

**Department of Chemical Engineering** 

# **Unit Operation Lab**

# ChE-422 Manual

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**SEMESTER / YEAR:** 

# EXPERIMENT #01 (UO-01)

#### **MOLECULAR DIFFUSION IN GASES**

#### **OBJECTIVE:**

To determine the diffusion coefficient

#### THEORITICAL BACKGROUND

Molecular diffusion or molecular transport can be defined as the transfer or movement of individual molecules through a fluid by means of the random, individual movements of the molecules.

The diffusivity of the vapor of a volatile liquid in air can be determined by winklemann's method in which liquid is contained in a narrow diameter vertical tube, maintained at a constant temperature. A stream is passed over the top of the tube to ensure that the partial pressure of the vapor is transferred from the surface of the liquid to the air stream by molecular diffusion. With this method, the diffusion is the same as gas A diffusing in stagnant, nondiffusing B.

$$N_A = D\left(\frac{C_A}{L}\right) \left(\frac{C_T}{C_{Bm}}\right) \tag{2.1}$$

The evaporation of the liquid A is expressed as follows

$$N_{A} = \frac{\rho_{L}}{M} \frac{dL}{dt}$$
(2.2)

Equating the above equations, integrating and rearranging the resulting equation, one can obtain as follows:

$$\frac{t}{(L-L_0)} = \left(\frac{\rho_L}{2MD}\right) \left(\frac{C_{Bm}}{C_A C_T}\right) (L-L_0) + \left(\frac{\rho_L C_{Bm}}{MDC_A C_T}\right) L_0$$
(2.3)

The above equation is indeed a linear correlation between  $\frac{t}{(L-L_0)}$  against  $(L-L_0)$  with slope =

$$\left(\frac{\rho_L}{2MD}\right) \left(\frac{C_{Bm}}{C_A C_T}\right)$$

Thus by plotting  $\frac{t}{(L-L_0)}$  versus  $(L-L_0)$ , one can easily calculate the slope and find the diffusivity coefficient of gas A in B.

$$C_T = \frac{1}{22.414} \left( \frac{T_{Abs}}{T_a} \right) \tag{2.4}$$

$$C_{Bm} = \frac{(C_{B1} - C_{B2})}{\ln\left(\frac{C_{B1}}{C_{B2}}\right)}$$
(2.5)

$$C_{B1} = C_T$$

$$C_{B2} = \left(\frac{P_a - P_V}{P_a}\right) C_T$$
(2.6)
(2.7)

$$C_A = \left(\frac{P_V}{P_a}\right) C_T \tag{2.8}$$

#### **DESCRIPTION OF THE EQUIPMENT**



Figure 2.1. Linear Conduction Heat Transfer Unit

The liquid that is to be volatilized is placed in capillary tube. The capillary tube is placed inside a water bath whose temperature is carefully controlled by a controller. The height of the liquid that decreases by time is observed using microscope equipped with Vernier height gauge.

#### **EXPERIMENTAL PROCEDURE**

- 1. Fill the capillary tube with acetone to a depth of approximately 35 mm.
- 2. Insert capillary tube through a rubber ring, inside the metal nut until the top of tube rest on the top of the nut.

- 3. Screw gently the above assembly onto the top plate, with the 'T' piece normal to the microscope
- 4. Connect flexible air tube to one end of the 'T' piece.
- 5. Set up the microscope as shown in figure 4.1. and adjust the object lens to within 20-30 mm from the tank.
- 6. Adjust the vertical height of the microscope until the capillary tube is visible (if the capillary tube is still not visible adjust the distance from the object lens to the tank until it is visible).
- 7. Adjust viewing microscope until the meniscus inside the capillary tube clear and well defined (note that the image in the microscope is upside down. That means the top of the image is the bottom of the capillary tube).
- 8. Make sure that the sliding Vernier scale should be aligned with a suitable graduation on the fixed scale.
- 9. Switch on the air pump.
- 10. Adjust the air flow rate using the Hoffman clip on the flexible tube so the flow rate is low.
- 11. Record the level inside the capillary tube.
- 12. Switch on the temperature controlled water bath and adjust set point on controller to 40  $^{\circ}$ C to obtain steady temperature at 40  $^{\circ}$ C.
- 13. Switch off the temperature controlled water bath after approximately 60 minutes to prevent air bubbles.
- 14. Record the change in level inside the capillary tube.
- 15. Repeat #12 #14.

#### REFERENCES

Geankoplis, C.J., "Separation Processes and Unit Operations, 4<sup>th</sup> edition, Prentice-Hall Inc., New Jersey, 2004

"CERa Experiment Instruction", armfield, 2011.

#### NOTATION

CA	:	Saturation concentration of acetone at interface, kmol/m <sup>3</sup>
C <sub>Bm</sub>	:	Logarithmic mean molecular concentration of vapor, kmol/m <sup>3</sup>
CT	:	Total concentration, kmol/m <sup>3</sup>
D	:	Diffusivity coefficient, m <sup>2</sup> /s
L	:	Effective distance of mass transfer (m)
Μ	:	Molecular weight of acetone, kg/mol
NA	:	Molar flux, mol/m <sup>2</sup> .sec
Pa	:	Atmospheric pressure, atm
Pv	:	Vapor pressure of acetone, atm

- $\rho_L$  : Density of acetone, kg/m<sup>3</sup>
- T : Time, sec
- T<sub>a</sub> : Bath temperature, K
- T<sub>abs</sub> : Absolute temperature, K

#### WORK SHEET

Time, ks	Liquid level (L-L <sub>0</sub> )	$\frac{t}{(L-L_0)}$

Do the following:

1. Plot between 
$$\frac{t}{(L-L_0)}$$
 and  $(L-L_0)$ 

2. Calculate diffusivity coefficient from the slope of the line.

3. Compare your findings with <u>theoretical value</u>. (refer to mass transfer course)

# EXPERIMENT #02 (UO-02)

### **CONVECTION DRYING**

#### **OBJECTIVE:**

- 1. To determine the reduction in weight and the drying rate over time during the drying process.
- 2. To investigate the effect of air flow rate in convection drying.

