

Mass Transfer Operations-II

Chemical Engineering Department

L.E.COLLEGE-MORBI

List of Experiments

Exp. No.	Objective	CO Mapped
1	To generate VLE data for binary liquid -liquid system.	3160501.1
2	To study the characteristics of the Steam distillation.	3160501.1 3160501.2
3	To study differential distillation and verify Rayleigh equation.	3160501.2
4	Virtual lab experiment-1: To study the operation of a batch rectification column under constant or total reflux condition. [http://vmt-iitg.vlabs.ac.in/Column_tray_efficiency(theory).html]	3160501.2
5	To study the humidification operation and to calculate all psychometric parameters of air-water system.	3160501.1
6	To study the performance of Cooling tower.	3160501.2 3160501.3
7	To generate drying rate curve for a batch drying test of a given material.	3160501.2
8	Virtual lab experiment-2: To study the drying characteristics of a solid under forced draft condition. [http://vmt-iitg.vlabs.ac.in/Forced_draft_tray_dryer(theory).html]	3160501.1 3160501.2
9	To study characteristics of adsorption of moisture on silica gel.	3160501.3
10	To verify the Freundlich's Isotherm equation for adsorption of dilute solution of acetic acid over activated charcoal.	3160501.3

Experiment No: 1.

Date:

VAPOUR LIQUID EQUILIBRIUM

AIM: To determine the vapor liquid equilibrium for the acetic acid water system and compare it with the theoretical values.

APPARATUS:- VLE apparatus, Round bottom flask, Measuring Cylinder, Conical flask, Burette, Pipette, Thermometer.

CHEMICALS: Water, Acetic Acid, Indicator, NaOH.

THEORY:

The successful application of distillation methods depends greatly upon an understanding of the equilibria existing between the vapor and liquid phases of the mixtures encountered. The vapor-liquid equilibrium (VLE) for each pure substance of the binary mixture is its vapor-pressure-temperature relationship. The vapor and liquid phases are allowed to reach the equilibrium. The graphical representation of vapor-liquid equilibrium is represented in two ways. First, the mole fraction of more volatile component in vapor phase (y^*) in equilibrium with liquid is plotted as the mole fraction of more volatile component in liquid phase (x). Second, the boiling temperature of the binary mixture is plotted as x and y^* .

PROCEDURE:

- Connect the VLE apparatus.
- Take 340 ml acetic acid and 110 ml water in the RBF.
- Start the water circulation through the condenser.
- Start the heating of solution and maintain constant temperature.
- Temperature should be maintained between the normal boiling points of acetic acid (118°C) and water (100°C).
- Allow condensate to reflux from the condenser to the RBF at least for 10 min.
- Take 10 ml sample of condensate and titrate it against 1 N NaOH.
- Take 10 ml sample of residue and titrate it against 1 N NaOH.
- Find out the x and y^* for a given temperature.
- Repeat the same procedure at different temperature.

OBSERVATIONS:

- Volume of Acetic Acid = _____ ml.
- Volume of Water = _____ ml.
- Temperature = _____ °C

OBSERVATION TABLE:

Sr.No	Temp.°C	Amount of acetic acid				Amount of water			
		Condensate		Residue		Condensate		Residue	
		Gms.	Mole	Gms.	Moles	Gms.	Moles	Gms.	Moles
1									
2									
3									
4									
5									

RESULT TABLE:

Sr.No	Temp. °C	X	y*
1			
2			
3			
4			
5			

CALCULATIONS:

FOR CONDENSATE:

- By performing titration find out the normality of solution.
- Mass of Acetic Acid = $N \times \text{eq.wt.} \times \text{volume of solution (in liter)}$
 = _____ gms.

- Moles of Acetic Acid = $\frac{\text{mass of Acetic Acid}}{\text{mol.wt. of Acetic Acid}}$
- Mass of Water = total mass of condensate – mass of Acetic Acid
- Moles of Water = $\frac{\text{mass of Water}}{\text{mol.wt. of Water}}$
- Mole fraction of water in vapor $y^* = \frac{\text{moles of water}}{\text{moles of (water+ acetic acid)}}$

FOR RESIDUE :

(1) Apply same calculation steps as the condensate.

(2) Mole fraction of water in liquid $x = \frac{\text{moles of water}}{\text{moles of (water + acetic acid)}}$

GRAPHS:

- $x \rightarrow y^*$
- $T \rightarrow x, y$

Conclusion:

Marks:

Signature:

Experiment No. : 2

Date:

STEAM DISTILLATION

Aim:- To study the characteristics of the steam distillation.

