Gazi University

Engineering Faculty

Chemical Engineering Department

ChE 482 Chemical Engineering Laboratory III

Experiment ID: 1a-PRESSURE CONTROL

Theory

      The aim of the pressure process training set is to give practical application of the control theory in the pressure control system. This can be accomplished by carrying out a series of experiments which will demonstrate the theory through experiments and thus reinforce the knowledge.

The training set is shown in Figure-1.
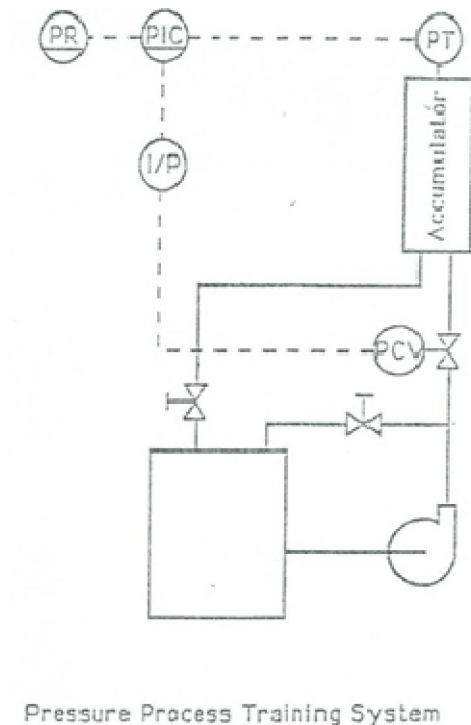

Pressure Process Training System

Figure 1.

Experimental Set-Up

      The experiment system includes a collecting container from which water is supplied by means of a centrifugal pump from a reservoir. The water passes through a control valve (PCV) so the pressure increases and can flow back to the reservoir by means of a manually

operated needle valve. The system has a manually controlled bypass valve that is capable of change the flow.

The pressure in the collecting container is measured by means of a pressure transducer mounted on the top of the container. The signal (PIC) from the pressure transmitter provides process variable to controller. The controller output provides PCV and consequent I/P flow to the pressure transmitter controlling the pressure in the flow and collecting container by regulating the air compressor.

PRESSURE CONTROL SYSTEM COMPONENTS

The purpose of the control system is to equalize the received process variable (PV) (pressure in the collecting container) to the required value or set point (SP). This is accomplished by measuring the deviation "d" within the controller. Here, the difference between the setpoint and the process variable, d, is expressed as

d=SP-PV   (1)

The output of the controller is;

O = f (d), which is a function of the deviation and control algorithm.

The controller output is fed to an I / P converter device that controls a valve and performs the task of reducing the deviation. That is, if the pressure is too low, the control valve will be opened, so more water will enter the collecting container and the pressure will increase.

A control system therefore has to have at least three elements.

1. A transmitter that will measure the amount of the controlled process variable and send the value to the controller.

2. A controller that applies control strategy to the value from the transmitter and provides output to the controller

3. Final controller element, i.e. a pneumatic valve

Controller type;

a. Proportional controller (P)

b. Proportional integral controller (PI)

c. Proportional integral derivative controller (PID)

1. Proportional Controller (P)

Objective: To improve the meaning of proportional band

Preparation

The following preparations should be made on the system before starting the experiment.

1. The water reservoir must be filled with 19 liters of water and 1 liter of corrosion inhibitor liquid.
2. Close the collecting container needle valve and open the bypass valve.
3. Bring the controller to manual mode and increase output by opening the control valve.
4. Increase the pressure in the collecting container to approximately 80 mb.
5. Close the control valve and disconnect the compressed air source by bringing the controller output to zero (the compressed air value should be set to 1.4 bar).
6. Turn-off the pump.

Controller

1. Set the RESET time value to 0 by pressing the FUNC key twice.
2. Press the same key again to bring PROP BD to 25.
3. Set RATE to OFF position.

Experimental procedure

1. If the output is still not 0 in the manual control mode with the controller, set this value to 0.
2. Return to the SPLOC screen and equalize the controller set point to the process variable.
3. Switch to AUTO mode from MANUAL mode.
4. Record the process value (PV), set point (SP), deviation (d) and output (O) values.
5. Repeat the same procedure for the new set point for PB values of 50,100,200 and 400 by increasing the set point in percentage of the PV value.

Calculations

1. Plot the results you find against the deviation (d versus O).
2. Calculate the slopes for each PB value.
3. Express output as a function of the deviation for proportional control.
4. What will be the output at a deviation of 13% from the setpoint at PB = 50?

Overflow control

Objective: The purpose of this experiment is to show the limits of proportional (P), proportional integral (PI) and proportional integral control (PID), in the case of overflow current.

Pre-experiment

1. Turn on the power switch and set the RESET value to 50.0 in the control algorithm after completing the controller autoset. If necessary, bring the inlet and setpoint interval to 500 or the highest possible pressure in the collecting container.

2. Close the collecting container needle valve and open the bypass valve.

3. Open the compressed air source and set the inlet pressure to 1.4 mb. By opening the pump, bring the pump speed to the maximum value and close the bypass valve.

4. Bring the controller to manual mode and set the output value to 40% by opening the control valve.

5. Open the outlet valve of the collecting container and change the valve position until the displayed pressure is approx. 200 mb. Wait until the pressure is steady.

Experimental procedure

1. In the manual control mode, try changing the pressure by the controller to 100 mb. This will cause the controller output to change.

2. Bring the controller output back to the old position of 40% and allow the pressure to come to the steady state. Set the following controller settings. (PB = 100, RATE = OFF and SP = PV, SP).

3. Bring the controller to automatic mode from manual control mode.

4. Increase the set point by 100 mb and record this value.

5. Wait until the pressure becomes steady and record the process final value.

6. Record these values at specific time intervals by following the digital indicator.

7. Calculate the final steady state deviation.

8. Set the controller to 40% in manual mode and bring it to the original set point. Wait for the system to become steady state, if necessary, adjust the set point to the process value in the manual control mode.

9. Repeat the experiment by changing the PB value to 40%.

10. Repeat the experiments for PB values of 30%, 20%, 15% and 12%.

Calculations

1. Calculate the latest steady state deviation for each experiment as a percentage of the set point change and plot the % deviation against the PB value.

2. Observe the change in the system by changing the pump speed. Degrading effect.

3. Interpret your experience to obtain a stable pressure equal to a new set point in manual control mode.

4. Discuss which PB value is appropriate for the system from the knowledge of how to change steady state value with PB after each change at the set point and suggest a PB value.

5. Discuss the response of the control system to the needle valve position and pump speed.

6. Suggest a mathematical relationship that defines a steady state deviation as a function of proportional control.

7. Discuss what might be the effect on the system of using PB's small values.

2. Proportional Integral Controller (PI)

Overflow integral control

In this section, observe deviations from the new steady-state value by changing the integral time or the reset ratio (IAt) for the PB values you used in the previous Experiments.

Objective: In this experiment, the aim is to show how the integral controller can reduce the steady-state deviation.

Pre-experiment

1. Open the power source. Once you have made sure that the controller has set its own settings, set the inlet and setpoint interval to 500 mb if necessary or the actual pressure in the collecting container.

2. Cut the collecting container needle valve and open the bypass valve.

3. Open the compressed air source and set the inlet pressure to 1.4 bar. Start the pump and set the speed to maximum. Close the bypass valve.

4. Bring the controller to manual mode and open the control valve to open the output about 40%.

5. Open the collecting container needle valve until the pressure inside the vessel is approximately 200 mb and allow the pressure to remain constant.

Experimental procedure

1. When the controller is in manual control mode, set the output to 40% and make the following settings. Record PB = 20, Rate = off, IAt = 4 and SP = PV, SP.

2. Bring the controller to automatic mode.

3. Record this value by increasing the set point by 100 mb.

4. Record the change that took place over time. t-PV

5. Wait until the pressure is stable.

6. Return to the original value of the set point. Wait until the pressure is stable.

7. Repeat the experiment with IAt = 2.

8. Repeat the experiment with IAt = 1 and 0.02.

9. Repeat these experiments for different PB values.

10. Find optimal PB and IAt values

11. Allow the conditions to remain constant and bring the needle valve to its original position by providing a small disruptive effect in the direction that will increase the overflow with the help of the needle valve.

12. Try the same thing at this time to reduce the overflow.

13. Repeat the process by changing the pump speed.

Calculations:

1. Compare integral control with proportional control only.

2. Compare the mode of change of the pressure inside the collecting container when there is proportional and proportional-integral control

3. Which IAt values should be used to reduce the steady-state deviation (Small or large).

4. What are the disadvantages of using very small IAt values. Give your answer by showing some results.

5. Propose PB and IAt values that will give the best response without oscillation according to your results.

6. Discuss how the controller system behaves with detrimental effects.

3. Proportional Integral Derivative Controller (PID)

Objective: Although the PID controller improves the pressure control, to show that it is not always possible to use differential efficiency when the set point changes with a ramp function and to examine the response of the pressure system by changing the set point in the overflow system as the ramp function.

Pre-experiment

1. Open the power switch and make sure that the controller performs its settings, then if necessary, set the input and setpoint range to 500 or the corresponding actual value.

2. Close the collecting container needle valve and open the bypass valve.

3. Open the compressed air source and switch the inlet pressure to 1.4 bar then start the pump and set the speed to maximum. Close the bypass valve.

4. With the controller in manual control mode, open the control valve and set the output value to 25%.

5. Open the collecting container needle valve and ensure that the pressure inside the container is approximately 100 mb.

6. Wait for the pressure to stabilize.

Experimental procedure

1. With the controller in manual mode, set PB (500) and IAt (5) to the most appropriate values determined in previous experiments. Set the derivative term Dat to OFF. Record these values by making SP = PV.

2. Make the setpoint ramp function of the controller effective (RATE = 999) and set the last SPLOC value to initial SPLOC plus 50 mb and record these values.

3. Set the Recorder speed to 600 mm/hr.

4. Bring the controller to automatic mode.

5. Wait a short time to save the starting values, and then start the ramp function by switching to automatic mode. This will make it possible to draw SP values on paper. Record the controller output and SPLOC values at specific time intervals. Wait for the pressure to reach the new set point.

6. Return to the original set point by giving a ramp entry again.

7. Repeat the experiment with dAt = 1 to give a minimum differential control and then increase the dAt values.

8. Remove the paper from recorder and mark the set points.

Calculations

1. When the setpoint changes with the ramp function, how does the expression of the derivative term change the performance of the pressure monitoring system? Will the performance of the pressure monitoring system improve?

2. Can there be disadvantages or advantages of adding derivative term to the pressure controller?

3. What are the limitations of adding derivative control to this system? Why might the process variable create difficulty in the addition of the derivative term to pressure control?

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Experiment ID: 2a-LIQUID-LIQUID EXTRACTION

Objective: Performing mass balance, determination of the mass transfer coefficient (MTC) and effect of the flow rate on MTC for the system where the water as continious phase.