#### THEORITICAL BACKGROUND

Drying is a physical process of removing the liquid adhering to a material and contained within it. In drying process, the water is usually removed as a vapor by air. It proceeds more slowly for the moisture that is located in capillaries and pores within the material to be dried. This is because the capillary forces or diffusion resistance must be overcome. The removal of water of crystallization contained in the material to be dried is even more difficult because it is bounded by relatively strong molecular forces. The material must be intensely heated to expel the water.

According to the type of heat transfer, drying process can also be classified as follows:

- 1. Convection drying when the heat for the drying is transferred to the material to be dried by a flowing gas.
- 2. Contact drying when the heat is transferred from the heater to the material by conduction due to physical contact between them.
- 3. Radiation drying when the heat is transferred by radiation. The source of energy can be in form of electromagnetic radiation, infrared, microwave or even radio frequency.

In many cases, drying is carried out using warm air at atmospheric pressure. This drying process is called air drying. If drying is done at significant sub atmospheric pressure, it is called vacuum drying. In some cases, the moisture in the material is cooled and frozen and using convection, conduction or radiation heat transfer, and the freezing moisture can be sublimed. This method of drying is known as freezing drying.

One of the most important analyses in drying is rate of drying curves. The equilibrium moisture contents of various materials cannot be predicted and must be determined experimentally. Similarly, since the basic mechanism of rates of drying is quite incomplete thus it is necessary in most cases to obtain some experimental measurements of drying rates.

#### **DESCRIPTION OF THE EQUIPMENT**



#### **EXPERIMENTAL PROCEDURE**

- 1. Remove drying plates from support frame.
- 2. Tare scale to zero
- 3. Insert drying plates individually into support frame, read off and note the weight of the individual drying plates on the digital scale.
- 4. Switch on fan and set speed using flow rate gauge e.g. 1.3 m/s.
- 5. Read off volumetric air flow from display and make a note of it.
- 6. Switch on heater and set the temperature controller e.g. 50°C.
- 7. Run air conditioning duct until it is warmed up evenly all along its length.
- 8. Spread material (gravel with grain size of 0.5-0.7 mm) to be dried in a thin layer on the drying plates.
- 9. Insert drying plates in the support frame and note the total weight.
- 10. Tare scale to zero.

- 11. Introduce water to the spread material e.g. 50 g
- 12. Start drying experiment, commence time measurement using stopwatch.
- 13. Record air temperature T<sub>2</sub> after material to be dried, relative humidity after material being dried, total mass m of material to be dried (material and drying plates).
- 14. Stop the experiment when the mass m of material to be dried does not change significantly.
- 15. Repeat the experiment for air speed of 2 m/s.

### REFERENCES

Transport Processes and Separation Process Principles, 4<sup>th</sup> ed., 2003, C.J. Geankoplis, Pearson Education, Inc.

CE 130 GUNT Experiment Manual.

# WORK SHEETS

### Experiment #1

Mass of drying plate 1 : Mass of drying plate 2 : Mass of drying plate 3 : Dry Gravel mass : Introduced water : Total Mass Air speed Heating level :

:

:

Time	T <sub>1</sub>	φ1	T <sub>2</sub>	φ2	WT
min	°C	% R.H.	°C	% R.H.	g
	Before	e plate	After	plate	
				Ē	

Expe	riment	: #2
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Mass of drying plate 1 Mass of drying plate 2 Mass of drying plate 3 Dry Gravel mass Introduced water Total Mass Air speed Heating level		: : : : :				
	Time	$T_1$	φ1	T <sub>2</sub>	φ2	WT
	min	°C	% R.H.	°C	% R.H.	g
		Before	plate	After	plate	

- 1. Plot the moisture content versus time for both experiments, comment on it in the discussion part.
- 2. Plot the drying rate versus time for both experiments, determine which part is constant drying rate period and which part is falling rate period. comment on it in the discussion part.
- 3. Find the graphical value of constant drying rate.
- 4. predict value of constant drying rate.
- 5. Compare the value from #3 and #4. Comment on the result.

# EXPERIMENT #03 (UO-03)

# WET COOLING TOWER

#### **OBJECTIVE:**

To investigate and understand the influence of volumetric air flow rate, volumetric water flow rate and packing density on the performance parameters of the cooling tower.

#### THEORITICAL BACKGROUND

Water or wet cooling tower is one of the most important types of process in the process industries. This type of cooling tower follows the principle of evaporative cooling. There are many cases in industry in which warm water is discharged from heat exchangers and condensers when it would be more economical to cool and reuse it than to discard it.

In a typical water-cooling tower, warm water flows counter currently to an air stream. Typically, the warm water enters the top of a packed tower and cascades down through the packing, leaving at the bottom. Air enters at the bottom of the tower and flows upward through the descending water. The tower packing often consists of slats of plastic or packed bed. The water is distributed by troughs and overflows to cascade over slat gratings or packing that provides large interfacial areas of contact between the water and air in the form of droplets and films of water.