Apparatus:- Steam distillation setup, measuring flask

Introduction

Steam distillation is a unit operation of considerable practical importance. The steam distillation is a term applied to a distillation process with the open steam i.e. where the steam is in direct contact with the distillation system in either a batch or the continuous operation. The steam distillation has the special value where it is desired to separate a substance at the temperature lower than their boiling point because of the heat sensitivity. The process of steam distillation is also applied for the separation of ortho and para nitro phenol to separate relatively small amount of the volatile impurity from the large amount of the material.

Theory

In the process of the steam distillation a liquid is distilled by feeding the open steam to the distillation still. The steam carries with its vapors of the volatile liquids and is then condensed to separate the liquid from the water. The process of the steam distillation is generally carried out with the cylindrical still with the injection of the steam through the coils placed in the bottom of the stills.

The process of the steam distillation is possible under the following conditions;

1. If the P is the total pressure of the system, P_A is the vapor pressure of the liquid, P_H is the vapor pressure of the water then,

$$P = P_A + P_H$$

For distillation at atmospheric conditions total pressure = 101.325 kN/m²

2. If the plot is made of P_A Vs temperature and $(101.325 - P_H)$ Vs temperature on the same graph then the point of intersection of the two curves gives the temperature of the distillation.

The steam requirement for the process of the steam distillation is as follows,

Let N_A and N_H represent the mole fractions of the liquid and the water respectively in a vapor phase than applying the Dalton's law of the partial pressure;

$$P_A^0 = P (N_A / (N_A + N_H))$$

Where, N_A and N_H represent the moles of liquid and water respectively. P_A is the partial pressure of A and the P is the total pressure.

A similar equation can be written for the aqueous phase thus,

$$P_A^0 / P_H^0 = N_A / N_H = [(W_A / M_A) / (W_H / M_H)]$$

Where, W_A and W_H represent the Kg of liquid and water respectively,

M_A and M_H represent the molecular weight of the liquid and water respectively. Hence we get

Putting $P_A^0 = P_A$ and $P_B^0 = P_B$

$$\frac{W_H}{W_A} = \frac{P_H}{P_A} \times \frac{M_H}{M_A}$$

- The obtained steam required in the steam distillation process, the values of P_H and P_A at the distillation temperature are substituted and the ratio of W_H / W_A is computed.
- This the amount of the total steam condensed to the amount of per unit weight of the liquid condensed.

Procedure

- The distillation unit is charged with the predetermined amount of the benzene.
- The cooling water to the condenser is started.
- The steam generated is filled with the water upto the indicated mark and the heating is started.
- The steam generated is injected to the distillation still and the process of the distillation is than started.
- The process of the distillation is than continued for the sufficient length of the time so that the amount of 60 – 70% of the benzene charge is distilled.
- The distillate collected on an accumulator is than separated into an organic layer and an aqueous layer.
- The two phases are than separately weighted.
- Similarly the components at the bottom are weighted after cooling to the room temperature.

Observations:

Density of water =

Density of nitrobenzene =

Vapor pressure of water at distillation temperature =

Vapor pressure of nitrobenzene at distillation temperature =

Volume of Water collected =

Volume of nitrobenzene collected =

Boiling point of nitrobenzene =

Temperature at which nitrobenzene boils (Distillation Temperature) =

Calculations:

Result:

Conclusion

Marks:

Signature:

Experiment No. : 3

Date:

Aim: To study differential distillation and verify Rayleigh equation

Apparatus: Distillation column, burette, pipette, measuring flask.

Chemical: Acetic acid, NaOH, water

Theory:

A batch of liquid is charged to a kettle or still fitted with some sort of heating device which such as a steam jacket. This is then boiled slowly and the vapors are withdrawn as rapidly as they form to a condenser where they are liquefied and the condensate is collected in receiver. The apparatus is essentially a large-scale replica of the ordinary laboratory distillation flask and condenser. The first portion of the distillate will be the richest in the more volatile substance, and as distillation proceeds, the vaporized product becomes leaner. The distillate is collected in different batch called cuts. This gives a series of distilled products of various purities. The vapor issuing from a true differential distillation is at any time in equilibrium with the liquid from which it rises but changes continuously in composition. From material balance,

$$\ln F/W = \int_{x_w}^{x_F} \frac{dX}{(y^*-x)}$$

This is the Rayleigh equation.