Theory

The separation of one or more components in a liquid mixture in most of the processes of Chemical Engineering is achieved by treating the mixture with an immiscible solvent. The separation is provided as a result of the different preferences of the components for this solvent. In some cases purification of the liquid, in some cases the extraction of the solute, is necessary for subsequent steps of the process. The rate of dissolution of a solute from one solvent to another depends on the interfacial area between the two immiscible liquids, along with many parameters. For this reason, it is advantageous to form these interfaces in the form of droplets or films (like in packed distillation column)

1. Mass Balance

System: Trichlorethylene - Propionic acid -Water

Amount of propionic acid from organic phase (Rafinate) = $V_0 (X_1-X_2)$

Acid amount extracted to the water phase (Extract) = $V_w (Y_1-0)$, $V_0(X_1-X_2) = V_w(Y_1-0)$

Here, V_w :water flow rate (1/sec)

V_0: Trichlorethylene flow rate (1/sec)

X: Concentration of propionic acid (P_A) in the organic phase (kg/l)

Y: Concentration of propionic acid (P_A) in water phase (kg/l)

Subindexes: 1- Column top   2- Column bottom

2. Mass Transfer Coefficient

Mass Transfer Coefficient, MTC (Based on raffinate phase)

$$MTC = \frac{\text{Acid Transfer Rate}}{(\text{Packed volume})(\text{Aver.Driv.Power})}$$

(1)

$$\text{LogAvg.Driving Force} = \frac{\Delta X_1 - \Delta X_2}{\ln\frac{\Delta X_1}{\Delta X_2}} = \frac{(X_0 - 0) - (X_1 - X_1^*)}{\ln\frac{(X_0 - 0)}{(X_1 - X_1^*)}}$$

(2)

Here;

ΔX_1 ; The driving force at the top of the column (X_2-0)

ΔX_2 ; Driving force under the column (X_1- X_1^*)

X_1 * is the concentration of organic that will be in equilibrium with the water phase with a concentration of Y_1. Calculate the equilibrium values using the distribution coefficient (K) data from the literature.

$$K = \frac{Y}{X} = \frac{\text{Distrubuted material concentration in extract phase}}{\text{Distrubuted material concentration in rafinate phase}}$$

(3)

3. Transfer Unit Concept

As a mole fraction "x" and "y";

$$H_{OR} = \left[\frac{R}{K_R a S (1-x)_{LM}} \right]_{average}$$

(4)

$$N_{OR} = \int_{x_2}^{x_1} \frac{(1-x)_{LM}}{(1-x)(x-x^*)} dx = \int_{x_2}^{x_1} \frac{dx}{(x-x^*)} + \frac{1}{2}\ln\frac{(1-x_2)}{(1-x_1)}$$

(5)

$$H_{OE} = \left[\frac{E}{K_E a S (1-y)_{LM}} \right]_{average}$$

(6)

$$N_{OE} = \int_{yx_2}^{y_1} \frac{(1-y)_{LM}}{(1-y)(y^*-y)} dy = \int_{y_2}^{y_1} \frac{dy}{(x-x^*)} + \frac{1}{2} \ln \frac{(1-y_1)}{(1-y_2)}$$

(7)

$$(1-y)_{LM} = \frac{(1-y)-(1-y^*)}{\ln \frac{(1-y)}{(1-y^*)}} \quad \text{and} \quad (1-x)_{LM} = \frac{(1-x^*)-(1-x)}{\ln \frac{(1-x^*)}{(1-x)}}$$

(8)

As mass fractions "x" and "y", $r = \dfrac{MW_{insoluble}}{MW_{soluble}}$

$$N_{OR} = \int_{x_2}^{x_1} \frac{dx}{(x-x^*)} + \frac{1}{2} \ln \frac{(1-x_2)}{(1-x_1)} + \frac{1}{2} \ln \frac{x_2(r-1)+1}{x_1(r-1)+1}$$

(9)

$$N_{OE} = \int_{y_2}^{y_1} \frac{dy}{(y-y^*)} + \frac{1}{2} \ln \frac{(1-y_1)}{(1-y_2)} + \frac{1}{2} \ln \frac{y_1(r-1)+1}{y_2(r-1)+1}$$

(10)

As mass ratios "x" ve "y",

$$N_{OR} = \int_{x_2}^{x_1} \frac{dx'}{(x'-x'^*)} + \frac{1}{2} \ln \frac{rx_2'}{rx_1'} \quad \text{and} \quad N_{OE} = \int_{y_2}^{y_1} \frac{dy'}{(y'-y'^*)} + \frac{1}{2} \ln \frac{ry_1'}{ry_2'}$$

(11)

The equations for diluted systems are: (in molar fractions)

$E(y_2-y_1)=R(x_1-x_2)$

$$Ka = \frac{\text{PA transfer amount}}{(\text{Packed volume})(\text{Avg.driving power})}$$

(12)

$$K_E a = \frac{E(y_2 - y_1)}{(V)\dfrac{\Delta y_1 - \Delta y_2}{\ln \dfrac{\Delta y_1}{\Delta y_2}}}, \quad \text{here,} \quad \Delta y_1 = y_1^* - y_1 \quad \text{ve} \quad \Delta y_2 = y_2^* - y_2 \tag{13}$$

$$H_{OE} = \left[\frac{E}{K_E a S(1-y)_{LM}}\right]_{\text{avarage}} = \left[\frac{E'}{K_E a S \overline{(1-y)_{LM}}}\right] \tag{14}$$

$$\overline{(1-y)_{LM}} = \frac{1}{2}\left[\frac{(1-y_1) - (1-y_1^*)}{\ln \dfrac{(1-y_1)}{(1-y_1^*)}} + \frac{1}{2}\frac{(1-y_2) - (1-y_2^*)}{\ln \dfrac{(1-y_2)}{1-y_2^*)}}\right] \tag{15}$$

$$N_{OE} = \int_{y_2}^{y_1} \frac{(1-y)_{LM}}{(1-y)-(y^*-y)}\,dy \cong \frac{(y_1 - y_2)}{(y^* - y)_{LM}} \tag{16}$$

$$(y^* - y)_{LM} = \frac{(y_1^* - y_1) - (y_2^* - y_2)}{\ln \dfrac{(y_1^* - y_1)}{(y_2^* - y_2)}} \tag{17}$$

$$Z_T = H_{OE}\, N_{OE} \tag{18}$$

$$K_R a = \frac{R(x_1 - x_2)}{(V)\dfrac{(\Delta x_1 - \Delta x_2)}{\ln \dfrac{\Delta x_1}{\Delta x_2}}} \quad \text{here,} \quad \Delta x_1 = x_1 - x_1^* \quad \text{ve} \quad \Delta x_2 = x_2 - x_2^* \tag{19}$$

$$H_{OR} = \left[\frac{R}{K_R a S(1-x)_{LM}}\right]_{\text{average}} \cong \frac{R'}{(K_R a)S\overline{(1-x)_{LM}}} \tag{20}$$

$$\overline{(1-x)}_{LM} = \frac{1}{2}\left[\frac{(1-x_1^*)-(1-x_1)}{\ln\dfrac{(1-x_1^*)}{(1-x_1)}} + \frac{1}{2}\frac{(1-x_2^*)-(1-x_2)}{\ln\dfrac{(1-x_2^*)}{1-x_2)}}\right]$$

(21)

$$N_{OR} = \int_{x_2}^{x_1} \frac{(1-x)_{LM}}{(1-x)-(x-x^*)}dx \cong \frac{(x_1-x_2)}{(x-x^*)_{LM}}$$

(22)

$$(x-x^*)_{LM} = \frac{(x_1-x_1^*)-(x_2-x_2^*)}{\ln\dfrac{(x_1-x_1^*)}{(x_2-x_2^*)}}$$

(23)

$$Z_T = N_{OR}\,H_{OR}$$

(24)

Experimental Procedure

1. Fill the organic phase feed tank with 10 liters of trichlorethylene and then add 100 ml of propionic acid. Mix well to provide a homogenous mixture.
2. Set the level control knob according to the bottom of the column.
3. Fill the water supply tank with 15 liters of water. By running the water supply pump, fill column with a high flow rate.
4. As soon as the water reaches the upper level of the packing material of the column, decrease the flow rate to 0.2 l/min.
5. Set the metering pump to a flow rate of 0.2 1/min.
6. Wait 15-20 minutes for the system to be stable. In the meantime, check the flow rates and observe that they are stable.
7. Take 5 ml samples from Feed, Rafinate and Extract.
8. Titrate 10 mL samples with 0.1 M NaOH.

Calculations

1.

	Spent amount of 0.1 M NaOH (ml)	Propionic acid concentration (kg/l)
Flow rate of water phase		
Flow rate of organic phase		
Feed		
Rafinate		
Extract		
Propionic acid amount from organic phase		
Propionic acid amount extracted to water phase		
Mass transfer coefficient		

2. a) Calculate H_{OE} and N_{OE} based on the extract phase.

b) Calculate H_{OR} and N_{OR} based on the raffinate phase

c) Find the required height of the extraction column using the values you find above (H and N).
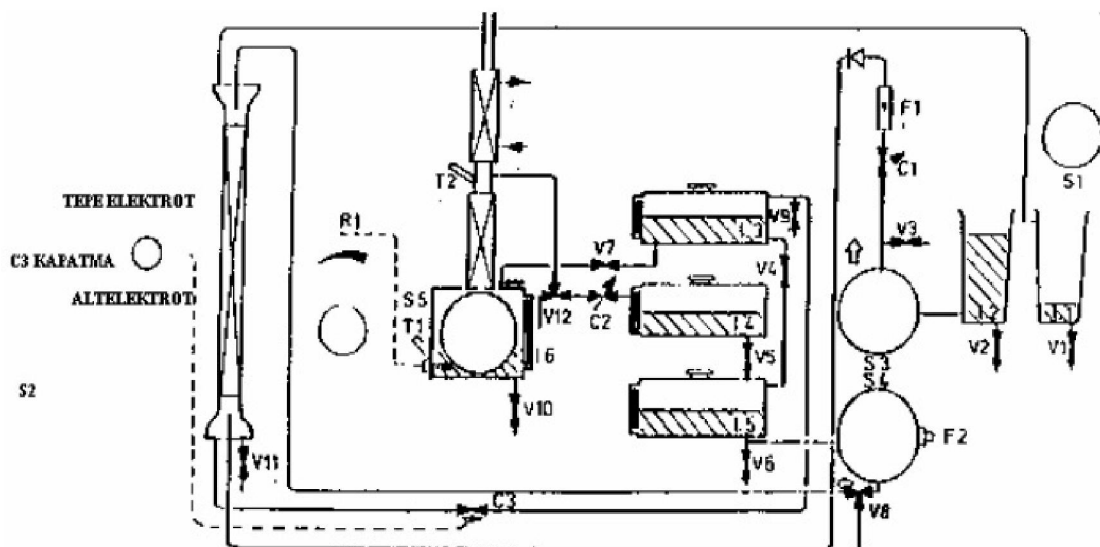