In this cooling tower, water evaporates into air because its vapor pressure in the air is less than the saturation vapor pressure. The amount of heat that is needed to evaporate the liquid is drawn from the air. Furthermore, this air cools water in contact with it. In this case, the wetbulb temperature which takes both temperature and humidity into account, as compared to the actual air temperature (dry-bulb temperature), is a measure of the potential for evaporative cooling. The greater the difference between the two temperatures, the greater the evaporative cooling effect. Therefore, wet-bulb temperature is essentially the lowest temperature which can be attained by evaporative cooling at a given temperature and humidity.

Several characteristic variables are calculated as follows

**Cooling range** which is the difference between the warm water temperature at the inlet to the cooling tower and the cold water temperature at the outlet from the cooling tower

$$z = T_4 - T_5$$
(1.1)

**Wet bulb approach** which is the difference between the cold water temperature at the cooling tower outlet and the cooling limit or wet bulb temperature.

$$a = T_5 - T_{f1} \tag{1.2}$$

**Cooling coefficient** is formulated as follows

$$\eta = \frac{T_4 - T_5}{T_4 - T_{f_1}} \tag{1.3}$$

Water loss due open circuit in cooling tower is calculated as follows

$$\dot{m}_W = (X_2 - X_1)\dot{m}_L \tag{1.4}$$

**Heat load** which is the heat which is taken from the water during the cooling process is formulated

$$\dot{Q}_W = \dot{m}_W C_{pW} z \tag{1.5}$$

Actual air mass flow rate is calculated as follows:

$$\dot{m}_L = \alpha \varepsilon c \sqrt{\frac{(P_1 - P_2)}{v_2}} \tag{1.6}$$

 $\alpha$  = 0.605;  $\epsilon$  = 0.98, c = 0.0073 and the volumetric air flow is calculated

$$\dot{V}_L = \dot{m}_L v_2 \tag{1.7}$$

Cooling capacity which is the difference in enthalpy between the air inlet and outlet

$$\dot{Q}_L = (h_2 - h_1)\dot{m}_L \tag{1.8}$$

#### **DESCRIPTION OF THE EQUIPMENT**



Figure 1.1 Wet Cooling Tower

The main wet cooling tower unit can be seen in figure 1. The water is heated using heating element in a tank (7), pumped (6) and then sprayed from the top of the column through nozzle (1) and intimately contact with cooling air due to the packing (2). The air comes in from the air chamber (4) at the bottom of the column. The air flow rate is adjustable using fan (5). Due to

evaporation during the course of the process, the make-up water (8) is required to maintain enough water for the process. All parameter readings including water and air flow rate as well as temperature can be collected manually from the display and control buttons (3). The column is also equipped with temperature/humidity sensor (9) at the top and the bottom.

#### EXPERIMENTAL PROCEDURE

Prior to experiment, it is important to do the following steps:

**Experiment #1:** investigation on the effect of water flow rate.

- 1. Fill the water tank and the supply tank (make-up water tank)
- 2. Choose the packing type 1 and assemble it.
- 3. Connect all sensors (combined temperature/humidity and temperature sensors)
- 4. Connect the system to the mains electricity supply.
- 5. Turn on the heater and select the heating level 2 i.e. 1 kW and heat the water in the tank up to 40  $^{\circ}$ C.
- 6. Turn on the fan and adjust the air flow rate so the pressure difference is approximately 120 Pa by changing the shutter opening.
- 7. Turn on the pump and adjust the volumetric water flow rate at 60 L/h.
- 8. After reaching steady state (app. 20 mins), take all measurement readings from the display and fill out worksheet.
- 9. Repeat the same procedure for water flow rate of 75 L/h and 90 L/h.

**Experiment #2:** investigation on the effect of air flow rate.

- 1. Reduce the speed down to 60 L/h.
- 2. After reaching steady state (app. 10 mins), take all measurement readings.
- 3. Repeat the same procedure for  $\Delta p$  of 140 pa and 160 Pa.

**Experiment #3:** investigation on the effect of packing density.

- 1. Turn off the heater, fan and pump. Disconnect all sensors and replace the packing type with type #4.
- 2. Connect all sensors (combined temperature/humidity and temperature sensors)
- 3. Connect the system to the mains electricity supply.
- 4. Turn on the heater and select the heating level 2 i.e. 1 kW and heat the water in the tank up to 40 °C.
- 5. Turn on the fan and adjust the air flow rate so the pressure difference is approximately 120 Pa by changing the shutter opening.
- 6. Turn on the pump and adjust the volumetric water flow rate at 60 L/h.
- 7. After reaching steady state (app. 20 mins), take all measurement readings from the display and fill out worksheet.

# REFERENCES

Geankoplis, C.J., "Separation Processes and Unit Operations, 4<sup>th</sup> edition, Prentice-Hall Inc., New Jersey, 2004

Jebavy, F., "WL 320 Experiment Instruction", GUNT, 2007.