Where, F = mole of charge of composition X_F

W = mole of residue liquid of composition X_w

Graphically we can plot $1/(y^*-x)$ and determined the area under the curve between the given limit

Procedure:

- Prepare 1 litre of 1N NaOH solution.
- Take 40 ml Acetic acid and 160 ml water in a round bottom flask.
- Heat a solution by using a burner note down the boiling point of a solution and heat it for 15 to 20 minutes after boiling point is reached.
- Then titrate the distillate with 1N NaOH and find concentration of Acetic acid in distillate.
- Calculate mole fraction of water in distillate and by the Rayleigh equation.

Observation:

1. Volume of feed=
2. Volume of A.A.=
3. Volume of water=
4. Weight of feed=
5. Weight of A.A.=
6. Weight of water=
7. Distillate collected=
8. Residue collected=

Buratte:

Pipette:

Indicator:

End point:

Buratte reading (distillate)=

Mass of A.A. in distillate=

Mass of water in distillate=

Calculation:

For distillate

$$N_1V_1 = N_2V_2$$

Result:

$$\ln (f/w) =$$

Marks:

Signature:

Experiment No. : 5

Date :

HUMIDIFICATION OPERATION

Objective: To study the humidification operation and to calculate all psychometric parameters of air-water system.

Apparatus: Dry bulb Thermometer, Wet bulb Thermometer.

Chemicals: Water

Theory: If liquid phase is pure liquid and is contacted with a gas mixture consisting of two or more components, then mass transfer from liquid to gas phase is known as humidification. If the mass transfer is in the opposite direction, the operation is known as dehumidification. In this operation not only the mass is transferred but simultaneously heat energy is also transferred. The purpose of such operation may include humidification of the gas, dehumidification and cooling of the gas, measurement of its vapor content and cooling of liquid.

Procedure:

- The bulb of the given simple thermometer is brought in to contact with the air; the thermometer will indicate the dry bulb temperature of the air.
- Now prepare a wet bulb thermometer.
- The bulb of the thermometer is covered with a piece of cotton and which is wetted by water.
- Thus a small water layer is formed on the bulb of the thermometer.
- Note down the reading from the thermometer, which indicates the wet bulb temperature.
- Calculate the other properties of the air-water system from the psychometric chart and tabulate in the result.
- For vapor pressure data use Perry's handbook