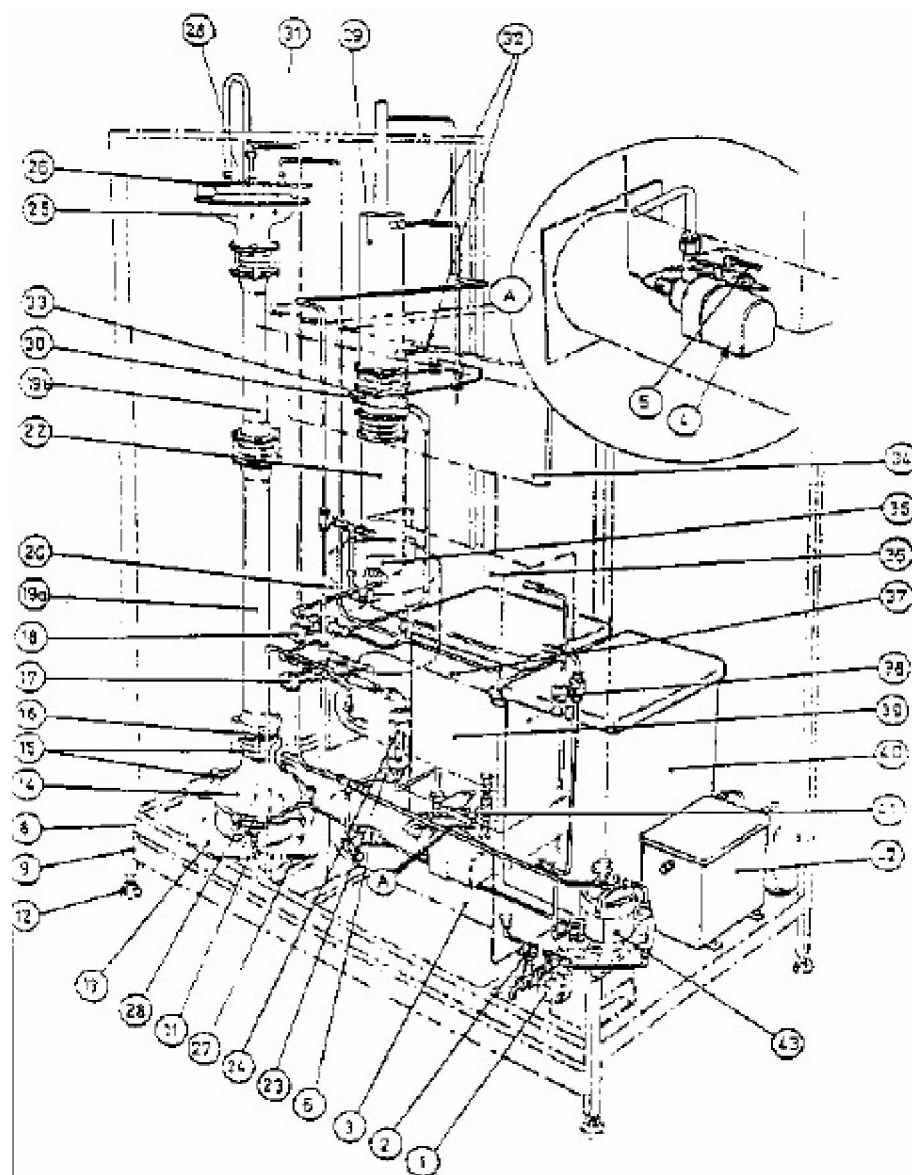Figure 1

Figure 2 Experimental set up:

The most important parts of the system are given below.

1.   : Stroke adjustment button

2. (V6)   : Sampling valve

3. (L5)   : Organic solvent feed tank

4.   : Centrifugal pump

6. (C3)   : Solenoid valve

17 (C2)   : Flow control valve

18. (V7)   : Valve to send the solvent to the distillation column boiler

19a/19b   : Extraction column

20. (LC)   : Distillation column boiler (Boiler)

23 (C1)   : Flow control valve

24. (F1)   Flowmeter

28   : Injector

29.   : Distillation column condenser (Condenser)

30. (T2)   : Thermometer measuring steam temperature

33.   : Glass reflux divider

34   : Control panel

36. (L3)   : Solvent collection tank

37. (L2)   : Water supply tank

38. (V4)   : Valve for continuous or intermittent operation of the system

39. (L4)   : One of three solvent tanks (middle)

40. (L1)   : Water collection tank

43. (F2)   : Measurement pump

(S2)   : Button for actuating the electrodes at below or the top of the column

In the vertical packed column in liquid/liquid extraction unit, first the column is filled with water and then solvent can be fed from above. Alternatively, the column may be filled first with the solvent and then the water to pass through the packing materials may be sent from the bottom of the column. In both cases, the process is continuous and the fluids are pumped to the system. The electrodes at the bottom and top of the column record whether the column is filled with water or solvent, according to the water level. The electrode system affects a solenoid valve that controls the flow of water through the effect of gravity from the column.

The solvent metering pump must be calibrated based on the maximum flow rate. The pump is calibrated as follows. First, F2 is set to 100% and valve V8 is set to the calibration position. and the flow from the pump is measured by a gradual and chronometer. Flow rates at 0% intervals (ml/min) are observed and then a calibration chart (% versus ml/min) is prepared. It is possible to work at any flow rate later by taking advantage of this graph.

The system also has a distillation unit to help recover the solvent.

Gazi University

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|		
Experiment ID	**:**	**3a – GAS ABSORPTION**
Objective	**:**	➢ To show the absorption of carbon dioxide mixed with air to the liquid phase caustic soda solution in a two phase countercurrent packed absorption column.
		➢ Determination of the gas phase overall mass transfer coefficient (K_{OG}).
Experimental Setup	**:**	Armfield UOP7 Gas Absorption Column

Theory

In gas absorption a soluble vapor is absorbed from its mixture with an inert gas by means of a liquid in which the solute gas is more or less soluble. The washing of ammonia from a mixture of ammonia and air by means of liquid water is a typical example. The solute is subsequently recovered from the liquid by distillation, and the absorbing liquid can be either discarded or reused. Sometimes a solute is removed from a liquid by bringing the liquid into contact with an inert gas; such an operation, the reverse of gas absorption, is desorption or gas stripping. A common apparatus used in gas absorption and certain other operations is the packed tower (Figure 1).

Figure 1. Packed tower

If the volumetric flow rates of the liquid phase entering and leaving the column are shown as L_i and L_o respectively, the molar flow rates of the total gas entering and exiting the column are shown as G_i and G_o respectively. Also, the molar fraction of carbon dioxide in the gas mixture entering and leaving the column are shown as y_i and y_o, respectively (Figure 2). Amount of carbon dioxide leaving the gas phase where the air is the inert gas (Equation 1) and carbon dioxide absorbed in the liquid phase (Equation 2) are expressed as below.
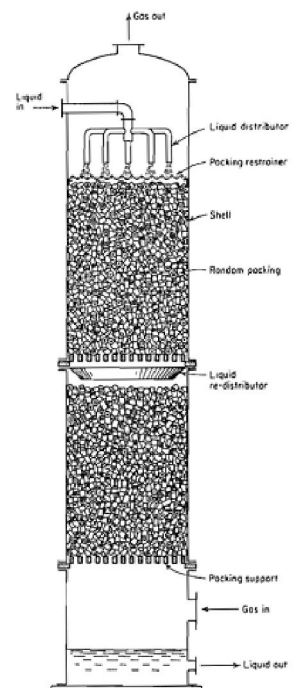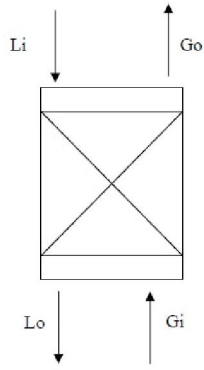
$$G_i - G_0 \text{ (gmol/second)} \tag{1}$$

$$L_o C_{No} - L_i C_{Ni} \text{ (gmol/second)} \tag{2}$$

Figure 2. Schematic diagram of system parameters

In the air-carbon dioxide-caustic solution system, the liquid flow rate does not change along the column ($L_o = L_i$), but gas flow rate changes due to carbon dioxide transport and the pressure drops throughout the column ($G_o \neq G_i$). In order to determine the change in the flow rate of gas phase, the following equation obtained from the material balance can be used.

$$G_o (1 - y_o) = G_i (1 - y_i) \tag{3}$$

The equation given for packed absorption columns (towers) is used to determine the gas phase overall mass transfer coefficient (K_{OG}).

In this equation, y and y* are the mole fraction of the gas that is at equilibrium with liquid at any point of the column and the mole fraction of the bulk phase, respectively. A is the cross-sectional area of column, H is the height of the packed material, a is the surface area of the unit volume of the packed material. For the diluted gas mixture, Equation 4 can be written as;

$$H = \frac{d[Gy]}{K_{OG}aA(y^* - y)} \tag{4}$$

$$K_{OG} = \frac{Gdy}{HaA(y^* - y)} \tag{5}$$

Since it is difficult to integrate the term on the right-hand side of Equation 4, K_{OG} can be determined from the following equation with simpler but less precision;

$$N = K_{OG} aAH \frac{(Pi - Po)}{\ln\left(\frac{Pi}{Po}\right)} \tag{6}$$

Here; N is the absorption rate in gmol/sec. If Equation 6 is rearranged Equation 7 is obtained.

$$K_{OG} = \frac{N \ln\left(\frac{Pi}{Po}\right)}{aAH(Pi - Po)} \tag{7}$$

Pre-experiment Preparation

1. Examine the experimental setup in the lab.
2. Draw a flow diagram of the experimental setup.
3. Create the detailed experimental procedure including the parameters.
4. Calculate the absorption rate in the liquid phase of the gas phase and the overall mass transfer coefficient for the gas phase.
5. Show that the experiment made is repetable and do an error analysis of the experiment.

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Experiment ID: 4a- ROTARY DRYER

Objective:

➤ To investigate mass and heat transfer phenomena in drying process.

➤ To determine the drying characteristics of the material and the efficiency of the dryer.

Theory

The drying of a solid material is generally defined as the removal of a small amount of water or volatile liquids in the solid to reduce the liquid content. In order to dry the solid material, various types of devices such as rotary dryers, exhaust dryers, tunnel dryers, flash dryers are used.

Rotary dryers are used when free flowing granular materials are difficult to dry in conveyors or trays. The material to be dried in the rotary dryer is moved from the feed point towards the outlet region by means of flaps in the dryer chamber while the dryer gas stream is in contact with the solid in the parallel or reverse direction. The drying mechanism generally includes the steps of convective heat transfer from the gas stream to the solid surface, heat transfer by conduction in solid and the moisture transfer from solid to outer environment.

Due to the rotation of the dryer chamber and the movement of the fins, the contact surface of the solid with gas is changed or renewed along the dryer. Due to that, drying rate in rotary dryers is higher than the drying rate in the tray or tunnel dryers. This high drying rate is an advantage if the gas used for drying is held unsaturated. Therefore, the gas should be pre-heated before being supplied to the dryer. In some cases, gas is heated by passing steam through the tubes placed in the dryer to prevent from reaching the saturation temperature.

The movement of the solid in the dryer is controlled by three different mechanisms.The first is that the solid particles are lifted by the movement of the fins and fall again during the rotation of the dryer.In each fall, the particle travels in the dryer as "X = D s" ; where "D" is the dryer diameter, and "s" is the slope of the dryer. If the length of the drier is L, and the speed of rotation is N (rpm), the retention time in the dryer is proportional to L/(D.s.N). The second mechanism is that the particles hitting the dryer wall, roll onto the other particles.Thus, the particles which are not lifted by the fins move forward in the dryer

chamber.This action is called "Kiln Action" and has a significant effect on the movement of solid particles. The third mechanism occurs due to gas motion. If the dryer gas is fed (parallel flow) in the same direction with the feed, it accelerates the movement of the solid particles. Dryer gas slows down the motion of solid particles in driers operating with counter current.