# NOTATION

а	:	Wet bulb approach, K
С	:	Constant, m <sup>2</sup>
C <sub>pW</sub>	:	Specific heat capacity of water, kJ/kg.K
$h_1$	:	Enthalphy of outlet air, kJ/kg
h <sub>2</sub>	:	Enthalphy of inlet air, kJ/kg
mL	:	Air mass flow rate, kg/s
m <sub>w</sub>	:	Water mass flow rate, kg/s
<b>P</b> <sub>1</sub>	:	Inlet pressure, Pa
$P_2$	:	Outlet pressure, Pa
Qw	:	Heat load, W
T <sub>4</sub>	:	Inlet water temperature, K
<b>T</b> 5	:	Outlet water temperature, K
T <sub>f1</sub>	:	Wet bulb temperature, K
$V_{L}$	:	Air volumetric flow rate, l/s
X1	:	Absolute humidity at outlet air, g/kg
X <sub>2</sub>	:	Absolute humidity at inlet air, g/kg
Z	:	Cooling range, K
α	:	Flow Coefficient
3	:	Expansion Coefficient
η	:	Cooling Coefficient
$v_2$	:	Kinematic Viscocity, m <sup>3</sup> /kg

# WORK SHEETS

# Experiment #1

Parameter	Run 1	Run 2	Run 3
$A_{B}, m^{2}/m^{3}$	110	110	110
T <sub>1</sub> , °C			
φ1, %			
T <sub>2</sub> , °C			
φ2, %			
T <sub>4</sub> , °C			
T <sub>5,</sub> °C			
$\Delta$ P, Pa	120	120	120
F <sub>w</sub> , l/min	60	75	90

# Experiment #2

Parameter	Run 1	Run 2
$A_{B}, m^{2}/m^{3}$	110	110
Τ <sub>1</sub> , °C		
φ1, %		
T <sub>2</sub> , °C		
φ2, %		
T <sub>4,</sub> °C		
T₅, °C		
$\Delta$ P, Pa	140	160
F <sub>w</sub> , l/min	60	60

# Experiment #3

Parameter	Run 1
A <sub>B</sub> , m²/m³	200
Т <sub>1</sub> , °С	
φ1, %	
T <sub>2</sub> , °C	
φ2, %	
T <sub>4,</sub> °C	
T₅, °C	
$\Delta P$ , Pa	120
F <sub>w</sub> , l/min	60

# .EXPERIMENT #04 (UO-04)

# **FLUIDIZED BED**

#### **OBJECTIVE:**

- 1. To investigate the loosening speed of a fluidized bed in air and water medium.
- 2. To determine the relationship between flow rate and the depth of the fluidized bed.
- 3. To investigate the effect of particle size on fluidization.

#### THEORITICAL BACKGROUND

Fluidized beds are widely used in process technology. One of important applications in chemical technology is fluidized bed reactor in which variety of multiphase chemical reactions is carried out. In a fluidized bed, a layer of fine granular solid matter is loosened by a fluid flowing through it to such extent that the particles of solid matter are free to move within certain limits. The layer of solid material takes on similar properties to a fluid.

To characterize a fluidized bed, the pressure loss of the fluid flowing through the bed can be used. When a fluid flows through the mass initially the pressure underneath the mass increases as the flow speed increases until the pressure forces match the weight of the mass, and the mass becomes suspended. With further increasing flow rate, the layer set in motion and reaches a fluidized state. At this condition the static solid-like state changes to dynamic-like state. The pressure loss now remains almost constant even with further increasing flow rate. From a certain flow rate the particles at the top no longer fall back into the fluidized bed, they are drawn off by the fluid flow and removed.

Pressure losses in fluidized bed can be calculated as follows:

$$\Delta p = g \left( 1 - \frac{\rho_f}{\rho_p} \right) h \rho_{ps} \tag{3.1}$$

Meanwhile loosening speed at which the mass of solid matter passes the transition to a fluidized bed is expressed

$$w_{lo} = \frac{Re_{lo}}{d_p} v_f \tag{3.2}$$

For irregular shape, loosening speed must be corrected,

$$w = w_{lo}\varphi \tag{3.3}$$

The void fraction of a bed is expressed

$$\varepsilon = 1 - \frac{\rho_{ps}}{\rho_p} \tag{3.4}$$

Reynolds' number is formulated as follows

$$Re_{lo} = 42.86(1-\varepsilon) \left( \sqrt{1+3.11 \times 10^{-4} A_r \frac{\varepsilon^3}{(1-\varepsilon)^2} - 1} \right)$$
(3.5)

With Archimedes' number is defined as follows

$$A_r = \frac{g d_p^3}{\mu^2} \frac{\rho_p - \rho_f}{\rho_f}$$
(3.6)



1. water overflow 2. test tank for water 3. 2-tube manometer 4. flow meter for water 5. U-tube manometer 6. flow meter for air 7. test tank for air 8. filter

#### **EXPERIMENTAL PROCEDURE**

Filling, emptying, and cleaning the single tube manometer, 2 tube manometer, and vessel with particles, including the calibration and testing of vessel for both water and air must be done by the trained person.

#### A. Calibration with empty test tank for water and air

- 1. Connect the pressure measuring connections to the manometer. On a two tube manometer, the display value can be set to the center of the manometer.
- 2. Fully open the by pass valve below the rotameter
- 3. Fully close the needle valve on the rotameter.
- 4. Turn on the pump

- 5. Increase the flow in small increments by opening the needle valve
- 6. Continuously note the flow rate and differential pressure in the table.
- 7. Continue the measurements up to the maximum flow.

#### B. Experiment with test tank for water

- 1. Record the initial bed height (h=100 mm) and ensure particle with  $d_{\rm p}$  of 0.505 mm is used.
- 2. Fully open the bypass valve below the rotameter.
- 3. Fully close the needle valve on the rotameter.
- 4. Turn on the pump.
- 5. Increase the flow in small increments by opening the needle valve.
- 6. Observe the particle movement, note the flow rate, bed depth, and pressure drop.
- 7. Continue increase the flow rate until flow rate of 1.5 L/min is obtained (above certain flow rate, the needle valve will be already fully open, further increase of flow rate must be done by closing the bypass valve).

#### C. Experiment with test tank for air

- 1. Fill the test vessel with particle i.e.  $d_p = 0.240$  mm, until the height of the bed is 50 mm.
- 2. Fully open the by pass valve below the rotameter.
- 3. Fully close the needle valve on the rotameter.
- 4. Turn on the compressor.
- 5. Increase the flow in small increments by opening the needle valve and observe the mass.
- 6. Continuously take note of the flow rate and differential pressure.
- 7. Take the note as soon as the first signs of the particle movements.
- 8. Continue until the a flow 30 L/min is achieved (above certain flow rate the needle valve will be already fully open, further increase of flow rate must be done by closing the bypass valve)
- 9. Repeat the same procedure for different particle size, i.e.  $d_p=0.505$  mm.

#### REFERENCES

"CE220 Experiment Instruction", GUNT, 2011.

#### NOTATION

$\Delta P$	:	Pressure drop, mm
G	:	Gravitational acceleration, m/s <sup>2</sup>
ρ <sub>f</sub>	:	Fluid density, kg/m <sup>3</sup>
ρ <sub>p</sub>	:	Particle density, kg/m <sup>3</sup>
H	:	Height of mass
$\rho_{ps}$	:	Particle mass density, kg/m <sup>3</sup>
Wlo	:	Fluid velocity between spherical particles, m/s
Relo	:	Reynolds' number
d <sub>p</sub>	:	Particle diameter, m

- v<sub>f</sub> : Kinematic viscocity
- W : Corrected fluid velocity, m/s
- $\phi$  : Form factor
- ε : Void fraction
- Ar : Archimedes' number
- $\eta$  : viscocity

# WORK SHEET

A. Calibration with empty test vessel for water and air.

	For water	
Q, L/min	$\Delta$ P,mm	W <i>,</i> m/s

For Air				
Q, L/min	∆P,mm	W <i>,</i> m/s		

B. Experiment with test vessel for water.

# Dp = mm

Q, L/min	∆P,mm	H <i>,</i> mm	W <i>,</i> m/s

C. Experiment with test vessel for air.

D<sub>p</sub> = mm

Q, L/min	$\Delta$ P,mm	W, m/s

D<sub>p</sub> = mm

$\Delta$ P,mm	W <i>,</i> m/s
	ΔP,mm

Do the following:

- 1. Plot pressure drop against fluid speed for all data.
- 2. Plot bed depth against flow rate for test vessel with water only.
- 3. Determine the loosening velocity  $(W_{1o})$  for experiment B and C from the plot.
- 4. Determine the loosening velocity ( $W_{lo}$ ) using equation and compare with that obtained from the plot.
- 5. Comment on the all above plots.

# EXPERIMENT #06 (UO-06)

# SOLID LIQUID EXTRACTION

#### **OBJECTIVE:**

- 1. To study the performance of discontinuous solid-liquid extraction process.
- 2. To study the influence of number of stages on counter-flow solid-liquid extraction process.
- 3. To study the effect of solvent temperature on the extraction performance.

#### THEORITICAL BACKGROUND

Many biological, inorganic, and organic substances occur in a mixture of different components in solid. Solid-liquid extraction is general term used for a process to separate either desired or undesired solute from the solid phase using a solvent/liquid. It is sometimes called *leaching* and if it is intended to remove undesirable component from solid using water, it is called *washing*.

In general the process of solid-liquid extraction can be described as follows; the solvent must be transferred from the bulk solvent solution to the surface of the solid. The solvent then must penetrate or diffuse into the solid and the solute dissolves into the solvent. The solute then diffuses through the solid solvent mixture to the surface of the particle. Finally, the solute is transferred to the bulk solution.

There are several factors influencing extraction performance for example:

- 1. Differences in concentration; the greater the difference in concentration of the usable substance in the extraction material and in the solvent, the greater the driving force when extracting.
- 2. Surface area; it is proportional to the extracted quantity of material.
- 3. Diffusion resistance; it depends on the size of the particles, the porosity and the penetrability of the extraction material for the solvent. The diffusion resistance should be as low as possible.
- 4. Temperature; The higher the temperature, the better the performance of the extraction.

Extraction can be done at different pattern as follows

- 1. Cross flow; the extraction material is sprayed with fresh solvent at each stage.
- 2. Uni-flow; the extraction material is mixed with solvent and move continuously from stage to stage in uni-flow pattern.
- 3. Counter flow; extraction material and solvent move continuously in a counter flow.

The counter flow method generally represents the optimum option. Compared to the uniflow method, a considerably higher proportion of usable substance is transferred from the extraction material into solvent. The concentration gradient as the driving force for the mass transfer is utilized more effectively with the counter flow method. Cross flow method results in even higher yields but requires a much more fresh solvent.

#### **DESCRIPTION OF THE EQUIPMENT**



Figure 5.1. Solid-Liquid Extraction Unit

Solid-liquid extraction unit as shown in the diagram (1) has a spiral conveyor (2) to feed the extraction material into revolving extractor in which the extraction process occurs. This spiral conveyor can feed the solid up to 20 l/h with power consumption up to 4 W. Revolving extractor is driven by a motor (4) with speed up to 9  $h^{-1}$  and power consumption up to 0.9 W. The solvent is then fed using several peristaltic pumps (5) into the revolving extractor. The pump can feed the solvent at maximum volumetric flow rate of 25 l/h at 300 rpm. Solvent (liquid), extract (liquid) and raffinate (solid residue) are charged into the tanks (6). The mode of extraction can be changed by changing the selector valves (7) and the solvent can also be heated using a heater (8) with power consumption of 330 W. All reading and controls are placed in a cabinet (9).

#### **EXPERIMENTAL PROCEDURE**

#### Preparation of Extraction Material

- 1. Add 10 L distilled water and 600 g potassium hydrogen carbonate and mix them until the salt is completely dissolved.
- 2. Add approximately 4 kg of aluminium oxide grains in the above solution, make sure all grains are perfectly immersed in solution and keep it in solution for 24 hours.
- 3. Take out the grains from the solution and dry it (if needed baking tray or oven can be used).

### **Experiment #1 : Extraction Performance for discontinuous process**

- 1. Set the valve position  $V_1$  and  $V_2$  to 1 stage which means the solvent is pumped to the first stage.
- 2. Weigh 70 g or aluminium oxide grains and add it to the cell extractor.
- 3. Make sure the cell extractor i.e. first stage is exactly positioned below the solvent feed.
- 4. Turn on process pump  $P_1$  and set the solvent flow rate of 15 l/h and turn on heating element  $W_1$  and set the temperature of 30 °C.
- 5. Take the concentration reading at specific time interval (as can be seen in worksheet).

#### Experiment #2: Effect of solvent temperature on extraction performance

- 1. Make sure the filling hopper is filled with sufficient quantity of solid material.
- 2. Synchronize the speed of the solid feeder and the extractor so that a material depth approximately 40 mm per cell is achieved.
- 3. Set the valve position  $V_1$  and  $V_2$  to 1 stage which means the solvent is pumped to the first stage.
- 4. Turn on process pump  $P_1$  and set the solvent flow rate of 15 l/h and turn on heating element  $W_1$  and set the temperature of 25 °C.
- 5. Take the concentration reading at specific time interval e.g. every 30 secs (as can be seen in worksheet).
- 6. Increase the temperature to 30 °C once the extract concentration from previous experiment does not increase anymore.
- 7. Take the concentration reading at specific time interval i.e. every 30 secs.
- 8. Increase the temperature to 40 °C once the extract concentration from previous experiment does not increase anymore.
- 9. Take the concentration reading at specific time interval i.e. every 30 secs.

#### Experiment #3: Effect of solvent flow-rate on extraction performance

- 1. Make sure the filling hopper is filled with sufficient quantity of solid material.
- 2. Synchronize the speed of the solid feeder and the extractor so that a material depth approximately 40 mm per cell is achieved.
- 3. Set the valve position  $V_1$  and  $V_2$  to 1 stage which means the solvent is pumped to the first stage.
- 4. Turn on process pump  $P_1$  and set the solvent flow rate of 10 l/h and turn on heating element  $W_1$  and set the temperature of 30 °C.
- 5. Take the concentration reading at specific time interval i.e. every 30 secs (as can be seen in worksheet).

- 6. Increase the flow rate to 15 l/h once the extract concentration from previous experiment does not increase anymore.
- 7. Take the concentration reading at specific time interval i.e. every 30 secs.
- 8. Increase the flow rate to 20 I/h once the extract concentration from previous experiment does not increase anymore.
- 9. Take the concentration reading at specific time interval i.e. every 30 secs.

#### Experiment #4: Influence of number of stages on extraction performance (optional)

- 1. Make sure the filling hopper is filled with sufficient quantity of solid material.
- 2. Synchronize the speed of the solid feeder and the extractor so that a material depth approximately 40 mm per cell is achieved.
- 3. Set the valve position  $V_1$  and  $V_2$  to 1 stage which means the solvent is pumped to the first stage.
- 4. Turn on process pump  $P_1$  and set the solvent flow rate of 15.5 l/h and turn on heating element  $W_1$  and set the temperature of 30 °C.
- 5. Turn on process pump  $P_2$  and  $P_3$ . Adjust the speed in such a way that as few air bubbles as possible occur in the relevant intake line.
- 6. Take the concentration reading at specific time interval i.e. every 30 secs (as can be seen in worksheet).
- 7. Set the valve position  $V_1$  and  $V_2$  to 2 stage which means the solvent is pumped to the first and second stage.
- 8. Keep the setting on process pump  $P_1$  the same as step #4.
- 9. Turn on and adjust the speed of process pump  $P_2$  and turn on heating element  $W_2$  and set the temperature of 30  $^{\circ}\text{C}.$
- 10. Take the concentration reading at specific time interval i.e. every 30 secs (as can be seen in worksheet).
- 11. Set the valve position  $V_1$  and  $V_2$  to 3 stage which means the solvent is pumped to the first, second and third stage.
- 12. Keep the setting on process pump  $P_1$  the same as step #4 and that on process pump  $P_2$  as step #9.
- 13. Turn on and adjust the speed of process pump  $P_3$  and turn on heating element  $W_3$  and set the temperature of 30  $^{\circ}\text{C}.$
- 14. Take the concentration reading at specific time interval i.e. every 30 secs (as can be seen in worksheet).

#### REFERENCES

Geankoplis, C.J., "Separation Processes and Unit Operations, 4<sup>th</sup> edition, Prentice-Hall Inc., New Jersey, 2004

"CE 630 Solid Liquid Extraction", GUNT, 2012.

#### WORK SHEETS

#### **Experiment #1 : Extraction Performance for discontinuous process** Solvent flow rate

Solvent f	low rate	: I/r	1				
Solvent t	emperature	: °C					
Time,s	Conc., g/l	Time,s	Conc., g/l	Time, s	Conc., g/l	Time, s	Conc.,g/l
0		150		360		630	
15		180		390		750	
30		210		420		870	
45		240		450		1000	
60		270		480		1360	
90		300		510		1720	
120		330		570		2000	

# Experiment #2 : Effect of solvent temperature on extraction performance

Solvent flow rate : I/h

\*indicate when you start to change the temperature.

Time,s	Cond-4	Time,s	Cond-4	Time, s	Cond-4	Time, s	Cond-4
0		1170		2340			
30		1200		2370			
60		1230		2400			
90		1260		2430			
120		1290		2460			
150		1320		2490			
180		1350		2520			
210		1380		2550			
240		1410		2580			
270		1440		2610			
300		1470		2640			
330		1500		2670			
360		1530		2700			
390		1560		2730			
420		1590		2760			
450		1620		2790			
480		1650		2820			
510		1680		2850			
540		1710		2880			
570		1740		2910			
600		1770		2940			
630		1800		2970			
660		1830		3000			
690		1860		3030			

720	1890	3060		
750	1920	3090		
780	1950	3120		
810	1980	3150		
840	2010	3180		
870	2040	3210		
900	2070	3240		
930	2100	3270		
960	2130			
990	2160			
1020	2190			
1050	2220			
1080	2250			
1110	2280			
1140	2310			

# Experiment #3 : Effect of solvent flow rate on extraction performance

°C

Solvent Temperature :

\*indicate when you start to change the flow rate

Time,s	Cond-4	Time,s	Cond-4	Time, s	Cond-4	Time, s	Cond-4
0		1170		2340			
30		1200		2370			
60		1230		2400			
90		1260		2430			
120		1290		2460			
150		1320		2490			
180		1350		2520			
210		1380		2550			
240		1410		2580			
270		1440		2610			
300		1470		2640			
330		1500		2670			
360		1530		2700			
390		1560		2730			
420		1590		2760			
450		1620		2790			
480		1650		2820			
510		1680		2850			
540		1710		2880			
570		1740		2910			
600		1770		2940			
630		1800		2970			

660	1830	3000		
690	1860	3030		
720	1890	3060		
750	1920	3090		
780	1950	3120		
810	1980	3150		
840	2010	3180		
870	2040	3210		
900	2070	3240		
930	2100	3270		
960	2130			
990	2160			
1020	2190			
1050	2220			
1080	2250			
1110	2280			
1140	2310			

#### Experiment #4 : Effect of number of stage on extraction performance °C

Solvent Temperature :

\*indicate when you start to change the flow rate

Time,	Cond	Cond	Cond	Time,	Cond	Cond	Cond	Time	Cond	Cond	Cond
S	-2	-3	-4	S	-2	-3	-4	, S	-2	-3	-4
0				1170				2340			
30				1200				2370			
60				1230				2400			
90				1260				2430			
120				1290				2460			
150				1320				2490			
180				1350				2520			
210				1380				2550			
240				1410				2580			
270				1440				2610			
300				1470				2640			
330				1500				2670			
360				1530				2700			
390				1560				2730			
420				1590				2760			
450				1620				2790			
480				1650				2820			
510				1680				2850			
540				1710				2880			

570	1740		2910		
600	1770		2940		
630	1800		2970		
660	1830		3000		
690	1860		3030		
720	1890		3060		
750	1920		3090		
780	1950		3120		
810	1980		3150		
840	2010		3180		
870	2040		3210		
900	2070		3240		
930	2100		3270		
960	2130				
990	2160				
1020	2190				
1050	2220				
1080	2250				
1110	2280				
1140	2310				

Student tasks:

- 1. Plot concentration (yE, g/L) against time (t, sec) for every experiment. Separate the plot if there is change in parameter such as temperature or flow rate. Comment on the plots.
- 2. Do material balance for the solid-liquid leaching once the system reach steady state (experiment #2 and #3)

# EXPERIMENT #07 (UO-07)

# PARTICLE SIZE ANALYSIS (SIEVING) and ANGLE OF REPOSE

#### **OBJECTIVE:**

- 1. To determine and analyze the size distribution of a fixed granular solid
- 2. To determine the natural angle of repose for a specific materials.

#### THEORITICAL BACKGROUND

Small particle is one of chemical engineering interest. An understanding of the characteristics of masses of particulate solids is necessary in designing processes and equipment for dealing with streams containing such solids.

One way to distinguish the particle size is by sieving. In each test sieve consists of a woven wire screen with square apertures of known size and is used for the measurement of particle size. The necessary vibrating motion is imparted by a mechanical shaker to ensure reproducible results in a relatively short time.

Particle size may be specified by quoting the size of two screens, one through which the particles have passed and the other on which they are retained. However sieving is usually used to measure size distribution. The results of sieve test may be presented in a variety of ways either in tabular or graphical form.

The other way to learn characteristics of solid material is by measuring the angle of repose. The angle of repose is the steepest possible angle with the horizontal at which the material will stand when piled. Moisture content of the material is often a controlling factor and the percentage of fine material in the mass has a decided influence on the angle as the fine materials carry the bulk of moisture. Similarly, the angle depends upon the type of material including the shape and smoothness of the individual particles and their overall compactness.

#### **DESCRIPTION OF THE EQUIPMENT**



#### **EXPERIMENTAL PROCEDURE**

#### A. Sieving Experiment

- 1. Weigh material e.g. sand that will be separated or graded using shaker and sieves. Use the last container which is the one at the bottom of sieve series to make sure that the volume of material does not exceed the volume of sieves.
- 2. Transfer the material to another container or beaker.
- 3. Put the sieve with the biggest screen size at the top and the smallest at the bottom (see fig 5.1)
- 4. Set shaker time to 20 min to make sure the material is completely graded.
- 5. Carefully detach each sieve and weigh the collected material from each sieve.
- 6. Record the mass obtained from each sieve in the work sheet.
- 7. Repeat the experiment twice.

#### B. Angle of Repose in a Hele-Shaw Cell

- 1. Place test material (dry or wet) in the funnel.
- 2. Measure the angle of repose by sliding the cell and repeat.
- 3. Empty the Hele-Shaw Cell by rinsing with water if required.
- 4. Ensure dry berfore refilling the system with another material
- 5. Repeat the experiment 2 times for each type of material.

#### REFERENCES

Instruction Manual for CEN-MKII, Armfield.

#### WORK SHEETS

#### Run #1

Screen Opening, mm	Average particle diameter	Mass, g
	Total mass	

#### Run #2

#### Run #3

Screen	Average	Mass, g
Opening,	particle	
mm	diameter	
	Total mass	

Screen Opening,	Average particle	Mass, g
mm	diameter	
	Total mass	

Run	Material Tested and its condition	Angle of Repose
1		
-		
	Average	
2		
	•	
	Average	
3		
	Average	
	Average	
T		
	Average	
2		
-		
	Average	
3		
-		
	Average	

# EXPERIMENT #08 (UO-08)

#### **SEDIMENTATION**

#### **OBJECTIVE:**

- 1. To study sedimentation process and investigate the zone occurs in sedimentation tank.
- 2. To study the effect of flow rate of fresh water and suspension in sedimentation process.

#### THEORITICAL BACKGROUND

Sedimentation is a physical process of separating suspended solid in a liquid using gravity. The suspended solids can settle down if the size is greater than 10  $\mu$ m. the smaller the size the more difficult for the solids to settle down because Brownian motion and electrostatic force balances the gravitational force. Unless coagulant is added, it is not likely to settle down very small particle naturally.



Figure 6.1. The Four Functional Zone of Continuous Sedimentation Process

In typical sedimentation tank, there will be four functional zones. Inlet zone is the zone where all solid particles are well mixed and flow in the same direction as the liquid. The settling occurs in settling zone as the liquid continue flowing to outlet zone. Most of solid particles will be settled and collected continuously in the sludge zone.

In this experiment, to determine the actual sediment content at inlet and outlet to the sedimentation tank, samples of the mixtures are taken. This batch system will show four different zones as can be seen in figure 6.2. In hindered settling, particles become quite close and the liquid is flowing upward due to displacement by settling particles. This will reduce the particle settling velocity.

Clear water		
Hindered Settling		
Transition zone		
Compression		
zone		

Figure 6.2. The four zones of Batch Sedimentation

At the bottom of sedimentation column, the particles can contact each other and indeed the particle concentration becomes very high. The settled solids are compressed by the weight of overlying solids and water is squeezed out while the spaces between solid particles are getting smaller. This zone is known as compression zone. Between hindered settling zone and compression zone there will be transition zone.

#### **DESCRIPTION OF THE EQUIPMENT**



- 1. Suspension flow meter
- 4. Bypass
- 7. Fresh water/suspension mixing zone
- 2. Fresh water flow meter
- 5. Suspension pump

Figure 6.3. Sedimentation Unit

8. Outlet

- 3. Switch box
- 6. Suspension tank
- 9. Sedimentation tank
- 10. Baffle plate

#### EXPERIMENTAL PROCEDURE

#### Preparation

- 1. Close fresh water, suspension and by pass valves.
- 2. Close drain valve of suspension tank.
- 3. Ensure that no hose lies in the suspension tank.
- 4. Fill the suspension tank with 80 L of fresh water, then add 1.6 kg of precipitated calcium carbonate.
- 5. Fully open bypass valve.
- 6. Switch on main switch and pump switch. The suspension is now mixed by the pump.

#### Performing the experiment

1. Drain sedimentation tank via drain valve and rinse with fresh water.

- 2. Remove or insert baffle plate (depending upon the mode of operation: undisturbed or disturbed sedimentation. If disturbed sedimentation is chosen then insert the baffle approximately 400 mm from inlet weir. Set a gap of 20-30 mm between bottom of baffle plate and base of sedimentation tank).
- 3. Insert plug into the inlet weir.
- 4. Close drain valve of sedimentation tank.
- 5. Carefully open fresh water valve and fill the sedimentation tank with fresh water, then set desired flow rate.
- 6. Move the scale on the riser for the suspension flow meter such that the zero mark is aligned with the water level in the riser.
- 7. Carefully open suspension valve and set desired flow rate (the suspension mixes with the fresh water in the inlet chamber and flows over the inlet weir into the sedimentation tank.
- 8. Keep checking suspension flow meter for constant flow rate, adjust suspension valve if necessary.
- 9. After 10 minutes, open the valves at both sampling points at the inlet and let the suspension flow to sedimentation cone.
- 10. Collect 1000 mL of the suspension from the inlet.
- 11. Immediately afterwards, open the valve at the sampling point at the outlet and let the suspension flow to another sedimentation cone.
- 12. Collect 1000 mL of the suspension from the outlet as well.
- 13. After 24 hour settling duration, read the amount of sediment in the sedimentation cones and compare (you can use flashlight to shine the sedimentation cone to enhance reading).

#### REFERENCES

HM 142 Gunt Experiment Manual.

#### NOTATION

#### WORK SHEETS

Time	Fresh water flow rate in l/h	Suspension flow rate in I/min	Amount of sediment in sedimentation cone at inlet in ml	Amount of sediment in sedimentation cone at outlet in ml	Calculated amount of sediment in sedimentation tank in ml
	100	0.5			
	100	1			
	100	2			
	200	2			
	400	2			