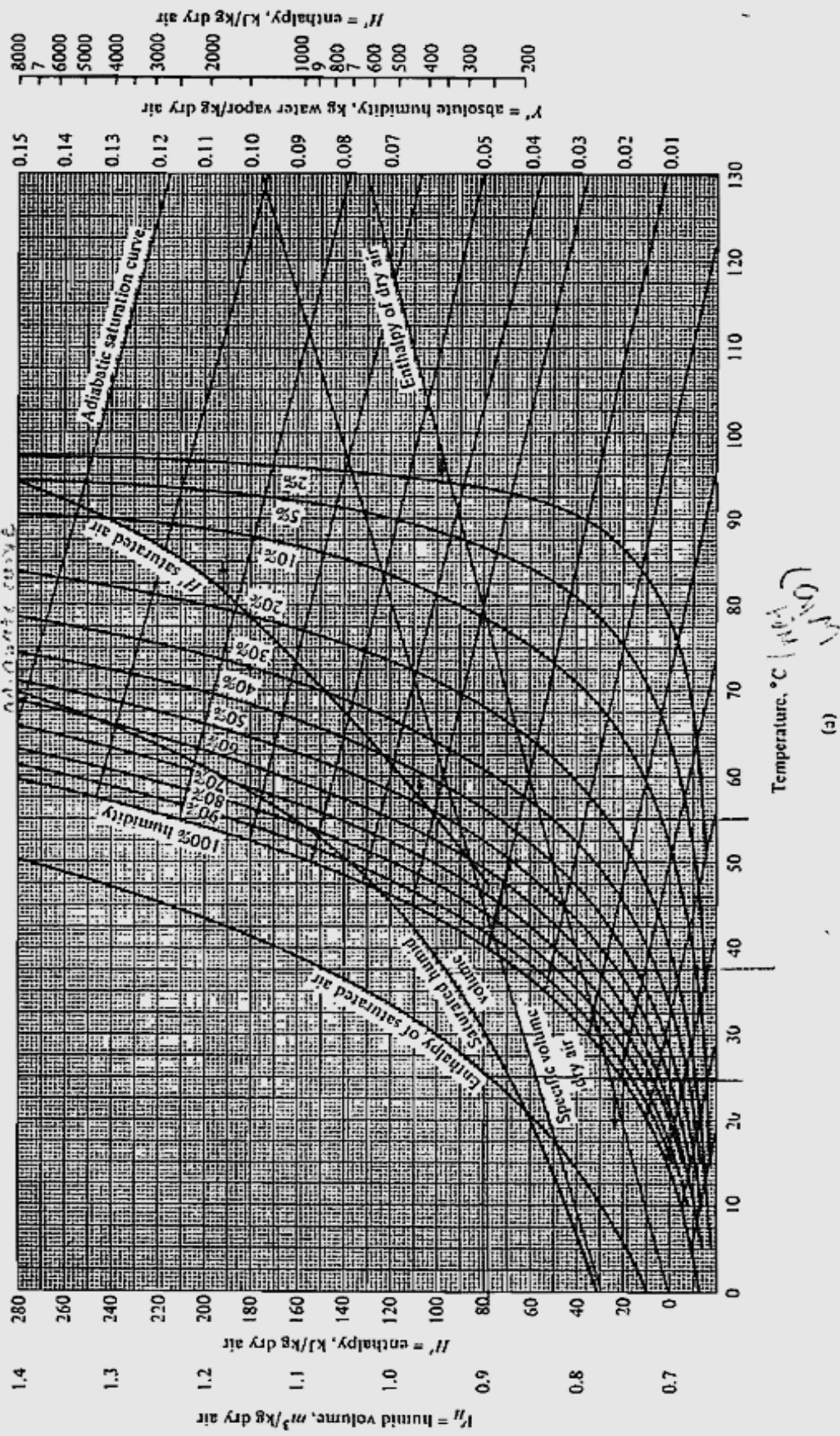
Calculations:-

calculate various parameters using reference books.

Result:

Sr. No	Parameter	Value with unit
1.	Dry bulb temperature	
2.	Wet bulb temperature	
3.	Absolute humidity (y)	
4.	Molal absolute humidity (Y)	
5.	Relative humidity (%RH)	
6.	Humid volume (v_H)	
7.	Humid heat (C_s)	
8.	Enthalpy(H)	

At 50°C and 50% RH humidity if need to cool, cool at 1000
 adiabatic curve



Psychrometric chart for air-water vapor, 1 std atm abs, in SI units. (a)

Experiment No. :6

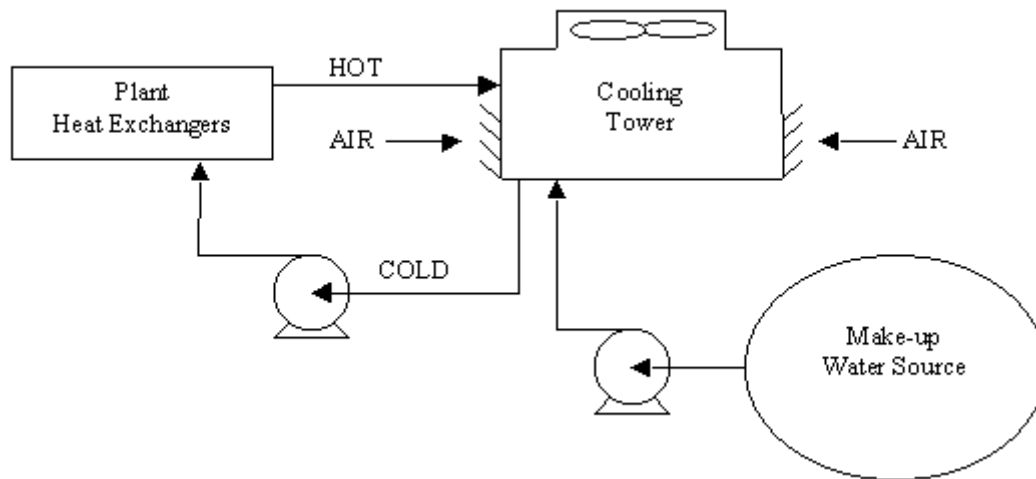
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COOLING TOWER

Objective: To study the performance of cooling tower

Introduction

Cooling towers are a very important part of many chemical plants. They represent a relatively inexpensive and dependable means of removing low-grade heat from cooling water.