When analyzing mass and heat transfer events in dryers, as the quantities at the inlet and outlet of the dryer do not change; inert/dry gas and dry solid are taken basis to simplfy the calculation. In this case, the moisture concentrations in the gas and the solid should also be defined on the basis of the inert substance.If the moisture content in the solid is expressedas x and the moisture content in the gas as y, the moisture content by inert substance basis is defined as:

$$X = \frac{x}{1-x} \left[\frac{\text{moisture, kg}}{\text{dry solid, kg}} \right] \qquad Y = \frac{y}{1-y} \left[\frac{\text{moisture, kg}}{\text{dry gas, kg}} \right]$$

By using these definitions; for the dryer given its block diagram given in Figure1, the material balances can be written by taking mass flow rate of dry solid (m_S) and dry gas (m_G) are constant;
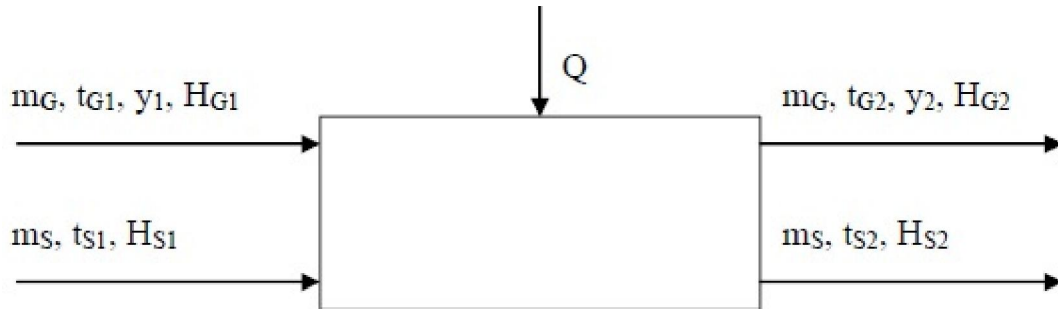

Figure 1. Block Diagram ofRotary Dryer

MassBalance

$$m_S X_1 + m_G Y_1 = m_S X_2 + m_G Y_2 \qquad (1)$$

$$m_S (X_1 - X_2) = m_G (Y_2 - Y_1) \qquad (2)$$

Here;

m_S: Solid flow rate, kg dry solids/s

m_G: Dryer gas flow rate, kg dry gas/s

For writing energy balance for this dryer operating in parallel flow (Figure 1), before giving some definitions should be given.

Enthalpy of Solid including moisture

$$H_S = C_D(t_S - t_R) + XC_0(t_S - t_R) \qquad (3)$$

Enthalpy of Gas including moisture

$$H_G = C_B(t_G - t_R) + Y[C_A(t_G - t_R) + \lambda_0] \qquad (4)$$

If the reference temperature t_R is selected as 0˚C for the mixture of air and water vapor, this equation can be written as;

$$H_G = [1005 + 1864Y]t + 2502300Y \qquad (5)$$

Now the energy balance can be written as;

Energy Balance

$$M_{S_1}H_{S_1} + M_{g1}H_{g1} = M_{S_1}H_{S_2} + M_{g2}H_{g2} + Q \qquad (6)$$

In the above equations;

Ms	: Flow rate of solid with moisture, kg/s
Mg	: Flow rate of gas with moisture, kg/s
C_A	: Heat capacity of steam, J/kg.K
C_B	: Heat capacity of dry steam, J/kg.K
C_D	: Heat capacity of dry solid, J/kg.K
C_0	: The heat capacity of the moisture as in liquid form, J/kg.K
t_S	: Temperature of the solid, °C
t_G	: Temperature of the gas, °C
t_R	: Reference temperature, °C
Q	: Net heat loss in the dryer, J/s
λ	: Evaporation latent heat, J/kg

Experimental Setup

The experimental setup consists of rotary dryer and its accessories. The dryer consists of a feed hopper with a butterfly valve, a gas supply fan, a gas supply heater, a cyclone separator, a dry solid container, a rotary dryer chamber and a motor.

The dashboard located on the front of the device, contains on-off switches of fan, heater and the motor that rotate the dryer chamber. While using these buttons, the order given in the method section should be considered.

The rotary dryer chamber has a 76 threaded wheel. The speed is adjusted according to any of the motor gears connected to the chain of this impeller. If the impeller is connected to the gearwheel of the motor 38, the rotational speed of the dryer chamber is 17.5 rpm, if it is connected to the 17 gear wheel, the rotational speed will be 7.83 rpm.

There are also two screws for providing the required slope on the base where the dryer is mounted. With these screws, the tube can be inclined at an angle of 0 ° to 4 °. Under low feed rate conditions; In the case of arranging the angle of 2° ,the tube gains a height difference equal to 41mm. In the case of an angle of 4˚, the tube gains a height difference equal to 89 mm.

One of the important accessories of the device is the orifice plates that used to determine the air flow rate. For these orifice plates mounted at the entrance of the fan, the air flow rate is given in Table 1 depending on the diameter of the orifice.

Table 1. Approximate air flow rates by orifice diameter

Orifice diameter(mm)	Air flow rate in dryer chamber(m/s)
34.0	4.60
29.5	2.55
23.0	1.53
19.5	1.02
16.5	0.70

Directly above the heater connected to the air inlet fan, there is a thermometer to measure the air inlet temperature before the feed hopper, and a second thermometer (immersion type) to measure the air outlet temperature.

At the exit of the chamber, there are glass-made collection container a, placed on spring-loaded system, and a cyclone separator to hold solid particles drifting by the air flow. At the exit of the chamber, an observation glass is also mounted to check the accumulation in the chamber.

Drying process can be studied as parallel flow and counter-current flow. The experimental system is suitable to work as parallel current but making some modifications on the system it is possible to work as counter flow to compare the dyer performance.

Experimental Procedure

In order to be able to perform the experimental work, discuss the experiment with the instructor at least 3 days prior to the experiment date. Below the interview, please note that 3 written materials have been carried out.

➢ Examine the experimental setup in the laboratory.

➢ Study the flow chart of the experimental setup.

➢ Suggest a detailed experimental method on how to do the experimental work and discuss it with the course instructor.

Calculations

➢ By using the wet and dry thermometer temperatures measured using hygrometer , obtain the absolute humidity of the laboratory air. Describe the working principle of the "Hygrometer" measuring wet and dry thermometer temperatures.

➢ From the data on the inlet and outlet temperatures of the dryer measured versus time, find the total amount of moisture removed from the material by using the air-humidity diagram (physicometric chart).(Adiabatic saturation can be accepted, why?)

➢ Weigh the sample before the dryer (m_1) and after the dryer (m_2). Determine the total amount of moisture removed from the sample at the dryer by weighing the sample obtained from dryer before putting (m_3) it to oven and after taken from the oven (m_4).

➢ Find the moisture content of the sample taken from the wet material by using the weights before putting the oven (m_5) and after drying in the oven (m_6). With this data, calculate the feed rate according to the dry solid basis.

➢ By using the values you have found above, calculate the efficiency of the dryer.

➢ Calculate the amount of heat that must be supplied to the dryer, by determining the average dryer temperature from the inlet and outlet temperatures of the air andby the latent heat of evaporation of the water at this temperature,.

References

➢ Foust, A.S.,Wenzel, L.A., Clump, C.W., Maus, L., Andersen, L.B., Principles of Unit Operations, John Wiley and Sons, 1980.

- McCabe, W.L., Smith, J.C. and Harriott, P., Unit Operations of Chemical Engineering, 6th Ed., McGraw-HillInc., 2001.

- Geankoplis, C.J., Transport ProcessesandSeperationProcessPrinciples, 4th Ed. Prentice Hall, 2003.

- Himmelblau, D.M., Basic Principles and Calculations in Chemical Engineering, 7th Ed., Prentice Hall, 2004.

Experiment Name: RotaryDryer

GroupNumber:

Groupmembers:

Experiment Date:

DATA

The dry air temperature of the laboratory air, T_k:

Wet thermometer temperature of laboratory air, T_w:

Feed flow rate of solid material to be dried:

The weight of the sample taken from the material to be dried, m_1:

Orifice diameter, mm:

Air flow rate, m/s:

Time (t), s	Inlet Air Temperature, ˚C	Outlet Air Temperature, ˚C

Total mass of solid collected in both collection containers during the experiment, m_2:

The mass of the sample taken from out of the dryer before the putting to the ovenm_3:

The mass of the sample taken from out of the dryer after drying in the oven, m_4:

The mass of the sample taken from wet material to be dried, m_5:

The mass of sample dried in the oven, m_6:

Instructor

NOTE:

➢ The data sheet should be filled with ballpoint pen and will be signed separately by the group members.

➢ The data sheet should be signed to the related Instructed after the experiment and the original copy will be added to the experiment report.

➢ The laboratory can be left after the experimental apparatus is cleaned and the laboratory staff is informed to turn off the device.

➢ The due date of your experiment reports is one week after the performed day of the experiment.

Gazi University

Faculty of Engineering

Chemical Engineering Department

ChE 482 Chemical Engineering Laboratory III

Experiment ID **:** **5 – SIZE REDUCTION AND SIEVE ANALYSIS**

Objective **:** To find the experimental and theoretical power of the ball mill. Determination of the size distribution of the samples.

Experimental Setup **:** Armfield Solids Study Handling Bench

Pre-Experimental Preparation

To repeat the basic principles of size reduction and sieve analysis.

Experimental Procedure

- Place 25-30 pieces of different size balls.
- Record the time for one turn of watt-meter without loading to mill.
- Weigh 300 g of coal load the mill. As the milling process continues and read number of turns of the watt -meter.
- After milling is completed, carry out the sieve analysis.

  At the end of the sieving process, each elected amount is determined by weighing.

Calculations

1. Calculate experimental and theoretical mill power.
2. Prepare a table showing the product size distribution differentialy and sieve analysis.
3. Determine particle size of the product, specific surface area and volume-surface average diameter by using differential and cumulative sieve analysis.

References

1. McCabe, W.J., Smith, J.C., Harriot, H., Unit Operations of Chem. Eng., 7th Int. Ed., McGraw Hill Book Co., New York, 2005.
2. Geankoplis, C.J., Transport Processes and Seperation Process Principles, 4th Ed., Prentice Hall, 2003.

Theory

Crushing and milling are processes that allow smaller particles to be separated into smaller pieces. Solid particles can be separated into smaller pieces in essentially four ways; (a) Compression, (b) Impact, (c) Friction, (d) Cutting. All of these are used in different places and the equipment was also designed for purpose.

The basic characteristics of the equipment to be used as a crusher and grinder can be listed as follows:
1. Capacity should be large,
2. The amount of power required for the unit amount of product should be small,
3. Desired size distribution should be obtained.

The main aim of crushers and grinders is to obtain small-grained solids from large grained solids. Small-grained particles may be desirable due to their large surface area or shape, size and number. Here, the energy efficiency of the size reduction process is measured by the amount of new surface that is obtained for the reduction in size. Crushers and grinders cannot produce single level dimension of products. It is only possible to obtain the product at a certain size range.

A. Power Calculation Required for Size Reduction:

Several theories have been announce for the calculation of the power required for size reduction processes. However, none of them sufficiently comply with practical conclusions. The developed theories assume that the energy E required to make a dx change in a particle of "X" dimension is proportional to the exponent of the X dimension.

$$\frac{dE}{dX} = -\frac{C}{X^n} \tag{1}$$

X: Particle size, mm
C, n: Constants (depending on size and type of material, type of machine)

Rittinger suggests that the required power is proportional to the new level created. Since the area is proportional to the length square, n = 2 according to this assumption. When the above expression is integrated, the following equation is obtained;

$$E = K_R \left(\frac{1}{X_2} - \frac{1}{X_1} \right) \tag{2}$$

K_R: Constant, X_2: Average diameter of the product, X_1: Average diameter of the feed

E: The work required to reduce the size from X_1 to X_2.

It was observed that this expression is generally more accurate for small particles.

