industrial control systems. A PID controller calculates an error value as the difference between a measured process variable and a desired set point. The controller attempts to minimize the error in outputs by adjusting the process control inputs.

The PID controller algorithm involves three separate constant parameters, and is accordingly sometimes called three-term control: the proportional, the integral and derivative values, denoted P, I, and D. Simply put, these values can be interpreted in terms of time: P depends on the present error, I on the accumulation of past errors, and D is a prediction of future errors, based on current rate of change. The weighted sum of these three actions is used to adjust the process via a control element such as the position of a control valve, a damper, or the power supplied to a heating element.

By tuning the three parameters in the PID controller algorithm, the controller can provide control action designed for specific process requirements. The response of the controller can be described in terms of the responsiveness of the controller to an error, the degree to which the controller overshoots the set point, and the degree of system oscillation. Note that the use of the PID algorithm for control does not guarantee optimal control of the system or system stability.

Some applications may require using only one or two actions to provide the appropriate system control. This is achieved by setting the other parameters to zero. A PID controller will be called a PI, PD, P or I controller in the absence of the respective control actions. PI controllers are fairly common, since derivative action is sensitive to measurement noise, whereas the absence of an integral term may prevent the system from reaching its target value due to the control action.

PID control theory The proportional, integral, and derivative terms are summed to calculate the output of the PID controller. Defining *u(t)* as the controller output, the final form of the PID algorithm is:

$$u(t) = MV(t) = K_p e(t) + K_i \int_0^t e(\tau) d\tau + K_d \frac{d}{dt} e(t)$$

Where
K_p: Proportional gain, a tuning parameter
K_i: Integral gain, a tuning parameter
K_d: Derivative gain, a tuning parameter
e: Error = SP – MV
t: Time or instantaneous time (the present)
τ: Variable of integration; takes on values from time 0 to the present t

Proportional control Proportional negative-feedback systems are based on the difference between the required set point (SP) and process value (PV). This difference is called the *error*. Power is applied in direct proportion to the current measured error, in the correct sense so as to tend to reduce the error (and so avoid positive feedback). The amount of corrective action that is applied for a given error is set by the gain or sensitivity of the control system.

At low gains, only a small corrective action is applied when errors are detected: the system may be safe and stable, but may be sluggish in response to changing conditions; errors will remain uncorrected for relatively long periods of time: it is over-damped. If the proportional gain is increased, such systems become more responsive and errors are dealt with more quickly. There is an optimal value for the gain setting when the overall system is said to be critically damped. Increases in loop gain beyond this point will lead to oscillations in the PV; such a system is under-damped.

Integral control The contribution from the integral term is proportional to both the magnitude of the error and the duration of the error. The integral in a PID controller is the sum of the instantaneous error over time and gives the accumulated offset that should have been corrected previously. The accumulated error is then multiplied by the integral gain K_i and added to the controller output.

The integral term accelerates the movement of the process towards set point and eliminates the residual steady-state error that occurs with a pure proportional controller. However, since the integral term responds to accumulated errors from the past, it can cause the present value to overshoot the set point value.

Derivative control The derivative of the process error is calculated by determining the slope of the error over time and multiplying this rate of change by the derivative gain K_d. The magnitude of the contribution of the derivative term to the overall control action is termed the derivative gain, K_d.

Derivative action predicts system behavior and thus improves settling time and stability of the system. An ideal derivative is not causal, so that implementations of PID controllers include an additional low pass filtering for the derivative term, to limit the high frequency gain and noise. Derivative action is seldom used in practice because of its variable impact on system stability in real world applications.

IV. Apparatus:

In this experiment MPS® PA Level Workstation Manufactured by FESTO will be used with Fluid Lab® software. The unit can be used for real control of level or in simulation mode.
The basic components of the unit are:
Figure 1: MPS® PA Level Workstation

- Water tanks
- Manual valves
- Manual valve for drain
- Piping system including four transparent segments
- Centrifugal pump
- Motor Controller for pump
- Analog ultrasonic sensor
- Two capacitive proximity switch for min/max level in lower tank
- Float switch for threshold function (electromechanical) in upper tank
- Float switch for overflow alarm monitoring in lower tank
- I/O Terminal
- Terminal for analog signals
- Signal converter: current to voltage
- PLC or closed-loop controller
- Control panel
- Service Unit
- 19’’ mounting frame.

V. Procedure:

Pre-operation procedure

1. Make sure that the lower tank is filled with water.
2. Start Fluid Lab® software.

Experiment

D. Two points closed loop control

1. Open valve 101 totally and open valve 112 by 2/3.
2. Open the **Closed-loop Control – 2 points** menu.
3. On the main window click on **Select manp. Value** tap and then select **Digital Output 3** as the manipulated value, this will control the pump operation to maintain the level in the tank at the desired set point.
4. Define the **Setpoint** (4 V for example).
5. Define the **Switching** value to be 0.5 V.
6. Start the process by clicking **Start** button.
7. Wait until the system reaches steady state.
8. Change the **Switching** values to 1 then to 0.2 and observe the operation of the pump and the change in process variable.
9. To save your results click on the save as jpeg in the documentation section at the bottom of the main window and save your file with an appropriate name.

10. Stop the process and exit.

E. Continuous closed loop control

1. Open valve 112 by 2/3.
2. Open the **Closed loop Control – continuous** menu.
3. On the main window click on **Select manp. Value** tap and then select **A_OUT1** as the manipulated value, this will control the pump in analog mode.
4. Set the **Actual Value** to 0 which represents **Channel 0** for the level sensor.
5. Switch the **Digital Output 2** to 1 to activate the pump in analog mode.
6. Define the **Setpoint** (2 V for example).
7. Chose **P** controller and set **K_p** to 2.0.
8. Start the process by clicking **Start** button.
9. Wait until the system reaches steady state then save your results. Then Stop the process.
10. Change the value of **K_p** as shown in the table below and repeat steps 7 – 10.
11. Repeat the procedure for **PI(DIN)**, **PD** and **PID**. The values of controller parameters are shown in the table below.

CONTROLLER TYPE	RUN 1			RUN 2			RUN 3			RUN 4		
	K_p	T_i	T_v	K_p	T_i	T_v	K_p	T_i	T_v	K_p	T_i	T_v
P	2.0	–	–	4.0	–	–	6.0	–	–	8.0	–	–
PI(DIN)	1.0	1.0	–	1.0	4.0	–	4.0	8.0	–	6.0	8.0	–
PD	1.0	–	0.2	1.0	–	0.6	5.0	–	0.8	6.0	–	0.8
PID	4.0	8.0	0.1	2.0	6.0	0.1	6.0	8.0	0.3	6.0	8.0	0.5

VI. Experimental Work:

From the data obtained from continuous control what is the time required to reach steady state for each type of controllers and for the different control parameters and what is the offset if there was any. Put your results in a tabulated form. Based on your results what is the best type of controllers for level control and compare with literature

References:

1. D. Seborg, T. Edgar, D. Mellichamp and F. Doyle III "Process Dynamic and Control", 3rd Ed., Wiley US, (2010).
2. C. Smith and A. Corripio "Principles and Practice of Automatic Control", 3rd Ed., Wiley US, (2005).
3. W. Luyben "Process Modeling, Simulation, and Control for Chemical Engineers", 2nd Ed., McGraw Hill US, (1996).
4. G. Stephanopoulos "Chemical Process Control: An Introduction to Theory and Practice", Prentice Hall US, 1984.

Experiment 9: FLOW CONTROL

I. Objective:

- The objective of this experiment is to test P, PI, PD, and PID controllers at different parameters to control the flow inside a pipe, also the optimum controller parameters will be calculated using Ziegler – Nichols closed loop method

II. Test Standard

No standards

III. Theory:

NOTE: *Refer to Level Control experiment for more theoretical back ground.*

Ziegler – Nicols method for tuning PID controllers

PID controllers are probably the most commonly used controller structures in industry. They do, however, present some challenges to control and instrumentation engineers in the aspect of tuning of the gains required for stability and good transient performance. There are several prescriptive rules used in PID tuning. An example is that proposed by Ziegler and Nichols in the 1940's. There are two methods to determine the optimum controller parameters. The first method depends on the system response to a unit step change in open loop system. The second one is based on the closed loop system response to increasing the K_p in a P only controller.

Open loop tuning

In this method, the response of the system is obtained to a unit step change in the input. The response curve may look S – shaped (see Figure 1). The S – shape can be characterized by two constants; delay time **L** and time constant **T**. the delay time and time constant can be determined by drawing a tangent line at the inflection point of the S – shaped curve and determine the intersections with the time axis and the steady state line K (see figure 1). The controller parameters can then be calculated (refer to Table 1 in the Appendix).

Closed loop tuning

In this method P only controller is used by setting Ki and Kd to zero. The Kp is increased from zero until it reaches a critical value Kp,cr where the output first exhibits sustained oscillation (see Figure 2). If the system does not exhibit sustained oscillation for whatever value of Kp, then this method do not apply.

The critical gain Kp,cr and the critical oscillation period Pcr can be determined experimentally and the controller parameters then can be calculated (refer to Table 2 in the Appendix)

IV. Apparatus:

In this experiment Fluid Lab® software will be used to demonstrate the flow control between two tanks in the simulation mode

V. Procedure:

Pre-operation procedure

1. Start Fluid Lab® software.
2. Click on setup tab then change from EASYPORT to Simulation and close the setup window. Now the simulation mode is active.

Experiment

A. Continuous closed loop control

1. Open the **Closed loop Control – continuous** menu.
2. Click on **Select closed** to select the process then click on **Flow** for flow control.

3. Activate the simulated process by clicking the **ON** button and a new window will appear showing a schematic simulation diagram of the process.
4. On the schematic simulation window set the process speed to 100%.
5. On the schematic simulation window close valve 112.
6. Switch the **Digital Output 0** to 1 to activate the simulation process.
7. Switch the **Digital Output 2** to 1 to activate the pump in analog mode.
8. Define the **Setpoint** (0.2 for example).
9. Chose **P** controller and set $\mathbf{K_p}$ to 2.0.
10. Start the simulation by clicking **Start** button.
11. Wait until the system reaches steady state then save your results.
12. Stop the simulation and close the schematic simulation window.
13. Change the value of $\mathbf{K_p}$ as shown in the table bellow and repeat steps 2 – 12
12. Repeat the procedure for **PI(DIN)**, **PD** and **PID**. The values of controller parameters are shown in the table below.

CONTROLLER TYPE	**RUN 1**			**RUN 2**			**RUN 3**			**RUN 4**		
	K_p	T_i	T_v	K_p	T_i	T_v	K_p	T_i	T_v	K_p	T_i	T_v
P	2.0	–	–	4.0	–	–	6.0	–	–	8.0	–	–
PI(DIN)	1.0	1.0	–	1.0	4.0	–	4.0	8.0	–	6.0	8.0	–
PD	1.0	–	0.2	1.0	–	0.6	5.0	–	0.8	6.0	–	0.8
PID	4.0	8.0	0.1	2.0	6.0	0.1	6.0	8.0	0.3	6.0	8.0	0.5

B. Tuning PID controller:

1. Repeat the steps above or P only controller, but this time start from $K_p = 0$.
2. Increase K_p gradually until the system exhibits sustained oscillation then save your results.

VI. Experimental Work:

1. From the data obtained from continuous control what is the time required to reach steady state for each type of controllers and for the different control parameters and what is the offset if there was any. Put your results in a tabulated form. Based on your results what is the best type of controllers for flow control and compare with literature.
2. Calculate the optimum controller parameters using Ziegler – Nichols method for closed system.

References:

3. D. Seborg, T. Edgar, D. Mellichamp and F. Doyle III "Process Dynamic and Control", 3rd Ed., Wiley US, (2010).
4. C. Smith and A. Corripio "Principles and Practice of Automatic Control", 3rd Ed., Wiley US, (2005).
5. W. Luyben "Process Modeling, Simulation, and Control for Chemical Engineers", 2nd Ed., McGraw Hill US, (1996).
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