Closed Loop Cooling Tower System

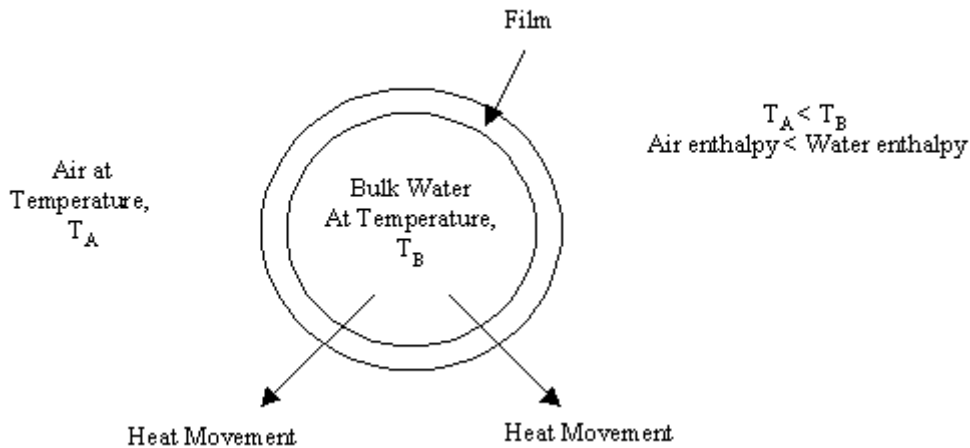
The make-up water source is used to replenish water lost to evaporation. Hot water from heat exchangers is sent to the cooling tower. The water exits the cooling tower and is sent back to the exchangers or to other units for further cooling.

Cooling of water can be carried out on small scale either by allowing it to stand in an open pond or by the spray pond technique in which it is dispersed in a spray form and then collected in a large, open pond. Cooling takes place both by the transference of sensible heat and by evaporative cooling as a result of which sensible heat in the water provides the latent heat of vaporization.

On the large scale, air and water are brought into countercurrent contact in a cooling tower, which may employ either natural draught or mechanical draught. The water flows down over a series of wooden slats, which give a large interfacial area and promote turbulence in the liquid. The air is humidified and heated as it rises, while the water is cooled mainly by evaporation.

Theory

The transfer of sensible and latent heat transfers heat from water drops to the surrounding air.



Water Drop with Interfacial Film

This movement of heat can be modeled with a relation known as the Merkel Equation:

$$\frac{KaV}{L} = \int_{T_2}^{T_1} \frac{dT}{h_w - h_a} \quad 1$$

Where,

- KaV/L = tower characteristic
- K = mass transfer coefficient (lb water/h ft²)
- a = contact area/tower volume
- V = active cooling volume/plan area
- L = water rate (lb/h ft²)
- T₁ = hot water temperature (°F or °C)
- T₂ = cold water temperature (°F or °C)
- T = bulk water temperature (°F or °C)
- h_w = enthalpy of air-water vapor mixture at bulk water

Temperature (J/kg dry air or Btu/lb dry air)

h_a = enthalpy of air-water vapor mixture at wet bulb temperature (J/kg dry air or Btu/lb dry air)

Thermodynamics also dictate that the heat removed from the water must be equal to the heat absorbed by the surrounding air:

$$L(T_1 - T_2) = G(h_2 - h_1) \quad 2$$

$$\frac{L}{G} = \frac{h_2 - h_1}{T_1 - T_2} \quad 3$$

Where:

- L/G = liquid to gas mass flow ratio (lb/lb or kg/kg)
- T₁ = hot water temperature (°F or °C)
- T₂ = cold water temperature (°F or °C)
- h₂ = enthalpy of air-water vapor mixture at exhaust wet-bulb

- Temperature (same units as above)
 h1 = enthalpy of air-water vapor mixture at inlet wet-bulb temperature
 (Same units as above)

The tower characteristic value can be calculated by solving Equation 1 with the Chebyshev numerical method:

$$\frac{KaV}{L} = \int_{T_2}^{T_1} \frac{dT}{h_w - h_a} = \frac{T_1 - T_2}{4} \left(\frac{1}{\Delta h_1} + \frac{1}{\Delta h_2} + \frac{1}{\Delta h_3} + \frac{1}{\Delta h_4} \right)$$