Kick suggested that the energy required for grinding was directly proportional to the size reduction ratio. In this case $n = 1$ and the integrated expression will be as follows:

$$E = C\ln\left(\frac{X_1}{X_2}\right) = K_K\log\left(\frac{X_1}{X_2}\right) \tag{3}$$

K_K: constant.

Finally Bond assumed that, the required work is proportional to the square root of surface area/volume ratio of the product ($n = 1,5$).

$$E = K_B\frac{1}{\sqrt{X_2}} \tag{4}$$

To use this equation, Bond proposed the Ei work index. E_1 is defined as the energy (kW-h/ton) required to pass through more than %80 of the particles from 100 μm sieve .

In the British unit system, Bond equation turns into the following form:

$$\frac{P}{T} = 1,46\, E_i\left(\frac{1}{\sqrt{D_P}} - \frac{1}{\sqrt{D_F}}\right) \tag{5}$$

P: Power, hp

T: Feeding speed, ton/min, D_F: Feeding diameter, ft, D_P: Product diameter, ft

Typical values for Ei are given in Table 1;

Table 1. E_i values

Material	E_i^*
Bauxite	9,45
Coal	11,37
Granite	14,39

• When the material is dry, these values should be multiplied by 1.34

B. Sieve Analysis:

One of the methods used to determine size distribution in a mixture of particles of various sizes is sieve analysis. In this method, the mixture is passed through serial sieves and divided into parts. The amounts of the substances remaining in each sieve are found by weighing. Using these values, the remaining amount in each sieve is determined as mass fraction. When describing particle size by sieve analysis, the sieve sizes should be given, which is the final sieve to particle passed. For example, the 14/20 term indicates that particle passes through the sieve size 14 and remain on the sieve size 20. This type of analysis is known as "derivative analysis". In Table 2, a sample derivative sieve analysis is given. Here, $\Delta\Phi_n$ is the mass fraction of the solid remaining in the "n" numbered sieve. Sieves are numbered starting from the top. D_{Pn} is the diameter equal to the sieve opening number "n".

Table 2. Differential sieve analysis

"Mesh"	$\Delta\Phi_n$	D_{Pn} cm	"Mesh"	$\Delta\Phi_n$	D_{Pn} cm
4/6	0.0251	0.3327	35/48	0.0102	0.0295
6/8	0.125	0.2362	48/65	0.0077	0.0208
8/10	0.3207	0.1651	65/100	0.0058	0.0147
10/14	0.257	0.1168	100/150	0.0041	0.0104
14/20	0.159	0.0833	150/200	0.0031	0.0074
20/28	0.0538	0.0589	in container	0.0075	
28/35	0.0210	0.0417			

The second method in sieve analysis is cumulative sieve analysis. The cumulative sieve analysis is found by using a derivative sieve analysis. The amount of material remaining in sieve are added on another, beginning the biggest sieve. Φ is defined by the following equation.

$$\emptyset = \Delta\emptyset_1 + \Delta\emptyset_2 + \ldots\ldots = \sum_{n=1}^{n_T} \Delta\emptyset_n \qquad (6)$$

The total sieve analysis gives the relationship between Φ and D_P (hole size of sieve "n"). The amount of Φ defines the amount of solid particles larger diameter than D_P. For the whole sample, Φ value will be equal to one. Table 3 shows the cumulative sieve analysis for the system, which is given a differantial sieve analysis at Table 2.

Table 3. Cumulative sieve analysis

"Mesh"	D_{Pn} cm	Φ	"Mesh"	D_{Pn} cm	Φ
4	0.4699	0.00	35	0.0417	0.9616
6	0.3327	0.0251	48	0.0295	0.9718
8	0.2362	0.1501	65	0.0208	0.9795
10	0.1651	0.4708	100	0.0147	0.9853
14	0.1168	0.7278	150	0.0104	0.9894
20	0.0833	0.8868	200	0.0074	0.9925
28	0.0589	0.9406	in container	-	1.0000

C. Properties of Solid Particles

A solid can be defined by size, shape and density. It will not be possible to define these properties for a smaller size but not homogeneous solid particle mixture obtained by fracture of large solid particles. In this case, shape factors and dimensions are used. However, these terms are defined randomly.For this reason, it is possible to find different definitions for shape and size in the literature.

Particle Shape

The shape factor of a single solid is defined by Φs:

$$\emptyset_s = \frac{6V_P}{D_P S_P} \tag{7}$$

Sp: The surface area of particle

Dp: Equivalent diameter or nominal diameter

Vp: Volume of a particle

The equivalent diameter is defined sometimes as the diameter of the cube in the same volume. However, it is not possible to find the exact volume in very small particulate mixtures. In this case, the D_P is the nominal diameter determined based on the sieve analysis. The surface area is found using the adsorption experiments or pressure drop in the packed column. For cracked solid particles, Φs is between 0.6 and 0.8. However, if the particles are corrode, Φs can be as high as 0.95. In Table 4, the shape factors are given for various materials;

Table 4. Shape factors

	Φ_s
Sphere, cube or short cylinder	1
Sand particle	0.83
Coal dust	0.73
Cracked glass	0.65
Mica fractures	0.28

Particle Size

The diameter definition can be used for equal sized particles. For non-equal-sized systems, the largest two-dimensional system is used for identification.

D. Mixed Particle Size and Dimensional Analysis

In a sample containing D_p diameter particle, the total volume of particles is m/ρ_p (m: the total weight of the samples, ρ_p: the density of the particles). The volume of a single sample is "v_p", the total number of particles in the sample;

$$N = \frac{m}{v_p S_p} \tag{8}$$

Total surface area;

$$A = N S_p = \frac{m S_p}{v_p \rho_p} = \frac{6m}{\Phi_s \rho_p D_p} \tag{9}$$

To apply the Equations (9) and (10) to mixtures of various sizes and densities, the mixture is first divided into parts of approximately constant density and size. N_i and A_i values calculated from equations (9) and (10) for each part. And then sum these values to find the A and N values for the sample.

Specific Surface Area of Mixture

For the sample with particle density ρ_p and shape factor Φs, is known, surface areas of each fraction are found from Equation (10) and sum of these is defined as the specific surface area, A_w.

$$A_W = \frac{6 x_1}{\Phi_s \rho_p \overline{D}_{p1}} + \frac{6 x_2}{\Phi_s \rho_p \overline{D}_{p2}} + \dots\dots\dots = \frac{6}{\rho_p \Phi_p} \sum_{n=1}^{n} \frac{x_i}{\overline{D}_{pi}} \tag{10}$$

X_1: Mass fraction

n: Number of gap

Dp_1: Average particle diameter (arithmetic mean of the smallest and largest diameters)

Average Particle Size

The average particle size of a mixture is defined in different way;

(a) The most commonly used volume surface average diameter is D_s.

$$\overline{D}_s = \frac{6}{\Phi_s A_w \rho_p} \tag{11}$$

$$\overline{D}_s = \frac{1}{\sum_{i=1}^{n} \frac{x_i}{\overline{D}_{pi}}} \tag{12}$$

When the number of particles in each fraction is known, the following equation can be used.

$$\overline{D}_s = \frac{\sum_{i=1}^{n} N_i D_{pi}^3}{\sum_{i=1}^{n} N_i D_{pi}^2} \tag{13}$$

(b) Arithmetic mean diameter $\overline{D}_N$

$$\overline{D}_N = \frac{\sum_{i=1}^{n} N_i \overline{D}_{pi}}{\sum_{i=1}^{n} N_i} = \frac{\sum_{i=1}^{n} N_i \overline{D}_{pi}}{N_T} \tag{14}$$

(c) Mass average diameter $\overline{D}_w$

$$\overline{D}_w = \sum_{i=1}^{n} x_i \overline{D}_{pi} \tag{15}$$

(d) Volume average diameter $\overline{D}_V$

$$\overline{D}_V = \left[\frac{1}{\sum_{i=1}^{n} \left(\frac{x_i}{\overline{D}_{pi}^3} \right)} \right]^{1/3} \tag{16}$$

Total Number of Particles in Mixture

To find the number of particles in the mixture with differential analysis, the number of particles in each mixture is found. And then sum these values to find the N values (total number of particles/unit mass). The particle volume is proportional to D_p.

$$V_p = a D_p^3 \tag{17}$$

The equations similar to those which are given for benefiting from differantial analysis are also given in sources for cumulative analysis.

Gazi University

Engineering Faculty

Chemical Engineering Department

ChE 482 Chemical Engineering Laboratory III

Experiment ID: 1b- TEMPERATURE CONTROL

Objective:

Controlling the temperatures of different points of the system with the feedback control system by applying the control theory to the temperature control system and seeing the effect of the PID control parameters.

Theory:

The purpose of a control system is to set the values of the process variables to the desired set point values. For this purpose, the amount of deviation is measured from the set point. Deviation (d) is the difference between the set point (SP) and the process variable value (PV)

$$d = SP - PV \qquad (1)$$

The signal that the controller sends to the final control element changes with this deviation (c = f (d)). The controller output is sent to the final control element (triac module + heater) and the temperature is controlled by increasing or decreasing the electric current to the heater. The temperature change feature depends on the controller output, the amount of water to be heated, and the transmission delay in the system.

There are three basic elements in a control system other than the process :

1. The measuring sensor and/or transmitter that will send the controlled variable to the controller by measuring.

2. A controller which will evaluate the measurement signal from the measurement sensor and send the output signal to the control element. Feedback controllers can be examined in three parts. The response of these controllers changes as the values of K_c, τ_i, τ_D change.

3. A final control element that will reduce the amount of deviation of the control element to the minimum value with the signal from the controller. In this system, the heater is used as a final control element together with the triac module. The triac module is placed between the heater and the controller, and reduces or increases the electricity

power of the heater according to the signal from controller. When the input to this device is between 4-20mA, the heater power is changed according to the output of the controller. In this way, the deviation between the set point and the process value is reduced to the minimum value according to the type of the controller

Experimental setup

The experiment system is shown in the Figure 1. The water is received from the resorvoir (2) by the centrifugal pump (1) and is returned to the reservoir (2) by passing through a heater (3) located on the line and then by passing through a helical pipe (4) with length 4 m and the cooler (5). There is a bypass valve (6) connected to the pump and manually adjustable to adjust the flow rate. Temperature at three different points; at the heater point (T1), at the heater outlet (T2), and at the point (T3), is measured with the thermocouples. A three-way valve connects the thermocouple output to the temperature transmitter (TT). This device converts the output to 4-20mA and sends to the controller (TIC) and the current of the heater is changed by the triac.
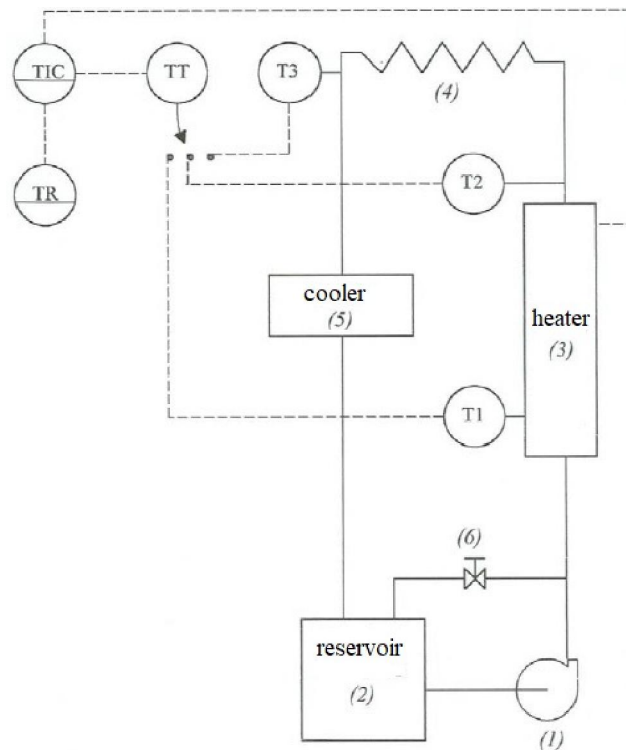
Figure 1. Temperature control system

TEMPERATURE CONTROL SYSYTEM

A. P, PI and PID control of flow temperature

Pre-experimantal Preparation:

1. Turn the thermocouple switch to T2 or T3

2. Set the flow rate to 0.3 L/min with pump or bypass. Check whether the flow is steady.

3. Set the controller to the MANUAL position, adjust the output to be 40%. Operate the heater by pressing the heater button. This temperature should not exceed 80 °C. Otherwise, reset the output value.

4. Enter the values for PROP, IAt and dAt according to your controller type, moving to the controller setting position.

5. To get the data, prepare a table in which you can display time (t), process variable (T1 or T2), setpoint (SP) and controller output (OP).

Experimental Procedure:

Week-I

a) P control

1. Adjust the set point to this temperature (SP = T2), waiting for the T2 (or T1) temperature to stabilize.

2. Change the controller from MANUEL to AUTO.

3. Increase the set point by 5 °C (t = 0). Continue to write T2, OP and SP values at specific time intervals until the flow temperature becomes constant.

4. Once the temperature (T2) becomes constant, change the set point again by 5 °C. Continue writing T2, OP and SP values until the flow temperature is constant at certain time intervals.

5. Repeat steps (1, 2, 3, 4) given above by changing the PROP value.

b) PI control

Repeat the experiment given in "a)" (up to 5.) by setting PROP (same as 5th) and IAt values.

Draw the T2-time and OP-time graphs based on the data you have obtained as a result of your experiments. Compare the control performances according to these graphs.

In the next experiment, how do you change your PI control parameters, why?

Week- II

a) PI control

Repeat the experiment given in week "a)" with the new PROP and IAt values you have decided in the last week of experimentation.

b) PID control

Repeat the experiment in "a)" by entering the value dAt.

B. The effect of a set point ramp to the control of flow temperature

Pre-experimantal Preparation:

1. Set thermocouple switch to T2
2. Set the output (O) to 25% when the controller is in MANUAL and turn on the heater.
3. Set the supervisor settings to PROP = 25% and IAt = 5 and dAt = 1.
4. To get the data, prepare a table showing time (t), process variable (PV), setpoint (SP) and controller output (O) values.

Experimental Procedure:

1. Set the Rate value and the last set value (SPLOC).
2. Change the controller from MANUEL to AUTO. Note the values (t = 0) at specific intervals.
3. When the SPLOC value is reached, the experiment is over.
4. Repeat the experiment by changing the Rate value.

C. The effect of a set point ramp with distance velocity lag to the control of flow temperature

Pre-experimantal Preparation:

1. Set the thermocouple switch to T3.
2. When the controller is in the MANUAL state, set the output (O) value to 25% and turn on the heater.
3. Set the controller settings to PROP = 100% and IAt = 2 and dAt = 1.
4. To get the data, prepare a table in which you can write; time (t), process variable (PV), setpoint (SP) and controller output (O) values during the experiment.

Experimental Procedure:

1. Wait for the temperature value to stabilize.
2. Set the Rate value and the last set value (SPLOC).
3. Move the controller from MANUEL to AUTO state. From this moment (t = 0), Note SP, PV, O with specific intervals.
4. When the SPLOC value is reached, the test is finished.
5. Repeat the experiment by changing the values of PROP and IAt..
6. Repeat the experiment by increasing the Rate value.

Calculations:

Using the data obtained during the experiments, plot the graphs showing the change in PV, O and SP values over time. Find the response parameters (such as overshoot, damping ratio and offset) if the response is oscillatory.

A. PID Control of Flow Temperature

a) Compare and interpret the change in water temperature for different control parameters.

b) By comparing your results, discuss the differential effect and the integral effect.

c) For PROP, IAt and dAt parameters, what can be the optimum values for the case where the temperature gives or does not give offset with the set point?

B. Control of flow temperature by ramp change by PID

a) Interpret how the T3 temperature follows the set point. How do the rate changes at different values given to the set point affect the temperature response?

C. Control of flow temperature with ramp change by PID

a) How does the transmission delay affect the control performance? Discuss by comparing figures.

b) How do you change the parameters to increase stability?

c) Interpret the results by comparing them for different PROP and IAt values.

Gazi University

Faculty of Engineering

Chemical Engineering Department

ChE 482 Chemical Engineering Laboratory III

Experiment ID : 2b – FRACTIONAL DISTILLATION

Objective   :
- Determination of ideal tray number in distillation column
- Calculation of column efficiency
- Calculation of thermal loads

Experimental   :
Setup

Pre-experiment preparation

Review theory of distillation you have studied in ChE302 Mass Transfer I and answer the following questions.

- Which physical properties of a mixture to be separated into its components should be investigated?
- How do you apply the total mass balance and component balance in a distillation column?
- How do you apply the energy balance in a distillation column? How do you calculate the thermal loads of the boiler and the condenser?
- What is Reflux ratio and how is it calculated? How does the change in Reflux ratio affect the composition of the product, the condenser and the boiler thermal loads?
- How do you plot "x-y" and "T-x, y" diagrams?
- How do you find the ideal number of trays? What are tray and overall efficiencies?
- What are the hazardous properties of the chemicals to be used in the experiment and what precautions should be taken when using these chemicals?

It would be useful to take a look the experimental setup in advance. "x-y" and "T-x,y" plots for ethanol-water system should be prepared and submitted to the instructor on time.

Experimental Procedure

- Check the positions of the valves.
- Be sure the ethanol-water mixture with known composition exists in the reboiler.
- Start feeding the condenser water.
- Start heating the mixture in the reboiler.
- Set the reflux ratios (R).

- ➤ Allow the column to reach steady-state conditions by waiting 10 minutes after the first liquid drop from the product condenser arrives.
- ➤ Record all temperature and pressure data.
- ➤ Determine the distillate flow rate using a graduated cylinder below the product condenser.
- ➤ Repeat the above procedure for different R values.
- ➤ Stop heating the reboiler.
- ➤ Continue to pass the cooling water from the condenser for at least half an hour.

The distillation column to be used in the experiment is a glass column with 20 trays, which has only the enrichment zone.

Calculations

- ➤ Calculate the minimum number of trays required for the system studied.
- ➤ Calculate the ideal tray number for each of the reflux ratio conditions studied by using McCabe-Thiele method.
- ➤ Determine overall efficiency if the actual tray number of column is 20.
- ➤ Calculate the thermal loads of the reflux and product condenser.
- ➤ Interpret your findings, taking into account the effect of heat losses from the column to the environment on column performance.

References

1. Geankoplis, C.J., Transport Processes and Seperation Process Principles, 4th Ed., Prentice Hall, 2003.
2. McCabe, W.J., Smith, J.C., Harriot, H., Unit Operations of Chem. Eng., 7th Int. Ed., McGraw Hill Book Co., New York, 2005.
3. Treybal, E.R., Mass Transfer Operations, 3rd Ed., McGraw-Hill, Tokyo, 1980.
4. Uysal, B.Z., Kütle Transferi : Esasları ve Uygulamaları, 2. Baskı, Gazi Üniversitesi Yayınları, Ankara, 2003.

Gazi University

Engineering Faculty

Chemical Engineering Department

ChE 482 Chemical Engineering Laboratory III

Experiment ID: 3b Ion Exchanger

Objective

- To investigate the behavior of the ion exchange column during loading and/or recovery process.
- To examine the properties of the ion exchanger.

Theory

Ion exchangers are insoluble solid substances containing exchangeable cations and anions. When exchangable ions contact with an electrolyte solution, they can be displaced by the stoichiometrically equivalent amount of other ions of the same sign. When ion exchanger that contains sodium (defined as X) as an exchangable cation contact with the solution containing calcium and magnesium ions, the sodium ion will be exchanged by calcium and magnesium ions as follows.

$$2\,Na\,X + Ca^{+2} \longrightarrow Ca\,X_2 + 2\,Na^+$$

$$2\,Na\,X + Mg^{+2} \longrightarrow Mg\,X_2 + 2\,Na^+$$

In the separation processes by means of ion exchange, a packed column is used in which the ion exchanger solid is placed and the continuous flow of solution is achieved. Let's examine a system in which a solution containing a B^+ ion at a concentration of C_0 is passed from a column containing an A^+ cationic ion exchanger. Initially, the entire column is filled with the cation exchanger in form A (Figure 1-a). At time t = 0, the B^+ -containing solution at the C_0 concentration from the top of the column is passed through the column. After a certain period of time, the cation A in the column will leave the site to the cation B. Therefore, the ion exchanger will gradually turn to form B, starting from the top of the column. For some time, a certain top portion of the column will contain an ion exchanger in form B, a certain portion in mixed form A and B, and a certain lower portion in A form (Figure 1-b). As the contact time

increases, the bottom region of the column in the form A will decrease. As a result, all of the column will become saturated with B cation (Figure 1-c). Since then, there will be not ion exchange. If the concentration of the B$^+$ cation at certain intervals in the outlet stream is measured, the concentration versus time curve will be obtained. The scanned area shown in the figure shows the ion exchange capacity of the column in equilibrium. In such systems the concentration-time curves are usually "S" shaped. Parameters affecting the shape of this curve include the ion exchange mechanism, the types of cations, the flow rate, and the concentration of cations in solution.
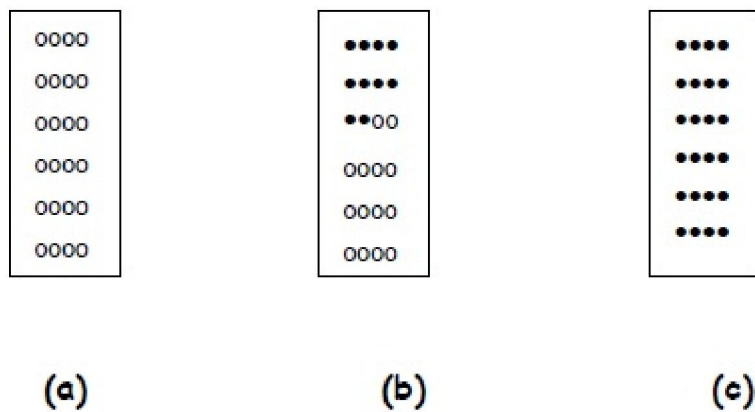
Figure 1. Steps of ion exchange in a packed column
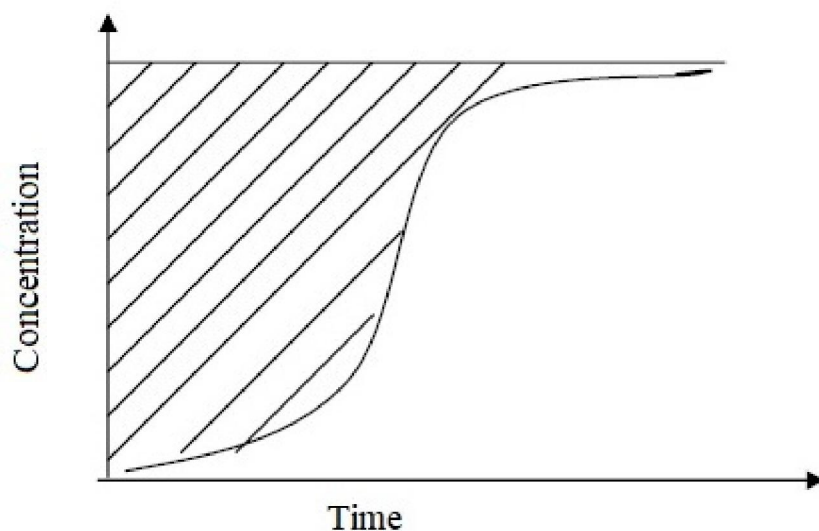
Figure 2. Typical concentration-time curve in a packed column

Experimental Procedure

Ion exchange experiments will be carried out on the ion exchange apparatus prepared by Armfield (UK) company (Figure 3). Before the students begin the experiment, they are obliged to examine and recognize the instrument. Jenway Brand Flame Photometer will be used for chemical analysis of the solutions. In the experimental study, the erlen (at least eight), the mesur (100 and 500 ml), the balloon jojoe (2 L) and the pipette (5 and 10 ml) are required.

Chemicals

Sodium chloride (1N stock solution)

Calcium chloride (1N stock solution)

Magnesium chloride (1N stock solution)

Preliminary study

Students are required to see the responsible lecturer to talk about the preparations for the experiment one week before the experiment date.

Calculations

- Plot the concentration-time curve separately for the exchangable cations.
- Plot again the same graphs as number of bed volumes versus normalized concentration (C/C_0).
- Calculate the theoretical ion exchange capacity of the column.
- Calculate the amount of cations exchanged during the experiment in grams of equivalent grams. Using this value, calculate the percent ion change by taking into account the ion exchange capacity of the ion exchanger in the column and the total amount of ion in the solution.
- Calculate the pressure drop in the column. Accordingly, discuss the suitability of the selected flow rate.
- How are the values calculated above affected by increasing or decreasing the flow rate? Discuss.
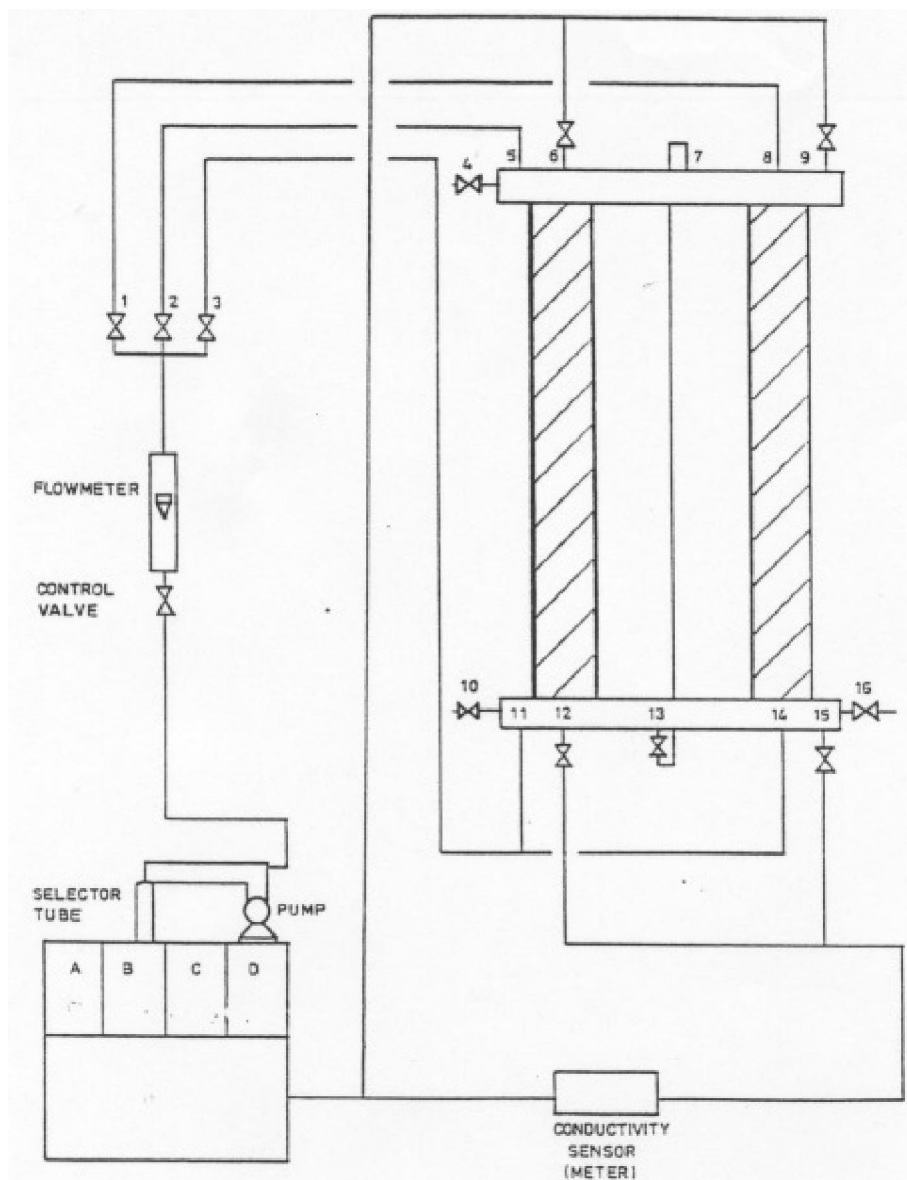
Figure 3. Experimental setup

Preparation Questions

1. How many grams of $CaCl_2$ is needed to prepare a 600 ppm 2 liter $CaCl_2$ solution?

2. In the experiment, the $CaCl_2$ solution was passed through the column earlier. To regenerate the system, 0.1 N NaCl will be passed through the column. Plot the graph of the time against the expected Na-concentration resulting from this experiment, showing the highest and lowest values, semi-quantitatively. How can you determine the degree of ion exchange in the experiment from this graph? Briefly explain.

3. Discuss the effects of flow rate on the system.

4. Write the measurements you need to do in the "Ion Exchange" experiment. Briefly explain how can you evaluate these raw data obtained.

5. Suggest method for determination of hardness in water.

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Experiment ID	**:**	**4b – SPRAY DRYER**
Objective	**:**	➢ To investigate parameters affecting drying performance in a laboratory scaled spray dryer.
		➢ To do the calculations about drying theory.
Experimental Setup	**:**	LabPlant SD-04 Spray Dryer

Pre-experiment Preparation

The following points should be examined.

1. Remember the definitions in the drying theory.
2. Review the system used in the drying process.
3. What are spray dryer types?
4. In what cases are spray dryers preferred?
5. In the experimental procedure section, examine the spray dryer shown schematically and compare with the system in the laboratory (Unit Operations Laboratory).
6. What are the factors that influence the drying performance of the spray dryer? Discuss the effects on product quality.

Students must prepare a 300 ml of 4 wt-% sodium carbonate solution before the experiment.

Experimental Procedure

Schematic representation of the spray dryer in which the experimental work is carried out is given in the Figure 1. The dryer in Unit Operations Laboratory is LabPlant SD-04 model spray dryer which is a nozzle type dryer. The spray dryer consists of a control panel, drying chamber, cyclone and sample collection bottle. The feed is sent to the system with a peristaltic pump and brought into contact with hot air and so is dried.
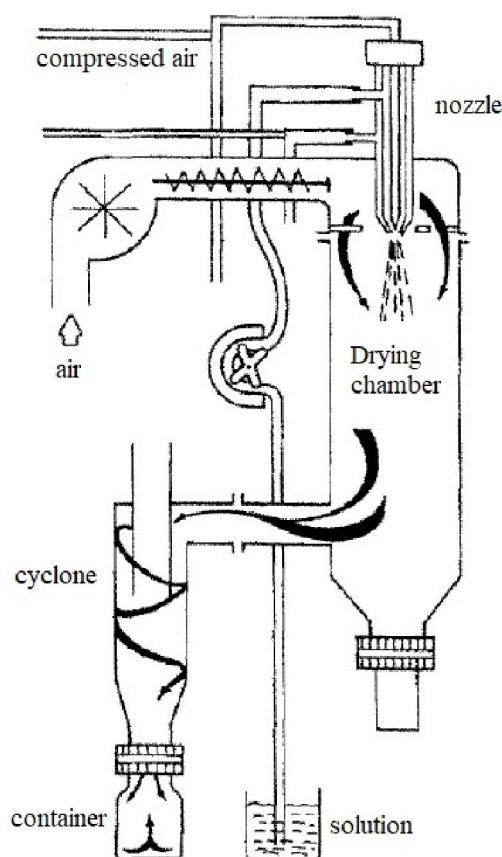
Figure 1. Schematic view of the experimental setup

Operating instruction of the system is given below:

1. Plug in the dryer system and switch on the power.
2. Fill an Erlenmeyer flask with distilled water and immerse the silicone tube of feed in water.
3. Set the required inlet temperature.
4. Switch on the air blower at the set speed.
5. Switch the heater on.
6. Switch on the compressor (do not change the adjusted compressor pressure).
7. Run the pump to feed the pure water after seeing that the outlet temperature of air starts to increase (check the temperature from display on control panel).
8. The speed of the pump should be adjusted for a suitable feed condition (water should not accumulate on the glass surfaces).
9. After making the above adjustments, start to record the output temperature against time.
10. Determine the volume of water that is fed to the dryer per 30 seconds and determine the liquid feed rate.
11. Once the system is at steady state conditions, begin to feed the previously prepared solution 4 wt-% Na_2CO_3 (withdraw the feed silicone tube from the pure water and quickly place it in the solution).

12. Observe that the dried particles enter the sample collection bottle by drawing spirals down the cyclone.

13. Without waiting for the solution in Erlenmeyer flask to be completely finished, replace the solution with pure water solution to ensure that pure water passes through the system and cleans it up again.

14. Switch off the system in the following order:

    > Pump,

    > Compressor,

    > Heater,

    > Air blown when the output temperature drops to 40°C,

    > Main button.

15. At first remove the sample collection bottle and weigh the sample.

16. Slacken the clamps and carefully remove glassware (cyclone and drying chamber) and wash them with tap water.

17. When the glassware pieces are fully dried up, put all the pieces in their places and make sure all pieces are fitted to clamps. Do not fully tighten the s.

Calculations

1. Spread the sample over a watch glass. Investigate if there is any moisture left in the sample after keeping the sample in a dry heat oven for 24 hours at 120 °C.

2. Draw the outlet temperature against time.

3. Set up the material balance for water. Determine the relative humidity value of the outlet air.

4. Determine the efficiency of solid holding of the dryer based on the amount of solids collected.

5. Discuss the effect of system parameters on efficiency using the results you obtained.

References

1. Perry, H. R. and D. Green, "Chemical Engineering's Handbook", 6th Ed., McGraw Hill, N. Y., 1984.

2. Treybal, E.R., Mass Transfer Operations, 3rd Ed. McGraw-Hill, Tokyo, 1980

3. McCabe, W.J., Smith, J.C., Harriot, H., Unit Operations of Chem. Eng., 7th Int. Ed., McGraw Hill Book Co., New York, 2005.

Data for Calculations

1. The value of the air flow rate under normal conditions is 20.3 m3/h, while controller is at the value of 100.

2. Monthly average relative humidity values for Ankara are given in the table below.

Months	Humidity, g water vapor / m^3 air
January	78
February	74
March	65
April	58
May	57
June	50
July	43
August	41
September	47
October	57
November	70
December	78

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Experiment ID	**:**	**5 – SIZE REDUCTION AND SIEVE ANALYSIS**
Objective	**:**	To find the experimental and theoretical power of the ball mill. Determination of the size distribution of the samples.
Experimental Setup	**:**	Armfield Solids Study Handling Bench

Pre-Experimental Preparation

To repeat the basic principles of size reduction and sieve analysis.

Experimental Procedure

- Place 25-30 pieces of different size balls.

- Record the time for one turn of watt-meter without loading to mill.

- Weigh 300 g of coal load the mill. As the milling process continues and read number of turns of the watt -meter.

- After milling is completed, carry out the sieve analysis.

  At the end of the sieving process, each elected amount is determined by weighing.

Calculations

4. Calculate experimental and theoretical mill power.

5. Prepare a table showing the product size distribution differentialy and sieve analysis.

6. Determine particle size of the product, specific surface area and volume-surface average diameter by using differential and cumulative sieve analysis.

References

3. McCabe, W.J., Smith, J.C., Harriot, H., Unit Operations of Chem. Eng., 7th Int. Ed., McGraw Hill Book Co., New York, 2005.

4. Geankoplis, C.J., Transport Processes and Seperation Process Principles, 4th Ed., Prentice Hall, 2003.

Theory

Crushing and milling are processes that allow smaller particles to be separated into smaller pieces. Solid particles can be separated into smaller pieces in essentially four ways; (a) Compression, (b) Impact, (c) Friction, (d) Cutting. All of these are used in different places and the equipment was also designed for purpose.

The basic characteristics of the equipment to be used as a crusher and grinder can be listed as follows:

1. Capacity should be large,
2. The amount of power required for the unit amount of product should be small,
3. Desired size distribution should be obtained.

The main aim of crushers and grinders is to obtain small-grained solids from large grained solids. Small-grained particles may be desirable due to their large surface area or shape, size and number. Here, the energy efficiency of the size reduction process is measured by the amount of new surface that is obtained for the reduction in size. Crushers and grinders cannot produce single level dimension of products. It is only possible to obtain the product at a certain size range.

A. Power Calculation Required for Size Reduction:

Several theories have been announce for the calculation of the power required for size reduction processes. However, none of them sufficiently comply with practical conclusions. The developed theories assume that the energy E required to make a dx change in a particle of "X" dimension is proportional to the exponent of the X dimension.

$$\frac{dE}{dx} = -\frac{C}{x^n} \tag{1}$$

X: Particle size, mm
C, n: Constants (depending on size and type of material, type of machine)

Rittinger suggests that the required power is proportional to the new level created. Since the area is proportional to the length square, n = 2 according to this assumption. When the above expression is integrated, the following equation is obtained;

$$E = K_R \left(\frac{1}{X_2} - \frac{1}{X_1} \right) \tag{2}$$

K_R: Constant, X_2: Average diameter of the product, X_1: Average diameter of the feed

E: The work required to reduce the size from X_1 to X_2.

It was observed that this expression is generally more accurate for small particles.

Kick suggested that the energy required for grinding was directly proportional to the size reduction ratio. In this case n = 1 and the integrated expression will be as follows:

$$E = C \ln\left(\frac{X_1}{X_2}\right) = K_K \log\left(\frac{X_1}{X_2}\right) \qquad (3)$$

K_K: constant.

Finally Bond assumed that, the required work is proportional to the square root of surface area/volume ratio of the product (n = 1,5).

$$E = K_B \frac{1}{\sqrt{X_2}} \qquad (4)$$

To use this equation, Bond proposed the Ei work index. E_1 is defined as the energy (kW-h/ton) required to pass through more than %80 of the particles from 100 μm sieve .

In the British unit system, Bond equation turns into the following form:

$$\frac{P}{T} = 1{,}46\, E_i \left(\frac{1}{\sqrt{D_P}} - \frac{1}{\sqrt{D_F}}\right) \qquad (5)$$

P: Power, hp

T: Feeding speed, ton/min, D_F: Feeding diameter, ft, D_P: Product diameter, ft

Typical values for Ei are given in Table 1;

Table 1. E_i values

Material	$E_i{}^*$
Bauxite	9,45
Coal	11,37
Granite	14,39

• When the material is dry, these values should be multiplied by 1.34

B. Sieve Analysis:

One of the methods used to determine size distribution in a mixture of particles of various sizes is sieve analysis. In this method, the mixture is passed through serial sieves and divided into parts. The amounts of the substances remaining in each sieve are found by weighing. Using these values, the remaining amount in each sieve is determined as mass fraction. When describing particle size by sieve analysis, the sieve sizes should be given, which is the final sieve to particle passed. For example, the 14/20 term indicates that particle passes through the sieve size 14 and remain on the sieve size 20. This type of analysis is known as "derivative analysis". In Table 2, a sample derivative sieve analysis is given. Here, $\Delta\Phi_n$ is the mass fraction of the solid remaining in the "n" numbered sieve. Sieves are numbered starting from the top. D_{Pn} is the diameter equal to the sieve opening number "n".

Table 2. Differential sieve analysis

"Mesh"	$\Delta\Phi_n$	D_{Pn} cm	"Mesh"	$\Delta\Phi_n$	D_{Pn} cm
4/6	0.0251	0.3327	35/48	0.0102	0.0295
6/8	0.125	0.2362	48/65	0.0077	0.0208
8/10	0.3207	0.1651	65/100	0.0058	0.0147
10/14	0.257	0.1168	100/150	0.0041	0.0104
14/20	0.159	0.0833	150/200	0.0031	0.0074
20/28	0.0538	0.0589	in container	0.0075	
28/35	0.0210	0.0417			

The second method in sieve analysis is cumulative sieve analysis. The cumulative sieve analysis is found by using a derivative sieve analysis. The amount of material remaining in sieve are added on another, beginning the biggest sieve. Φ is defined by the following equation.

$$\emptyset = \Delta\emptyset_1 + \Delta\emptyset_2 + \ldots\ldots = \sum_{n=1}^{n_T} \Delta\emptyset_n \tag{6}$$

The total sieve analysis gives the relationship between Φ and D_P (hole size of sieve "n"). The amount of Φ defines the amount of solid particles larger diameter than D_P. For the whole sample, Φ value will be equal to one. Table 3 shows the cumulative sieve analysis for the system, which is given a differantial sieve analysis at Table 2.

Table 3. Cumulative sieve analysis

"Mesh"	D_{Pn} cm	Φ	"Mesh"	D_{Pn} cm	Φ
4	0.4699	0.00	35	0.0417	0.9616
6	0.3327	0.0251	48	0.0295	0.9718
8	0.2362	0.1501	65	0.0208	0.9795
10	0.1651	0.4708	100	0.0147	0.9853
14	0.1168	0.7278	150	0.0104	0.9894
20	0.0833	0.8868	200	0.0074	0.9925
28	0.0589	0.9406	in container	-	1.0000

C. Properties of Solid Particles

A solid can be defined by size, shape and density. It will not be possible to define these properties for a smaller size but not homogeneous solid particle mixture obtained by fracture of large solid particles. In this case, shape factors and dimensions are used. However, these terms are defined randomly.For this reason, it is possible to find different definitions for shape and size in the literature.

Particle Shape

The shape factor of a single solid is defined by Φs:

$$\emptyset_s = \frac{6 V_P}{D_P S_P} \tag{7}$$

Sp: The surface area of particle

Dp: Equivalent diameter or nominal diameter

Vp: Volume of a particle

The equivalent diameter is defined sometimes as the diameter of the cube in the same volume. However, it is not possible to find the exact volume in very small particulate mixtures. In this case, the D_P is the nominal diameter determined based on the sieve analysis. The surface area is found using the adsorption experiments or pressure drop in the packed column. For cracked solid particles, Φs is between 0.6 and 0.8. However, if the particles are corrode, Φs can be as high as 0.95. In Table 4, the shape factors are given for various materials;

Table 4. Shape factors

	Φ_s
Sphere, cube or short cylinder	1
Sand particle	0.83
Coal dust	0.73
Cracked glass	0.65
Mica fractures	0.28

Particle Size

The diameter definition can be used for equal sized particles. For non-equal-sized systems, the largest two-dimensional system is used for identification.

D. Mixed Particle Size and Dimensional Analysis

In a sample containing D_p diameter particle, the total volume of particles is m/ρ_p (m: the total weight of the samples, ρ_p: the density of the particles). The volume of a single sample is "v_p", the total number of particles in the sample;

$$N = \frac{m}{v_p s_p} \tag{8}$$

Total surface area;

$$A = N s_p = \frac{m \, s_p}{v_p \, \rho_p} = \frac{6m}{\Phi_s \rho_p D_F} \tag{9}$$

To apply the Equations (9) and (10) to mixtures of various sizes and densities, the mixture is first divided into parts of approximately constant density and size. N_i and A_i values calculated from equations (9) and (10) for each part. And then sum these values to find the A and N values for the sample.

Specific Surface Area of Mixture

For the sample with particle density ρ_p and shape factor Φs, is known, surface areas of each fraction are found from Equation (10) and sum of these is defined as the specific surface area, A_w.

$$A_W = \frac{6 x_1}{\Phi_s \rho_p \overline{D}_{p1}} + \frac{6 x_2}{\Phi_s \rho_p \overline{D}_{p2}} + \dots\dots\dots\dots = \frac{6}{\rho_p \Phi_p} \sum_{n=1}^{n} \frac{x_i}{\overline{D}_{pi}} \tag{10}$$

X_1: Mass fraction

n: Number of gap

Dp_1: Average particle diameter (arithmetic mean of the smallest and largest diameters)

Average Particle Size

The average particle size of a mixture is defined in different way;

(e) The most commonly used volume surface average diameter is D_s.

$$\overline{D}_s = \frac{6}{\Phi_s A_w \rho_p} \tag{11}$$

$$\overline{D}_s = \frac{1}{\sum_{i=1}^{n} \dfrac{x_i}{\overline{D}_{pi}}} \tag{12}$$

When the number of particles in each fraction is known, the following equation can be used.

$$\overline{D}_s = \frac{\sum_{i=1}^{n} N_i D_{pi}^3}{\sum_{i=1}^{n} N_i D_{pi}^2} \tag{13}$$

(f) Arithmetic mean diameter $\overline{D}_N$

$$\overline{D}_N = \frac{\sum_{i=1}^{n} N_i \overline{D}_{pi}}{\sum_{i=1}^{n} N_i} = \frac{\sum_{i=1}^{n} N_i \overline{D}_{pi}}{N_T} \tag{14}$$

(g) Mass average diameter $\overline{D}_w$

$$\overline{D}_w = \sum_{i=1}^{n} x_i \overline{D}_{pi} \tag{15}$$

(h) Volume average diameter $\overline{D}_V$

$$\overline{D}_V = \left[\frac{1}{\sum_{i=1}^{n} \left(\dfrac{x_i}{\overline{D}_{pi}^3} \right)} \right]^{1/3} \tag{16}$$

Total Number of Particles in Mixture

To find the number of particles in the mixture with differential analysis, the number of particles in each mixture is found. And then sum these values to find the N values (total number of particles/unit mass). The particle volume is proportional to D_p.

$$V_p = a\, D_p^3 \tag{17}$$

The equations similar to those which are given for benefiting from differantial analysis are also given in sources for cumulative analysis.