Where :

Δh_1 = value of $h_w - h_a$ at $T_2 + 0.1(T_1 - T_2)$

Δh_2 = value of $h_w - h_a$ at $T_2 + 0.4(T_1 - T_2)$

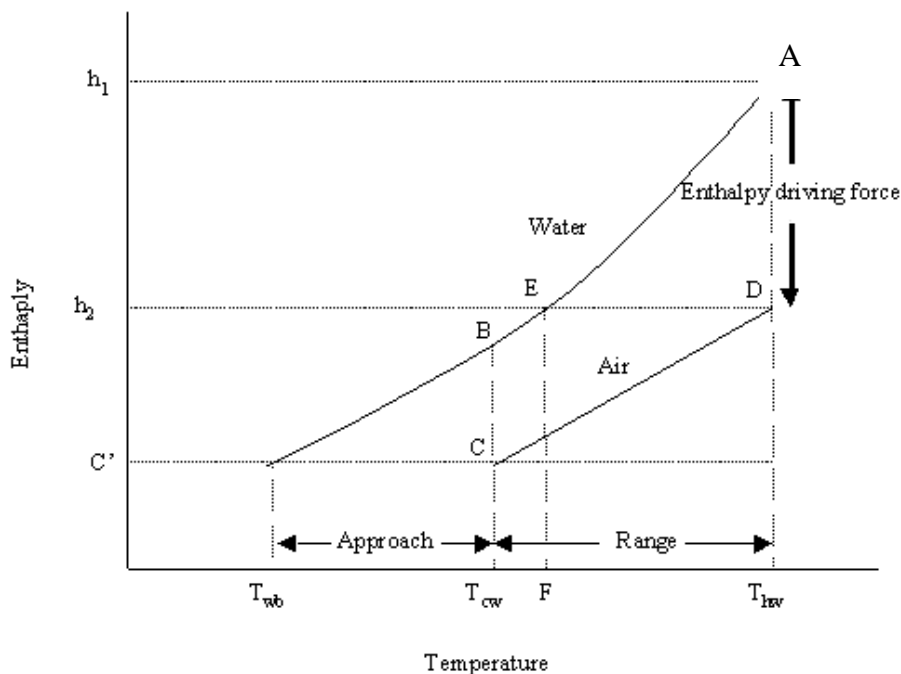
Δh_3 = value of $h_w - h_a$ at $T_1 - 0.4(T_1 - T_2)$

Δh_4 = value of $h_w - h_a$ at $T_1 - 0.1(T_1 - T_2)$

4

The following represents a key to Figure 2:

- C' = Entering air enthalpy at wet-bulb temperature, T_{wb}
 BC = Initial enthalpy driving force
 CD = Air operating line with slope L/G
 DEF = Projecting the exiting air point onto the water operating line and then onto the temperature axis shows the outlet air web-bulb temperature



1: Graphical Representation of Tower Characteristic

As shown by Equation 1, by finding the area between ABCD in a figure-2, one can find the tower characteristic. An increase in heat load would have the following effects on the diagram as shown in Figure-2:

1. Increase in the length of line CD, and a CD line shift to the right
2. Increases in hot and cold-water temperatures
3. Increases in range and approach areas

The increased heat load causes the hot water temperature to increase considerably faster than does the cold-water temperature. Although the area ABCD should remain constant, it actually decreases about 2% for every 10 °F increase in hot water temperature above 100 °F. To account for this decrease, an "adjusted hot water temperature" is used in cooling tower design.

Procedure

- Fill the storage tank with water and switch on the heater.
- Set the temperature of heater to the desired temperature. (around 45 – 50°C)
- Mix the water periodically to ensure uniform temperature in the tank by switching on the pump intermittently with the bypass valve in totally open position.
- Fill the water in the bucket for the wet bulb temperature thermocouple using wick provided at the bottom as well as top of the column. Make sure that the wick is properly dipped in the water filled.
- After achieving the desired temperature in the tank as indicated in digital temperature indicator, switch on the air compressor and allow the air to flow from the bottom distributor.
- Adjust the flow of air by using damper, if provided for air flow control.
- Note down the dry bulb and wet bulb temperature of the inlet air as shown by the digital temperature indicator on respected position mounted on the control panel.
- Switch on the pump and adjust the desired flow rate of water in the range of (2 – 4 LPM) using Rotameter provided on the delivery line. Note down the inlet temperature of hot water.
- Allow the tower to run in this condition at least for 5-10 minutes.
- After 5-10 minutes note down the temperature of the water outlet and air outlet temperature (wet bulb and dry bulb).
- Calculate the air flowrate by noting air velocity on Anemometer.
- Also note the pressure drop across the column as indicated by the inclined manometer.
- Change the flow rate of water and repeat the experiment or change the inlet temperature of hot water and repeat the experiment.

Observations:

1. Inlet water flow rate(LPM)=
2. Inlet air velocity(m/s) =
3. Inlet water temperature(T1)=
4. Outlet water temperature(T2)=
5. Inlet air dry bulb temperature(T3)=
6. Inlet air wet bulb temperature(T4)=

Calculation:

Result:

Conclusion:

Marks:

Signature:

Experiment: 7

Date:

ATMOSPHERIC DRIER

Objective: To generate drying rate curve for a batch drying test of a given material.

Apparatus: Tray Drier

Theory :

The term drying refers generally to the removal of moisture from a substance. Generally in this operation relatively small amount of water or other liquid may also be removed from the solid material to reduce the content of residual liquid to an acceptably low value. Drying is usually the final step in series of operations, and the product from a dryer is often ready for final packaging.

Water or other liquids may be removed from solids mechanically by pressing or centrifuging or thermally by vaporization. The liquid content of a dried substance varies from product to product. Occasionally the product contains no liquid called bone dry.

There is no simple way of classifying drying equipment. Some dryers are continuous and some operate batch wise, some agitate the solids and some are essentially unagitated. Operation under vacuum may be used to reduce the drying temp. Some dryers can handle almost any kind of material, while other is severely limited in the type of feed they can accept. A major division may be made between (a) Dryers in which the solid is directly exposed to a hot gas (usually air) and (b) dryers in which heat is transfer to the solid from an external medium such as condensing steam, usually through a metal surface which the solid is in contact.

Procedure:

1. First clean the drying chamber remove any dust or another particles in the unit.
2. Position the wet bulb /dry bulb thermometer at the proper place on the unit.
3. Take known quantity of dry material (sand of known size) and add to it known quantity of water.
4. Note the reading of the dry tray, area of the tray and total weight of the tray with sample
5. Switch the bower and heater box. Maintain the constant flow rate of air.
6. Achieve the steady state, note down dry bulb & wet bulb temp at both the end of drying chamber.
7. Load the tray in the drying chamber on the tray support.
8. Close the door of chamber, so that no air can be leaks out through the door.
9. Wait for some time and note the values of the dry and wet bulb temp. at the entry and exit of the drying chamber, weight of the tray, at constant flow rate of air at an interval of 10 min.
10. Take 5 such readings.

11. For taking reading of the flow of air through the chamber an orifice manometer arrangement is provided and for regulating the flow one gate valve is also provided.

Observations:

1.	Weight of empty tray	=	
2.	Weight of the dry sample (S _s)	=	gm
3.	Weight of water used	=	gm
4.	Total Weight	=	
5.	Total area of the tray (A)	=	

Observation Table:

Sr.No	Time (t) (min.)	Moisture retained (gm)	Δx	Δt
1.				
2.				
3.				
4.				
5.				
6.				

Calculations:

1. Calculate the values of moisture content (X), where $X = \frac{\text{weight of moisture retained in Kg}}{\text{Dry weight of solid in Kg}}$
2. Calculate rate of Drying (N) using $N = \frac{S_s * (\Delta X)}{A * (\Delta t)}$

Result:

Sr.No	X	Rate of drying N

Graph : Plot the graph of (1) X Vs t (2) N Vs X 3. N Vs t

Conclusion:

Experiment No. : 9

Date:

ADSORPTION

Objective: To study the characteristics of adsorption of moisture in silica gel.

Apparatus :- Glass tube to form adsorption in silica gel, dryer,blower, weight box.

Material:- Silica gel.

Theory :-

When solid is coming into contact with a mixture of gas or liquid, it preferentially adsorbs specific substance on its surface e.g., when atmospheric air is passed through a bed containing silica gel the moisture present in the air gets adsorbed on the surface of silica gel. Similarly the unwanted colored material can be removed from aqueous solution of sugar.

The amount of substance adsorbed depends upon the nature of solid; the temperature and gas pressure. When the adsorbent is fresh the rate of adsorption is maximum. As the time passes the adsorbent rate falls to zero. By removing adsorbate from adsorbent with the help of different separation process the cycle can be once again started.

Procedure:-

- Take about 50 gms. Of silica gel in a watch glass. Keep it in a dryer at about 80⁰C.
- Place the silica gel on the plate in the dryer.
- Then, place silica gel in glass tube and blow the air from a blower for five minute.
- Repeat the process of air flowing for five minutes and again weight it.
- Repeat the process of air flowing till constant weight is reached due to saturation of silica gel.
- Tabulate the readings.

Observation Table:-

Sr. No.	Time (min.)	Weight of silica gel (gms.)	Moisture content

Graph:- Plot graph of Weight of silica gel (gms.) Vs. Time (min.)

Result: -

Marks:

Signature:

Experiment No: 10

Date:

FREUNDLICH ISOTHERM (ROOM TEMPERATURE)

Objective: To study and verify the Freundlich's Isotherm equation for adsorption of dilute solution of acetic acid over activated charcoal.

Apparatus: Conical flask 250 ml (6 Nos.), Beaker 500 ml (2 Nos.), Measuring Cylinder

Chemicals: Water, Acetic Acid, Charcoal, NaOH, Phenolphthalein.

Theory:

The adsorption operation exploits the ability of certain solids preferentially to concentrate specific substance from solution onto their surfaces. In this manner the component of either gaseous or liquid solution can be separated from each other. There are two types of adsorption phenomena. Physical adsorption: a readily reversible phenomenon is the result of intermolecular forces of attraction between molecules of the solid and substance adsorbed. Chemical Adsorption: which is the result of chemical interaction between solid and adsorbed substance.

Over a small concentration range and particularly for dilute solutions, the adsorption isotherms can frequently be described by an empirical expression Freundlich's i.e.

$$Y = m \cdot X^n$$

Where,

Y= gm of solute per gm of solvent

X= gm of solute / gm of adsorbent

N= component in isotherm

M= slope of equilibrium adsorption isotherm

Taking log both side,

$$\log Y = \log m + n \log X$$

Now, plot of log Y v/s log X straight-line slope is n and intercept is log m.

Procedure:

1. Prepare Acetic Acid solution (1N, 0.5N, 0.125 N, 0.625N) by dissolving require Acetic Acid in 250 ml water in 500 ml beaker.
2. Weigh 2 gm of activated charcoal five times separately in five conical flasks. Add 100 ml acetic acid into those five conical flasks.
3. Shake the flasks gently and keep them for 15 min. with intermittent shaking. Filter the contents of flasks by using filtration assembly.
4. After weighing the filtrate titrate it against 0.1 N NaOH solutions after adding phenolphthalein.
5. The weight of acid adsorbed is calculated from the normality of filtrate and graph of

log Y v/s log X.

Observations:

1. Solution Temperature _____ °C
2. Weight of Activated Charcoal taken in each flasks = 1 gm.
3. Burette - 0.1 N NaOH solution.
4. Pipette - Acetic Acid Solution.
5. Phenolphthalein as indicator.
6. Color change → color less to Pink.

Observation Table:

Sr. No.	Normality of Acetic acid before adsorption	Initial concentration of acetic acid (gm/lit)	Weight of acetic acid in sample (A)	Final concentration of acetic acid (gm/lit)	Weight of acetic acid in sample after adsorption (B)	Weight of adsorbed, A-B=X (gm)	Normality of Acetic acid after adsorption

Sr.No.	Weight of adsorbent, M gm	X/M	ln X/M	ln Co

Calculation:

Normality of filtrate :

- N_1 = Normality of acetic acid in filtrate
- N_2 = Normality of NaOH
- V_1 = volume of filtrate
- V_2 = volume of NaOH (Burette Reading)

$$N_1 V_1 = N_2 V_2$$

$$N_1 = \text{_____} N.$$

Conc. of filtrate(C^*) in gm/Litre = $N \times \text{Eq. Wt}$

$$C^* = \text{_____} \text{ gm/Liter}$$

Weight of acetic acid in filtrate = $C^* / \text{volume of sample for filtration}$

Plot the graph of $\ln X/M$ vs $\ln C_o$ and obtain the values of slope and intercept from the graph.

Slope (n) =

Intercept = $\ln k =$

Result: Freundlich constants $n =$ _____ ; $k =$ _____

Conclusion: