



الجامعة الألمانية الأردنية
German Jordanian University

School of Medical Sciences

Pharmaceutical and Chemical Engineering Department

Laboratory Manual

For

SEPARATION PROCESSES

PCE562



Edition: 2022

General Information:

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Lab: Separation Processes Lab , M 015

Course Objective:

Applying Separation concepts, heat and mass transfer principles on different experimental fields.

Course Evaluation:

Short Reports	30%
Full Reports	15%
Quizzes	15%
Final Exam	40%
Total	100%

Attendance:

- Attendance in all class sections is **REQUIRED**.
- You should inform the instructor as soon as possible if you must miss a class.
- University attendance regulations state that if you may miss more than 2 lab sessions for unexcused reasons or risk, you will be permanently dismissed from the lab. Dismissal from the lab automatically results in removal from the lecture.
- If you miss any lab experiment, you will get **ZERO** in its report.

Lab Preparation and Lab Reports:

You must be prepared for the experiment **BEFORE** you come to lab in order to complete the experiment in the allotted time. Each lab section is full and there will be neither make-up sessions nor space for you to work in other sections. If you miss a lab, you **MUST** discuss the issue with the course instructor (preferably beforehand) to see what options you have.

Lab Reports:

Short Reports **MUST** be graded, one for each group, **AT THE BEGINNING** of the lab session that follow the one you perform the experiment in. If you don't complete the report and turn it in at the beginning of the next lab, you will **NOT** be allowed to participate in the lab that day.

After each lab session you will be required to analyze the data you collected, analyze the sources of error, and make discussion and conclusions about the work you did.

Short Report should consist of the following:

- Formal Cover Page
- Data Tables and Graphs.
- Sample of Calculations.
- Results and Discussion.
- Conclusion and Recommendations.

Your **Full report** **MUST** be graded, one for each student, two weeks after performing the experiment. It should consist of the following:

- Formal Cover Page.
- Abstract.
- Table of Contents.
- List of Figures and Tables.
- List of Symbols.
- Introduction.
- Theory.
- Experimental Details (apparatus and procedure).
- Results and Discussion.
- Conclusion and Recommendations.
- References.
- Appendices.

Student Conduct:

- Any student that is more than 20 minutes late to the lab will be asked to leave. *There will be no chance for a make-up lab in this situation.*
- Every student must have his/her own lab coat and lab glasses or goggles in order to enter the lab. We have only a small supply of goggles for borrowing if you should forget yours, and doing so will “cost” points off of your technique grade in the class.
- All labs, instruments, glassware, and tables are to be cleaned and neat before you leave the room.
- All cases of *cheating, plagiarism, or dishonesty* will receive a zero and be reported to the Dean of Students. All work you submit **MUST** be your own. Unless specifically noted in class, **EACH** student must turn in his/her own lab reports.

Lab Schedule

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EXPERIMENT 1

SPRAY DRYER

Theory

The “spray drying” process is largely used in industry for drying substances in solution, suspension, emulsion and so on. What results from the process is a homogenous powder composed by generally spherical particles with diameter between 10-140 μm according to the operative conditions.

Classical examples obtained with this procedure are the powdered milk, the instant coffee, the powder detergents, and so on.

In “spray drying” the liquid is pumped into an atomizing nozzle spraying its content into small drops, inside a chamber crossed through by a heated air flow at a temperature over the boiling one of the solvent. When in contact with the hot air, the solvent contained into the drops evaporates and is removed by the same air flow from the chamber, together with the solid.

At the chamber outlet, a cyclone separates the solid particles that are collected into a proper container.

Apparatus

Mod. MSD/EV is a laboratory spray dryer which, thanks to the main components made in glass, enables to follow and visualize the drying process from the atomizer nozzle up to the separator cyclone outlet.

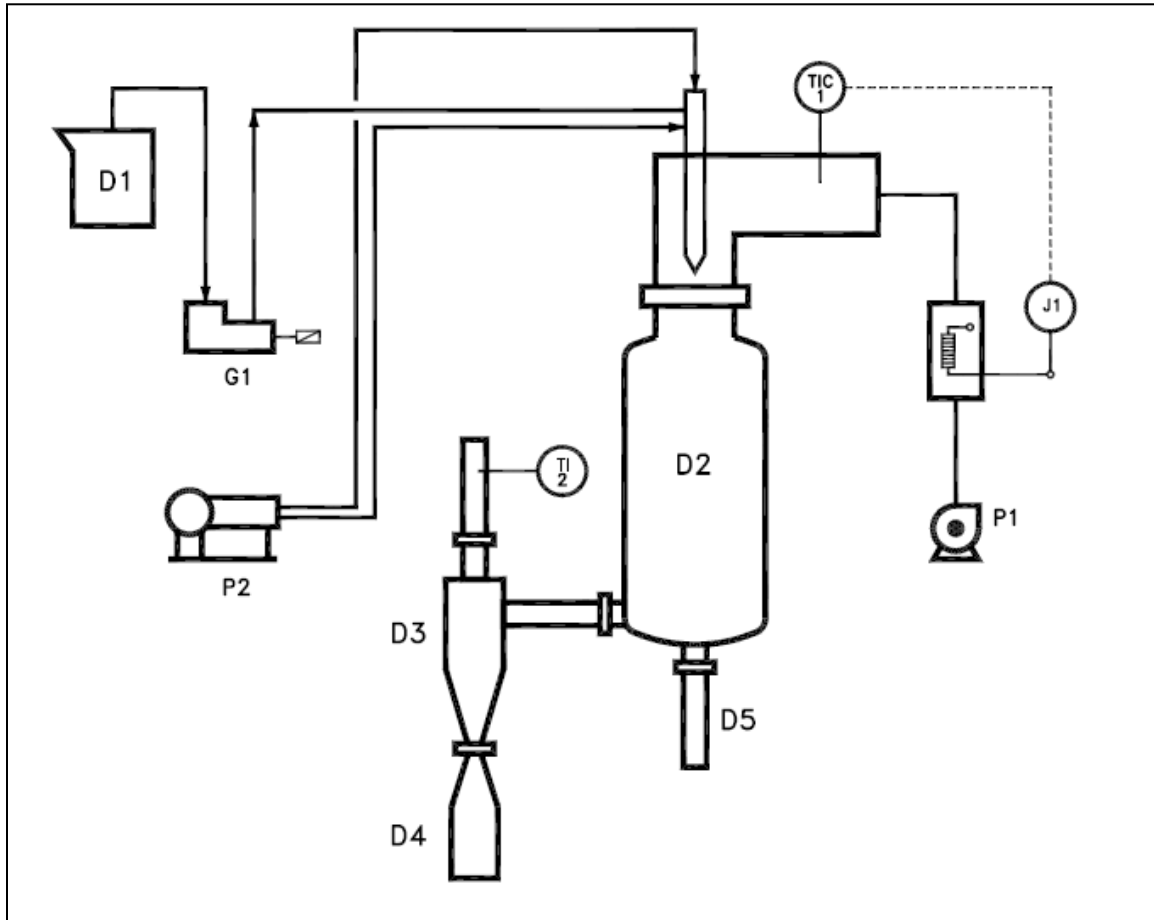


Figure 1.1: Spray Dryer configuration.

The solution to be dried is put into the container D1 and then it is sent by the peristaltic pump G1 to the atomizer nozzle spraying into the drying chamber D2 in equicurrent to the hot air flow produced by the blower P1.

The air temperature (TI1), heated by the resistor J1, is controlled by a PID controller; the one coming out from the cyclone (TI2) is only measured.

The bottom of the drying chamber is provided with a tank D5 for collecting the condensates or coarse particles.

The air flow from the chamber D2 enters the cyclone D3 where the solid particles are separated and collected into the tank D4.

The atomizer nozzle is two-fluid kind:

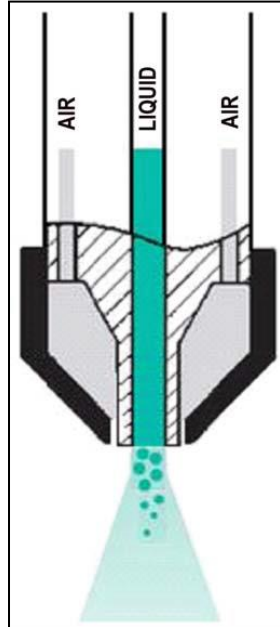


Fig. 1.2: Two-fluid nozzle

The liquid is pumped through a hole (0.7 mm for the standard nozzle) at which output it is mixed with compressed air, produced by the compressor P2, creating a spray of very small droplets.

The nozzle is provided with a pneumatic cleaning needle controlled with a timed solenoid valve.

The system is equipped with a “touch screen” panel enabling to check the drying cycle in manual or automatic mode.

In particular the panel can be used to:

- Switch on and off the feed pump, the compressor, the blower and the nozzle cleaning system
- Check the air inlet temperature
- Adjust the feed pump flow rate, the air flow rate and the nozzle cleaning frequency
- Display the air outlet temperature

Procedure

Operating:

- Fill a container with the product to be dried and another one with distilled water
- Insert the suction pipe of the peristaltic pump into the container with distilled water
- Use the touch screen to set the air inlet temperature, the blowers speed, the feed pump and the nozzle cleaning frequency as follows:
 - **Production of powdered coffee**
200 g of instant coffee (Nescafé) are mixed with 300 g of distilled water obtaining a solution at the 40%
- The following operative parameters are set:
 - Inlet temperature = 180 °C
 - Blower speed = 80%
 - Pump speed = 50%
 - Cleaning frequency = 10 sec
- Press “START” on the “AUTO MENU” window (i.e. the one with the diagram of the installation).
- When the air outlet temperature is stabilized, transfer the suction pipe of the peristaltic pump into the container with the solution.
- When the product reaches the nozzle, the drying operation is started and it is possible to observe the powdered product going in a descending spiral into the cyclone toward the collection tank.
- To stop the system you must press the “STOP” inscription on the “AUTO MENU” window (i.e. the one with the installation diagram).

Cleaning:

- **Be careful:** at the end of the process the glass parts are extremely hot. Wait for the drying chamber and the cyclone to cool down.
- It is advisable to employ two persons to dismount the glass parts
- When the glass parts are cooled down, remove the product collection tank opening the tightening flange (9)
- Remove the bottom tank of the drying chamber opening the tightening flange (11)
- Loosen the ring nut (7) and remove the cyclone opening the tightening flange (6)

- Remove the drying chamber opening the tightening flange (2)
- Switch the unit off (pushing the green pushbutton) and disconnect it from the water mains.
- The dismantled glass parts can be cleaned, in most cases, rinsing them with a lot of water and using a brush, if necessary.
- At the end of each experiment, clean the external part of the nozzle and the surrounding area with a cloth moistened with water

Figures and Calculations

- Plot inlet and outlet air temperatures as a function of operating time.
- Find the percent recovery of the solid based on total solid input.
- Find the percent loss and percent yield of the solid based on total solid input.
- What are the advantages and the disadvantages of spray drying?
- What are the parameters affecting the Spray drying operation?

EXPERIMENT 2

TRAY DRYER

Introduction

A large number of manufacturing processes require the drying of a product or material. The type of equipment used in these industrial operations depends on the particular process and characteristics of the material being dried. For example, the value of many food products is high and so is the requirements for its processing are more stringent than for agricultural fertilizer materials. The design of driers for these purposes will be dictated by the relative importance of such factors as heat sensitivity, porosity, bulk density and particle size of the dried material. In fact, since the dried solid is generally a valuable product, its shape, color, stability, stickiness and hence its overall sale ability, all depend upon the drying processes to which it has been subjected.

Tray drying is essentially a semi-batch operation with stationary product and a continuous airflow. Batch operations are expensive as they are labor intensive and the operation scale is typically small.

Drying involves the transfer of liquid from a wet solid to an unsaturated gas phase such as air, and the solid itself can exert a considerable influence on the drying process.

Equipment Diagrams

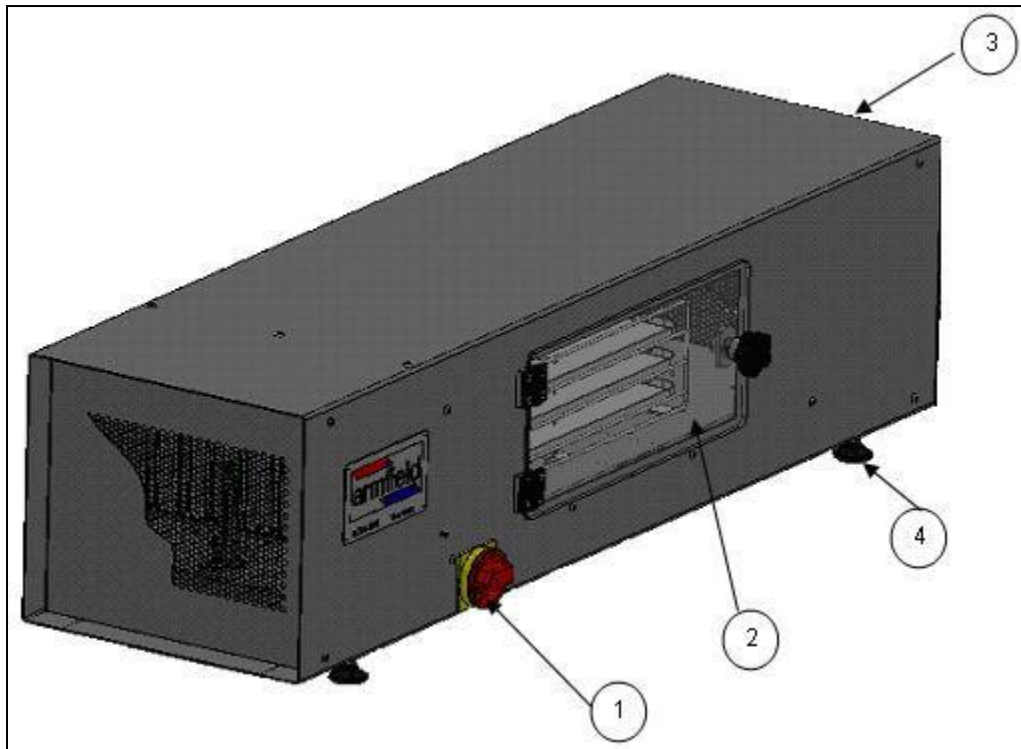


Figure 2.1: UOP8 MKII Tray Drier

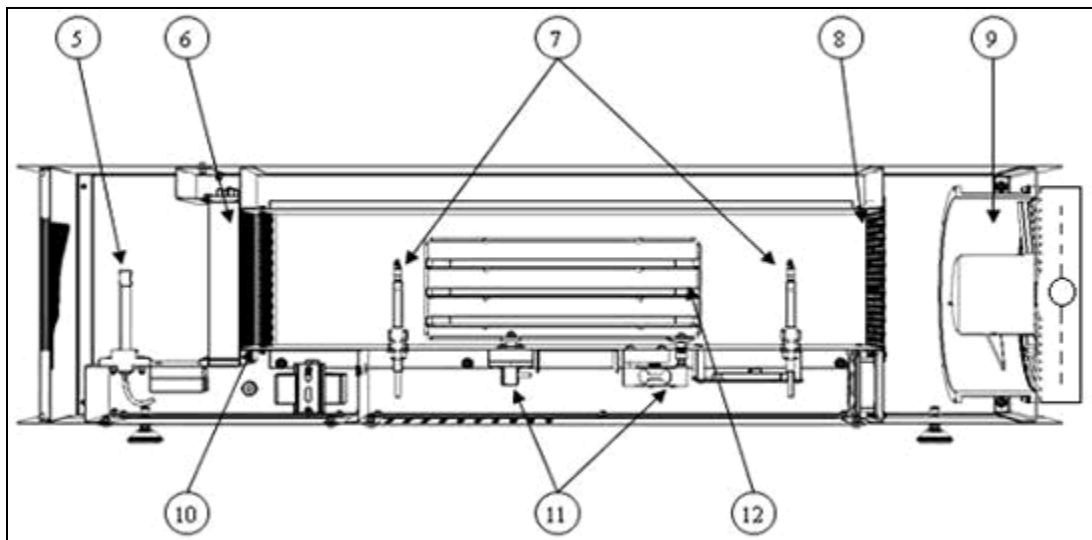


Figure 2.2: Section of UOP8 MKII

The UOP8MK-II comprises a square section air duct fabricated from stainless steel with variable speed fan, and removable trays for solids drying. Air is drawn into the duct by an electronic motor driven axial fan impeller (9).

The air flow velocity can be manipulated through a combination of motor speed variation and use of an adjustable louvre (3).

The air entering the duct passes over an electric heating element (6) to heat the air to temperatures up to 80°C at low air velocity. The temperature of the air can be easily controlled through a PID controller built into the UOP8 MKII Armsoft® software which varies the electrical supply to the heating element.

There are three, removable trays that can be loaded with material for drying in the central section of the duct. The trays are loaded into the duct on a support frame (12) so that air can freely pass over the drying material in the trays.

The support frame sits on three load cells (11) which are used to determine the change in weight of the material as it dries.

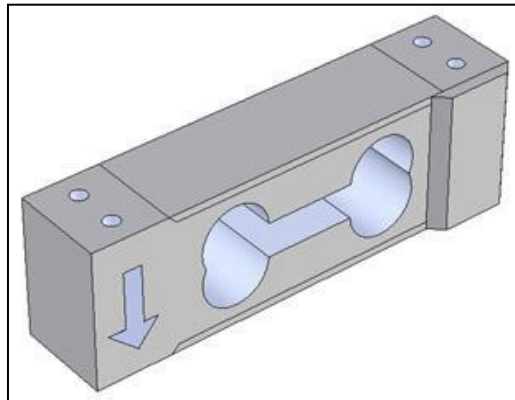


Figure 2.3: Load Cell

The trays are inserted and removed from the duct through a latched side door (2). This side door is an acrylic panel to allow visual inspection of the drying trays. Feet (4) on the bottom of the duct can be adjusted to ensure the duct is level.

After passing over the drying trays, the air is discharged to atmosphere through an outlet duct section. The air flow rate is determined using an air velocity transmitter (5) which is located at the air inlet.

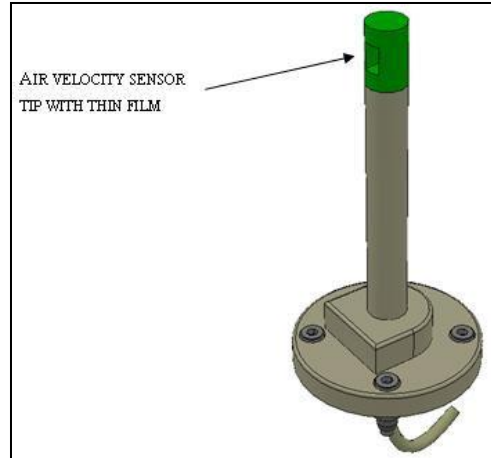


Figure 2.4: Air velocity sensor

Air (dry bulb) temperature and relative humidity are continuously measured at two points (7); before and after the trays.

The wet bulb temperature can be calculated from Equation 1 or can be found on the Psychrometric Chart in the Laboratory Teaching Exercises.

$$\text{Relative Humidity}(\%) = \frac{\text{Wet Bulb Temperature}}{\text{Dry Bulb Temperature}} \quad \text{Equation (2.1)}$$

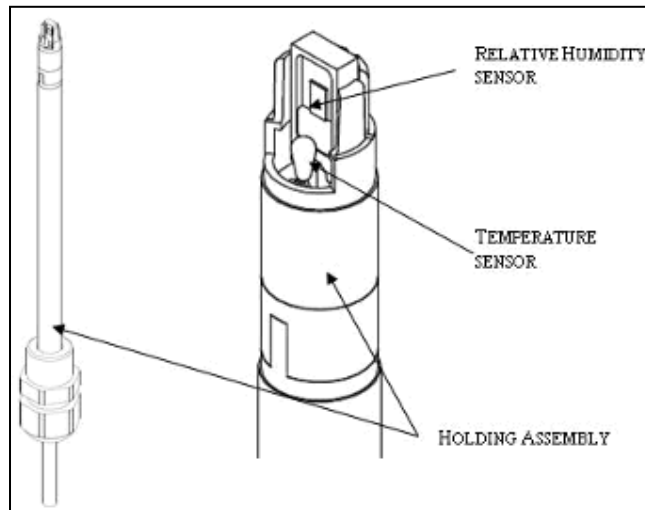


Figure 2.5: Temperature/Relative Humidity (T/RH) sensor block

Cooling fans in the electrical compartment will operate to minimise rise in the components during operation.

The equipment needs to be connected to a suitable PC (not included) to allow remote control and data acquisition with the UOP8 MKII Armssoft® software.

Exercise A

Objective

To produce a drying and a drying rate curve for a wet solid being dried with air of fixed temperature and humidity.

Theory

Immediately after contact between the drying media (wet solid) and the drying medium (hot air), the solid temperature adjusts until it reaches a steady state. If the solid is initially very wet the surface is essentially covered in a thin film of liquid which is considered to be unbound moisture. The solid temperature and the rate of drying may increase or decrease to reach the steady state condition. At steady state, the temperature of the wet solid surface is the wet bulb temperature of the drying medium. Temperatures within the drying solid also tend to equal the wet bulb temperature of the air. However, lag between the movement of mass and heat result in some deviation. Once the media temperatures reach the wet bulb temperature of the air, the temperatures of the media and air become stable and the drying rate remains constant. This is the “constant rate drying”. The moisture is transported to the surface of the media by capillary forces and drying is limited only by the rate at which the heat is supplied. This period ends when the solid reaches the critical moisture content. The surface film of moisture over the solid has been reduced by evaporation to a point where any further drying causes dry spots to appear on the solid surface. Beyond the critical moisture content the surface temperature of the solid rises and the drying rate falls off rapidly. This is the “falling rate” period and can last for a significantly longer time than the constant rate period. This holds true even though the moisture removal may be less. The drying rate approaches zero as the moisture content reaches equilibrium. This is the lowest moisture content obtainable with the solid under the drying conditions used.

Equipment Set-Up

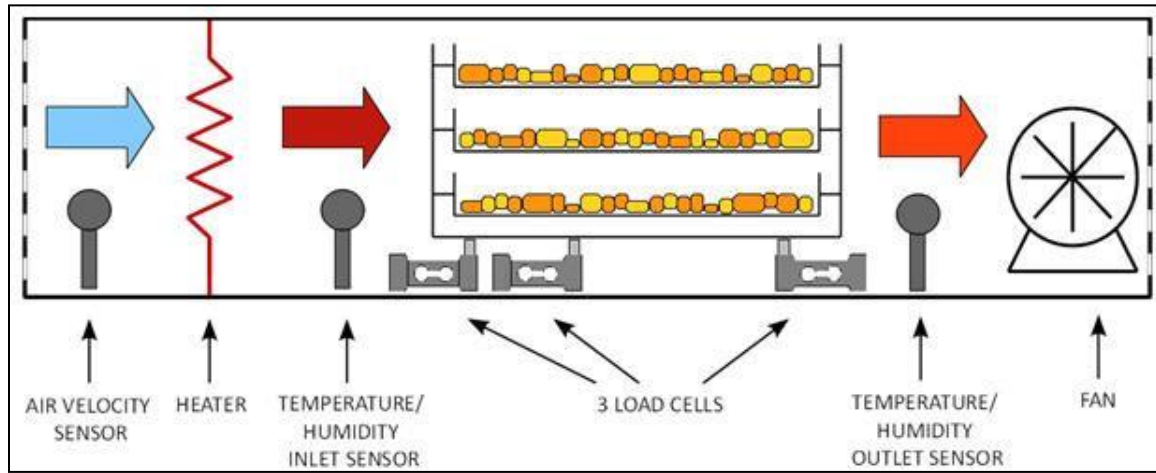






Figure 2.6: Schematic of UOP8 – MKII

Procedure

Start-up (Pre-Heating & Zeroing load cells):

1. Make sure the unit is switched off and remove the three drying trays from the UOP8-MKII and set aside.
2. Turn on the UOP8-MKII tray drier using the main switch on the drier and also by clicking the “power on” button (so that it appears as ) on the Armsoft software.
3. Adjust the fan speed and louvre as required to achieve an inlet air velocity of 0.6m/s.
4. Note the temperature of T1 on the mimic diagram and enter this temperature as the ambient air temperature by clicking on the  button on the mimic diagram.
5. Open the heater PID () and set to automatic with a set point of 55°C. Be aware that you must choose the option Automatic from the PID menu. Check that the preheat temperature sensor rises then stabilizes approximately at the set point temperature.





Note: Load cells typically take 15 minutes to reach operating temperatures. During this time step 1 of the experiment can be carried out.

6. After 15 minutes, change the option mode on the PID heater to Manual and return the empty drying trays onto the tray holder in the UOP8-MKII drier and zero each load cell by clicking each zero button () on the Armsoft software. All weight readings should now equal approximately 0.0 g.




Note: Take care when returning drying trays into the UOP8-MKII drier as the internals may be hot and harmful to touch.

- Is important to return the Heater control to Manual operation whilst the side door opens in order to protect the initial conditions set on the tray.
- 7. Immediately after zeroing the load cells, remove the three drying trays from the drier.
- 8. Ensure the side door is closed.

The UOP8-MKII drier is now ready to begin the experiment:

1. Sufficient pumice (or another appropriate drying media) to fill the 3 trays to a depth of 4.5 – 6.5mm (approximately 150-200g of 1.4 – 3mm pumice grit per tray) should submerged and soaked in water (typically ten minutes soaking in water is a sufficient length of time).
2. Remove the pumice from the water and drain using a strainer to remove excess ‘free’ water.
3. Evenly distribute the wet pumice onto each drying tray.
4. Adjust the sampling configuration by clicking on the  button from the top menu and set the sampling operating to automatic, and set the sample interval to 2 minute intervals.
Note: If this is not the first set of results recorded during this session, ensure to begin a new set of results in the data logger by clicking the  button from the top menu.
5. Place the loaded drying trays on to the tray holder and lock the side door closed.
Note: Take care when placing the drying trays into the UOP8-MKII drier as the internals may be hot and harmful to touch.
6. Record the total weight indicated (material and water).
7. Change the operation back to Automatic on the Heater PID menu.
8. After only a few minutes the weight readings should stabilise. Then select the  icon to begin the data recording of the sensor readings into the results table.
9. Set up the graph to plot the total weight vs. time by clicking the  button when viewing the GraphForm and selecting “Time Elapsed” in the X-Axis Content drop down menu and “Total Weight (Wt) [g]” on the Primary Y-Axis.

Note:

- The results Table page can be viewed by clicking on the  button on the top menu.
- The GraphForm page can be view by clicking on the  button on the top menu.
- To return to the mimic diagram, click on the  button on the top menu.

10. The end of drying occurs when there is no change in the total weight for 5 – 10 minutes.

Note: Typically this experiment takes approximately one hour.

- 11.** Switch the UOP8-MKII drier off using the main switch on the drier.
- 12.** When end of drying has been confirmed from the graph select the icon to stop recording data.
- 13.** Set the heater PID to “OFF” to begin cooling the equipment. When T2 reaches room temperature set the fan speed to 0%.
- 14.** Switch the UOP8-MKII drier off using the main switch on the drier.
- 15.** Remove the trays from the tray holders and return the dried pumice.
- 16.** Return the trays back onto the tray holders and lock the side door closed.

Results

$$X_E^1 = \frac{\text{weight of liquid}}{\text{weight of dry solid}} = \frac{\text{weight of wet solid} - \text{weight of dry solid}}{\text{weight of dry solid}} \quad \text{Equation (2.2)}$$

$X_E^1 = \text{Equilibrium Moisture content}$

Using Equation (2.2), calculate the moisture content removed from the solid.

Plot the drying curve relating moisture content as a function of time. On the secondary axis plot the temperature after the trays (T_2).

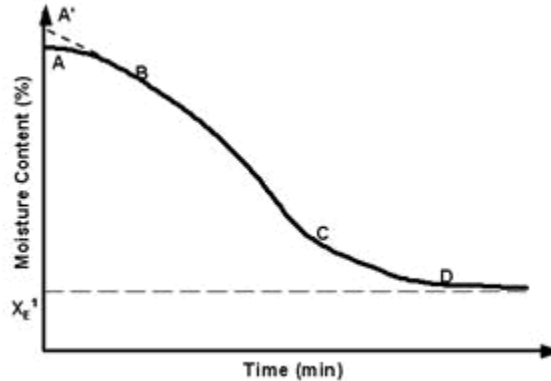


Figure 2.7: Typical Drying Curve

Produce the drying rate vs. moisture content curve by differentiating the data from the drying curve. Identify the points A, B, C and D as shown on the examples for a drying curve and drying rate curve. These points are where the drying passes from one regime to the next.

$$\text{Drying Rate (kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}) = -S_s \frac{\Delta X}{\Delta t} \cdot \frac{1}{A} \quad \text{Equation (2.3)}$$

Where:

S_s (kg) = Mass of dry solid

ΔX = Change in moisture content

Δt (h) = Time period

A (m^2) = Drying area over which the gas blows

The dimensions for each tray are 254mm x 304.8mm x 9.5mm.

Conclusion

Comment upon the results obtained and relate the curves to the mechanism by which drying occurs. What is the significance of the equilibrium moisture content?

If results did not reflect that which the theory suggests, what may have caused this discrepancy?

When the “critical moisture content” occurs, what happens to T_2 ?

Exercise C

Objective

To investigate the influence of air velocity on the drying rate of a wet solid being dried with air of fixed temperature and humidity.

Theory

Once the media temperatures reach the wet bulb temperature of the air, the temperatures of the media and air become stable and the drying rate remains constant. This is the “constant rate drying” period. The moisture is transported to the surface of the media by capillary forces and drying is limited only by the rate at which the heat is supplied. It is shown in Equation C1 that the drying rate during the constant rate period is proportional to the heat transfer coefficient and the difference between the dry bulb temperature of the air and the wet bulb temperature at the solid surface. The heat transfer coefficient is dependent on the velocity of the air passing over the media. As velocities increase, the rate of evaporation of moisture from the surface of the solid also increases along with the mass transfer coefficient of moisture transported to the solid surface. Hence, higher air velocities give a higher drying rate during the constant rate drying period. This period ends when the solid reaches the critical moisture content.

$$N_c \propto h_c(T_v - T_l) \quad \text{Equation (2.4)}$$

Where:

N_c = drying rate during constant rate period

h_c = total heat transfer coefficient (essentially convective)

T_v = temperature of the drying gas

T_l = temperature of the liquid/gas interface

If radiation and conduction through the solid are insignificant as is the case with tray drying then Equation 2.4 becomes:

$$N_c \propto G^{0.71} \quad \text{Equation (2.5) (Treybal, 1980)}$$

Where:

G = air mass velocity.

Equipment Set-Up

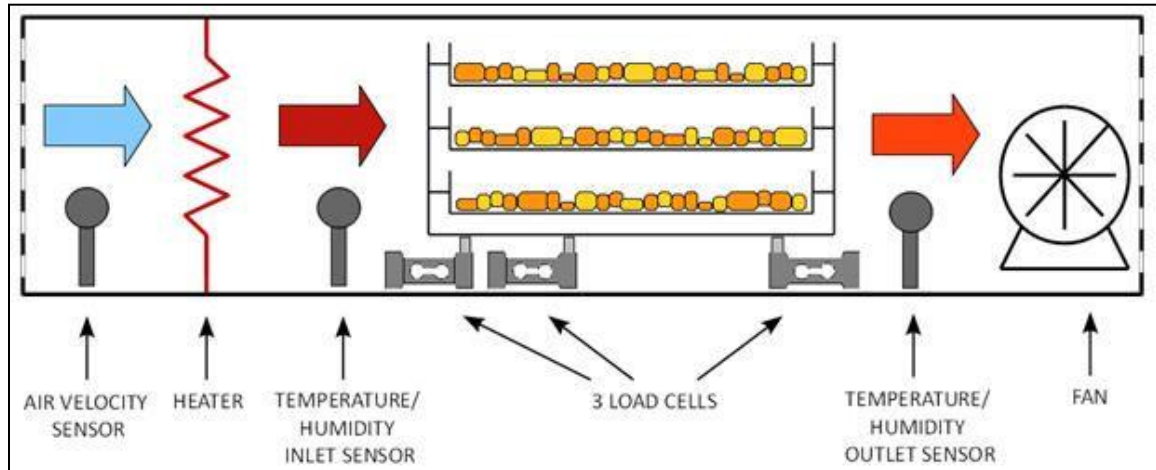






Figure 2.8: Schematic of UOP8 – MKII


Procedure


Start-up (Pre-Heating & Zeroing load cells):

1. Make sure the unit is switched off and remove the three drying trays from the UOP8-MKII and set aside. Leave the support tray inside the unit.
2. Turn on the UOP8-MKII tray drier using the main switch on the drier and also by clicking the “power on” button (so that it appears as ) on the Armsoft software.
3. Adjust the fan speed and louvre as required to achieve an inlet air velocity of 1.2 m/s.
4. Note the temperature of T1 on the mimic diagram and enter this temperature as the ambient air temperature by clicking on the  button on the mimic diagram.
5. Open the heater PID () and set to automatic with a set point of 40°C. Be aware that you must choose the option *Automatic* from the PID menu. Check that the preheat temperature sensor rises then stabilises at approximately the set temperature.
Note: Load cells typically take 15 minutes to reach operating temperatures. During this time step 1. of the experiment can be carried out.
6. After 15 minutes, change the option mode on the PID heater to *Manual* and return the empty drying trays onto the tray holder in the UOP8-MKII drier and zero each load cell by clicking each zero button () on the Armsoft software. All weight readings should now equal approximately 0.0 g.
Note: Take care when returning drying trays into the UOP8-MKII drier as the internals may be hot and harmful to touch.

- It is important to return the Heater control to *Manual* operation whilst the side door opens in order to protect the initial conditions set on the tray.
7. Immediately after zeroing the load cells, remove the three drying trays from the drier.
 8. Ensure the side door is closed.

The UOP8-MKII drier is now ready to begin the experiment:

1. Sufficient pumice (or another appropriate drying media) to fill the 3 trays to a depth of 4.5 – 6.5mm (approximately 150-200g of 1.4 – 3mm pumice grit per tray) should be submerged and soaked in water (typically ten minutes soaking in water is a sufficient length of time).
2. Remove the pumice from the water and drain using a strainer to remove excess ‘free’ water.
3. Evenly distribute the wet pumice onto each drying tray.
4. Adjust the fan speed and louvre to achieve an inlet air velocity of 1.5 m/s.
5. Adjust the sampling configuration by clicking on the  button from the top menu and set the sampling operating to automatic, and set the sample interval to 2 minute intervals.

Note: If this is not the first set of results recorded during this session, ensure to begin a new set of results in the data logger by clicking the  button from the top menu.

6. Place the loaded drying trays on to the tray holder and lock the side door closed.
Note: Take care when placing the drying trays into the UOP8-MKII drier as the internals may be hot and harmful to touch.
7. Record the total weight indicated (materials and water)
8. Change the operation back to *Automatic* on the Heater PID menu.
9. After only a few minutes the weight readings should stabilise. Then select the icon to begin the data recording of the sensor readings into the results table.
10. Set up the graph to plot the total weight vs. time by clicking the button when viewing the GraphForm and selecting “Time Elapsed” in the X-Axis Content drop down menu and “Total Weight (Wt) [g]” on the Primary Y-Axis.

Note:

The results Table page can be viewed by clicking on the button on the top menu.

The GraphForm page can be viewed by clicking on the button on the top menu.

To return to the mimic diagram, click on the button on the top menu.

11. The end of drying occurs when there is no change in the total weight for 5 – 10 minutes.

Note: Typically this experiment takes approximately one hour.

12. When end of drying has been confirmed from the graph select the icon to stop recording data.
13. Repeat steps 1-10 setting air velocities in step 4 to 2.0m/s and 2.5m/s for respective runs. It is important that the mass of media (4.5 - 6.5mm) and drier temperature be the same as those in previous runs during this experiment.
14. Set the heater PID to “OFF” to begin cooling the equipment. When T2 reaches room temperature set the fan speed to 0%.
15. Switch the UOP8-MKII drier off using the main switch on the drier.
16. Remove the trays from the tray holders and return the dried pumice.
17. Return the trays back onto the tray holders and lock the side door closed.

Results

$$X_E^1 = \frac{\text{weight of liquid}}{\text{weight of dry solid}} = \frac{\text{weight of wet solid} - \text{weight of dry solid}}{\text{weight of dry solid}} \quad \text{Equation (2.6)}$$

Using Equation 2.6, calculate the moisture content removed from the solid.

Plot the drying curve relating moisture content as a function of time for each test on the same graph. On the secondary axis plot the temperature after the trays (T2).

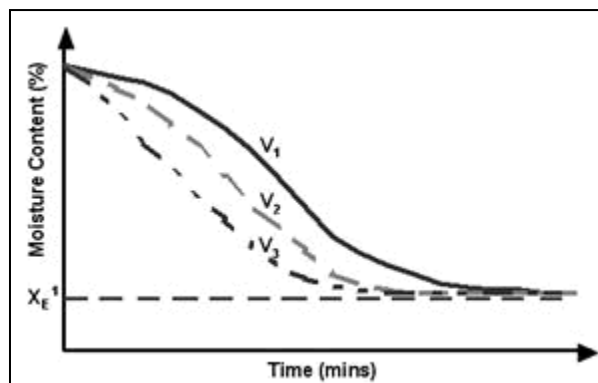


Figure 2.9: Typical Drying Curve for Different Air Velocities

X_E^1 = Equilibrium moisture content

Produce the drying rate vs. moisture content curve by differentiating the data from the drying curve.

$$\text{Drying Rate (kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}) = -S_s \frac{\Delta X}{\Delta t} \cdot \frac{1}{A} \quad \text{Equation (2.7)}$$

Where:

S_s (kg) = Mass of dry solid

ΔX = Change in moisture content

$\Delta t(\text{h})$ = Time period

A (m²) = Drying area over which the gas blows

The dimensions for each tray are 254mm x 304.8mm x 9.5mm.

Figure 2.10 is an example of an ideal drying rate curve for 3000 μm and 300 μm
 X_C = Critical moisture content

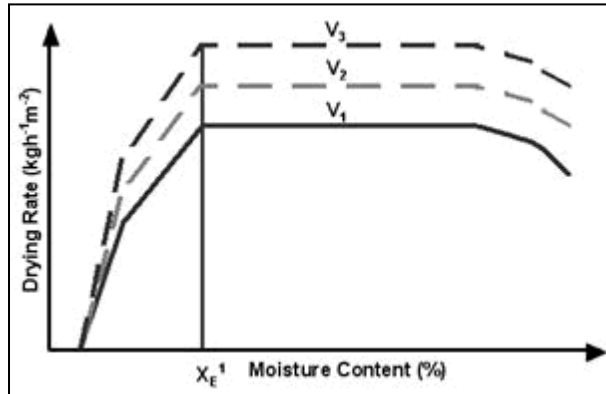


Figure 2.10: Typical Drying Rate Curve for Different Air Velocities

Due to noise and accuracy of the weight measurements some smoothing of results may be required to give a clear curve.

Conclusion

Comment upon the results obtained, noting in particular the relationship between air flow rate and drying rate during the constant rate period.

If results did not reflect that which the theory suggests, what may have caused this discrepancy?

Was the observed relationship predicted by the theoretical analysis?

Did the air velocity have such a direct influence during the falling rate period?

Did the critical moisture content or the equilibrium moisture content (X_E^1) exhibit any dependency on the air velocity?

Suggest physical explanations for the observed results.

EXPERIMENT 3

BATCH DISTILLATION

Theory

Batch distillation refers to the use of distillation in batches, meaning that a mixture is distilled to separate it into its component fractions before the distillation still is again charged with more mixture and the process is repeated.

This is in contrast with continuous distillation where the feedstock is added and the distillate drawn off without interruption.

Batch distillation has always been an important part of the production of seasonal or low capacity and high-purity chemicals. It is a very frequent separation process in the pharmaceutical industry and in wastewater treatment units.

Apparatus

The plant is mounted on a wheeled framework of stainless steel and it consists of a column of borosilicate glass with 7 sieves plates (C1) and temperatures probes equipped with reboiler (E1) including electric heater (J1), and with glass condenser (E2).

The flow rate of the cooling water entering the condenser is measured with a variable area flowmeter (FI1). The product condensed in the condenser (E2) is collected in the tank of distillate (D1) or refluxed into the column, according to whether the solenoid valve (S1) is open or closed.



Figure 3.1: Batch Distillation Unit

The opening and closing times of the solenoid valve can be adjusted with two programmable timers. This plant can be equipped (optional) with a spare column (C2) of borosilicate glass with Raschig rings packing and a bubble cup column of borosilicate glass (C3).

Procedure

➤ Determining the composition of a mixture with the refractometric method

A rapid and accurate method for determining the composition of a binary mixture consists in plotting a calibration line of the index of refraction versus the composition. If the index of refraction of a mixture is known, this straight line will enable to determine its unknown composition.

Consider the example of a water-ethanol mixture and carry out the following operations:

- Measure the indices of refraction of the pure ethanol and of the distilled water
- Prepare 3 ethanol-water mixtures with mole fraction $x = 25\%$; 50% and 75% ; the following equation can be used to determine the volume of water and ethanol having to be mixed in order to obtain a mixture with mole fraction x :

$$x = \frac{\frac{V_{EtOH} \cdot \rho_{EtOH}}{MW_{EtOH}}}{\frac{V_{EtOH} \cdot \rho_{EtOH}}{MW_{EtOH}} + \frac{V_{H_2O} \cdot \rho_{H_2O}}{MW_{H_2O}}} \cdot 100 \quad \text{Equation (3.1)}$$

Where V = volume, ρ = density; and MW = molecular weight

- Close the valves V1, V2 and V5
- Open the valves V3 and V4
- Switch on the automatic/differential switch
- Switch on the digital thermometers
- Fill the reboiler E1 with the mixture to be distilled until the heater J1 is covered up to 3 cm to the end of upper part
- Open the valve V1 partially and adjust the flow rate to 100 l/h on the flow meter FI1
- Turn the switch of the heater J1 available on the switchboard to “1” and turn the respective potentiometer in clockwise direction until the maximum (**heating rate =10**)
- When the vapors condensed in E2 start being refluxed into the column, reduce the power down to 50%
- Set the “ON” and “OFF” timers available on the switchboard at 5 : 2 s for instance
- Distillation can continue until the heater J1 is completely wetted by the mixture; if not, switch off the heaters to avoid heater damage
- When the temperature TI1 is constant in time, that means that the whole volatile component has been distilled or that a maximum azeotrope has been reached; in this case distillation can be stopped
- End the distillation by turning the switches of J1 and S1 to “0”
- After some minutes close the valve V1
- Wait until the temperature in the reboiler E1 has dropped to approximately 40°C , then drain the reboiler and the tanks

- Switch off the digital thermometers
- Record the values of the refractive index versus composition, on a table

Mole Fraction	0%	25%	50%	75%	100%
Index of refraction					

- Plot the index of refraction versus the mole fraction, on a chart, and extract the equation of the calibration line
- Repeat the operation to obtain a calibration line more accurate for low concentration of ethanol in water

Mole Fraction	0%	5%	10%	20%	25%
Index of refraction					

➤ DETERMINING THE EFFICIENCY OF THE COLUMN

The number of theoretical plates N can be calculated with Fenske's equation:

$$N + 1 = \frac{\ln\left(\frac{x_d}{1-x_d} \cdot \frac{1-x_b}{x_b}\right)}{\ln\alpha_{AB}} \quad \text{Equation (3.2)}$$

Where x_d = mole fraction of the more volatile component in the distillate; x_b = mole fraction of the more volatile component in the reboiler.

$\alpha_{AB} = \sqrt{(\alpha_d\alpha_b)}$ = geometric average relative volatility at the temperature of distillate and of bottom product.

Relative Volatility of the Bottom product:

$$\alpha_b = \frac{y_{LKb}/x_{LKb}}{y_{HKb}/x_{HKb}}$$

x_{LKb} : mole fraction of the light key component in liquid state in the reboiler.

y_{LKb} : mole fraction of the light key component in vapour state at equilibrium in the reboiler
(From Appendix A at x_{LKb})

x_{HKb} : mole fraction of the heavy key component in liquid state in the reboiler = $1 - x_{LKb}$.

y_{HKb} : mole fraction of the heavy key component in vapour state at equilibrium in the reboiler
= $1 - y_{LKb}$

Relative Volatility of the Distillate:

$$\alpha_d = \frac{y_{LKd}/x_{LKd}}{y_{HKd}/x_{HKd}}$$

x_{LKd} : mole fraction of the light key component in liquid state in the Distillate.

y_{LKd} : mole fraction of the light key component in vapour state at equilibrium in the distillate
(From Appendix A at x_{LKd})

x_{HKd} : mole fraction of the heavy key component in liquid state in the distillate = $1 - x_{LKd}$.

y_{HKd} : mole fraction of the heavy key component in vapour state at equilibrium in the distillate
= $1 - y_{LKd}$

Efficiency η is the result of the following equation:

$$\eta = \frac{N}{N_R} \cdot 100 \quad \text{Equation (3.3)}$$

Where N_R = actual number of plates (7).

Determining the composition of distillate and of bottom product and knowing the corresponding relative values of volatility enable to calculate the efficiency of the column easily.

- The column is set to work in batch mode, with total reflux, that is the whole condensate is recycled in the column (valve S1 closed); for instance, the reboiler is filled with a 20% (vol) ethanol-water mixture
- Adjust the power so that vapour can bubble on plates regularly
- Take a sample of distillate by opening the solenoid valve S1 for a little and collect 5-10 ml in the tank D2. Measure the index of refraction of this sample and calculate its composition by using the calibration line determined before.
- Repeat the operation on a sample taken from the valve V2.
- It is better to repeat the operations indicated in the last two points at least 4-5 times to calculate an average value of the composition.
- Repeat the whole procedure for different values of heating power

Refractive Index of distillate	1	
	2	
	3	
	4	
	5	
Average value of composition of distillate (%)		
X_{LKD}		
Refractive Index reboiler	1	
	2	
	3	
	4	
	5	
Average value of refractive index of reboiler		
X_{LKB}		
TI1 (°C)		
TI4 (°C)		
α_d		
α_b		
$\alpha_{AB} = \sqrt{\alpha_d \alpha_b}$		
N		
η (%)		

Data Sheet

Mole Fraction	0%	25%	50%	75%	100%
Index of refraction					

Mole Fraction	0%	5%	10%	20%	25%
Index of refraction					

Refractive Index of distillate	1	
	2	
	3	
	4	
	5	
Average value of composition of distillate (%)		
X_{LKD}		
Refractive Index reboiler	1	
	2	
	3	
	4	
	5	
Average value of refractive index of reboiler		
X_{LKB}		
TI1 (°C)		
TI4 (°C)		
α_d		
α_b		
$\alpha_{AB} = \sqrt{(\alpha_d \alpha_b)}$		
N		
η (%)		

Temperature [°C] Time [min]	T1(°C)	T2(°C)	T3(°C)	T4(°C)	T5(°C)	T6(°C)
0						
5						
10						
15						
20						
25						
30						
35						
40						
45						
50						
55						

Figures and Calculations

- Plot the calibration curve for refractive index vs. mole fraction of ethanol for high concentration of ethanol in water.
- Plot the calibration curve for refractive index vs. mole fraction of ethanol for low concentration of ethanol in water.
- Plot temperature values (T1,T2,T3,T4) throughout distillation process vs time on the same graph at each heating rate and reflux ratio.
- Plot temperature values (T5,T6) which represent inlet and outlet temperatures of cooling water in shell and tube heat exchanger vs time on the same graph at each heating rate and reflux ratio.
- Calculate $XLKD$, $XLKB$, $XHKD$, $XHKB$, $YLKD$, $YLKB$, $YHKD$, $YHKB$
- Calculate α_d , α_b , α_{ab}
- Calculate η without rounding the theoretical number of trays.
- η with rounding the theoretical number of trays.
- Determine the values of T_{I1} and T_{I4} which represent the steady state values of T_1 and T_4 (last value in the table) and fill these values in the tables given in the datasheet.
- **NOTE:** these calculations must be repeated for all parts done using the batch distillation apparatus.

Appendix A

Table A.1: Equilibria in Ethanol-Water System at Pressures Less Than Atmospheric; x - liquid mole fraction, y - vapor mole fraction.*

x_1 [mol/mol]	y_1 [mol/mol]
0.01600	0.14700
0.03150	0.25050
0.06000	0.37650
0.08550	0.43000
0.14650	0.50050
0.20600	0.54150
0.23600	0.56000
0.34950	0.59450
0.46750	0.64100
0.48750	0.64250
0.58000	0.68900
0.65250	0.72555
0.70000	0.74950
0.71750	0.76800
0.78900	0.81110
0.84200	0.84880
0.87490	0.87680
0.89670	0.89730
0.94850	0.94400
0.97270	0.96920

* Reference: Baker E.M.: Equilibria in Ethanol-Water System at Pressures Less Than Atmospheric. Ind.Eng.Chem. Ind.Ed. 34 (1942) 1501-1504

EXPERIMENT 4

FIXED AND FLUIDIZED BED APPARATUS

INTRODUCTION

Many important industrial processes rely upon intimate contact between a fluid (liquid or gas) and a granular material (drying, catalytic reactions, ion exchange, combustion etc.) In the traditional technique, the solid is supported on a grid and the fluid is fed through the stationary solid.

At very low flow rates, the fluid will pass between the particles, through the interstices in the bed. The flow will not be even distributed across the bed with preferential flow; this phenomena is called “*channelling*” and the consequences are clear.

In this condition the bed is called “*static beds*”, are simple, robust and cheap but relatively poor from the point of view of the fluid/solid contact.

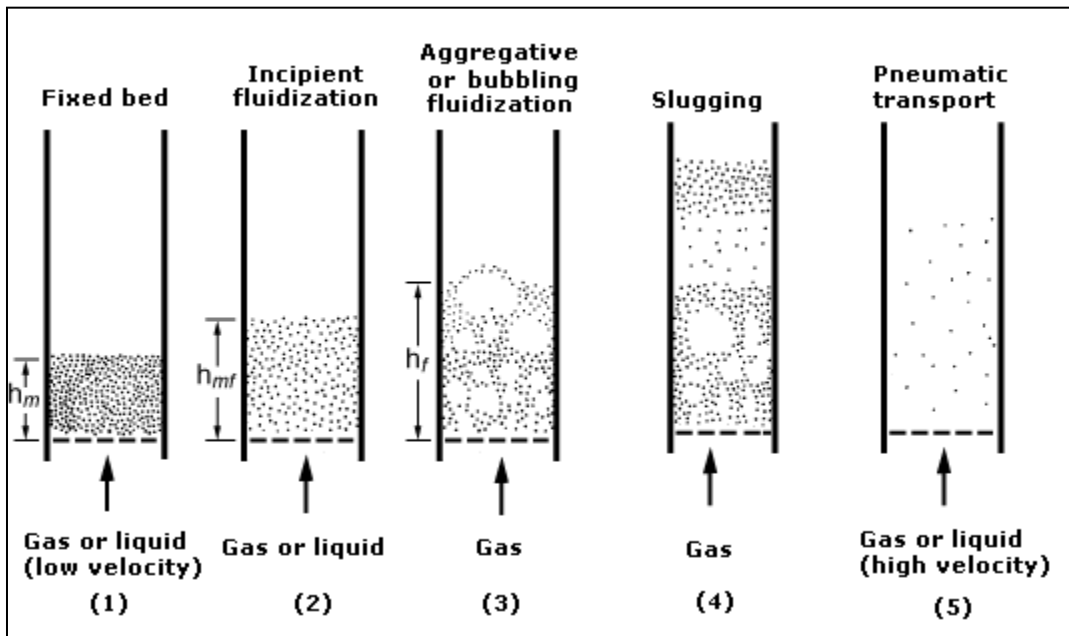


Fig. 4.1:

As the flow rate is increased the force exerted by the fluid on the particles will become sufficient to lift the particle from the bed and to separate it from its neighbours; this phenomena is called “incipient fluidisation” and the bed “*fluidised bed*”.

In this state, there is an immediate improvement of solid/fluid contact, since all the solid surface is available to the fluid.

At this stage, there is little or no relative movement of the particles and the fluidised bed is similar to the previous static bed stretched a little to separate the particles.

A further increase of flow will start a mixing process which can become very violent indeed, such that the whole bed resembles a boiling liquid.

This state represents the real advantage of fluidisation, for the mixing gives very good solid/fluid contact and mass transfer and heat transfer are improved compared to conventional transfer rates.

To this stage the fluidisation process is described as “*dense phase*” and the bed will be contained within the tube and have a readily recognized upper surface.

However, as the fluid flow rate is yet further increased, drag forces will exceed gravitational forces and the particles will be lifted out of the bed; this removal being known as “*elutriation*”. In the extreme, all the solid is displaced and hydraulic transport or dispersed phase fluidization results.

The major fluidisation interest is therefore in the dense phase, good mixing aspect. Where a liquid is the fluidising medium, the particles will remain substantially separate and the bed density, at given velocity, is the same in all section of the bed (uniform fluidization). This is called “*particulate fluidisation*” mode.

Where a gas is the medium, the gas passes somewhat through the fluidised bed as bubble free of solid; the nonuniform nature of the bed was attributed to aggregation of the particles, and the terms “*aggregative fluidisation*” was applied; but there is evidence that the particles stick together, and the term “*bubbling fluidisation*” is a better description of the phenomena.

This mode is less desirable practically and less easy to treat theoretically but it is the typical mode and, therefore, neither easy nor cheap to change.

The two major operational imperfections which are met in fluidised bed work are “channelling” and “slugging”.

The former, essentially dependent upon the solid and fluid properties, originates with the static beds.

Slugging is essentially a function of apparatus design, with the properties of solid and fluid playing a minor role. It is the condition in which bubbles of the fluid agglomerate to approach the diameter of the bed (for ex. small diameter column, deep bed), the solids being pushed up like a piston and then raining down through the bubble or causing a disintegration of the bubble. The phenomenon is most common in gaseous fluidisation - in fact, it is reasonable to say that most problems are far more severe with a gas than a liquid medium.

FLUIDISATION EQUATIONS

The pressure drop required for a fluid to flow in a porous media, like a bed of solid particle, can be calculated using several equations.

The **Ergun equation** that is commonly employed is given below.

$$f_p = \frac{150}{Re_p} + 1.75 \quad \text{Equation (4.1)}$$

Here, the friction factor, f_p , for the packed bed, and the Reynolds number, Re_p , are defined as follows.

$$f_p = \frac{\Delta P}{H} \times \frac{D_p}{\rho u_s^2} \times \frac{\varepsilon^3}{1-\varepsilon} \quad \text{Equation (4.2)}$$

$$Re_p = \frac{D_p u_s \rho}{(1-\varepsilon)\mu} \quad \text{Equation (4.3)}$$

The various symbols appearing in the above equations are defined as follows:

Δp : pressure drop

H = length of the bed

D_p = equivalent spherical diameter of the particle defined by:

$D_p = 6$ (volume of the particle / surface area of the particle)

ρ = density of the fluid

μ = dynamic viscosity of the fluid

u_s = superficial velocity ($u = Q/A$ where Q is the volumetric flow rate of the fluid and A is the cross-sectional area of the bed)

ε = porosity or volume fraction of voids of solid bed (Σ is the ratio of the void volume to the total volume of the bed)

Two simpler results, each obtained by ignoring one or the other term in the Ergun equation also are in use.

One is the **Kozeny-Carman equation**, used for flow under very viscous conditions.

$$f_p = \frac{150}{Re_p} \quad Re_p \leq 1 \quad \text{Equation (4.4)}$$

The other is the **Burke-Plummer equation**, used when viscous effects are not as important as inertia.

$$f_p = 1.75 \quad Re_p \geq 1000 \quad \text{Equation (4.4)}$$

When a fluid flows through a bed of particles in a tube, it will exert a drag force upon the particles resulting in a pressure drop across the bed.

As the fluid's superficial velocity is increased, pressure drop is magnified. In an unrestrained bed that has fluid flowing upwardly through it, a condition will be reached where, with increasing fluid velocity, the drag forces will cause the bed to expand.

This expansion allows the particles to offer less resistance to the fluid flow.

When the drag force is sufficient to support the weight of the particles in the bed, the bed is said to be fluidized. The fluid/solid system shows fluid-like properties, and the bed can be made to flow from one vessel to another.

The pressure drop across the bed, Δp , then remains constant (even with further increase in the fluid velocity) and equal to the effective weight of the bed per unit area:

$$\Delta P = g(1 - \varepsilon)(\rho_p - \rho_f) \cdot h \quad \text{Equation (4.5)}$$

g = gravitational acceleration

ε = porosity or volume fraction of voids of solid bed

ΔP = pressure drop across the bed

ρ_p = density of particles

ρ_f = fluid density

h = length of the bed

As the gas velocity, u , is raised beyond that required to bring the bed to a fluidized condition, u_{mf} , the equation 4.5 becomes:

$$\Delta P = g(1 - \varepsilon_{mf})(\rho_p - \rho_f) \cdot h_{mf} \quad \text{Equation (4.5)}$$

ε_{mf} = minimum porosity at incipient fluidization.

Typically, for a bed of small particles ($D_p \delta 0.1$ mm), the flow conditions at this stage are such that the Reynolds number is relatively small ($Re < 10$) that we can use the Kozeny-Carman Equation, applicable to the viscous flow regime, for establishing the point of onset of fluidization. This yield:

$$u_{mf} = \frac{(\rho_p - \rho_f) \cdot g \cdot D_p^2}{150\mu} \times \frac{\varepsilon_{mf}^3}{1 - \varepsilon_{mf}} \quad \text{Equation (4.6)}$$

When the superficial velocity u is equal to u_{mf} , we refer to the state of the bed as one of **incipient fluidization**. The void fraction, Σ , at this state depends upon the material, shape, and size of the particles.

For nearly spherical particles (small), ε is in the range 0.40 – 0.45 increasing with particle size.

For large particles ($D_p \varepsilon 0.1$ mm), inertial effects are important, and the full Ergun equation must be used to determine u . When in doubt, use the Ergun equation instead of a simplified version of it.

Now, we consider the condition we must impose on the superficial velocity so that particles are not carried out with the fluid at the exit.

This would occur if the superficial velocity is equal to the settling velocity of the particles. Restricting attention to small particles so that Stokes law can be used to calculate their settling velocity, we can write:

$$u_{settl} = \frac{(\rho_p - \rho_f) \times g \times D_p^2}{18\mu} \quad \text{Equation (4.7)}$$

If we now use the result for the minimum fluidization velocity for the case of small particles, given above, we see that the ratio

$$\frac{u_{sett}}{u_{mf}} = \frac{25}{3} \times \frac{1-\varepsilon}{\varepsilon^3} \quad \text{Equation (4.8)}$$

For ε lying in the range 0.40 – 0.45, this yields a ratio ranging from 50 to 78. Since, not all particles are of the same size and that is only an average size, we see that fine particles are likely to be carried out in this situation.

For materials which have no internal porosity, the voidage can be estimated from the density of the solid ρ_p and the density of the bed ρ_b :

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \quad \text{Equation (4.9)}$$

$$\varepsilon_{mf} = 1 - \frac{\rho_{b_{mf}}}{\rho_p} \quad \text{Equation (4.10)}$$

$$\rho_{b_{mf}} = \frac{\text{mass of particle of the bed}}{\text{Volume of the bed at } u_{mf}}$$

OBSERVATION ON SOLID BED

We consider the behaviour of a bed of particles when the upward superficial fluid velocity is gradually increased from zero past the point of fluidization, and back down to zero.

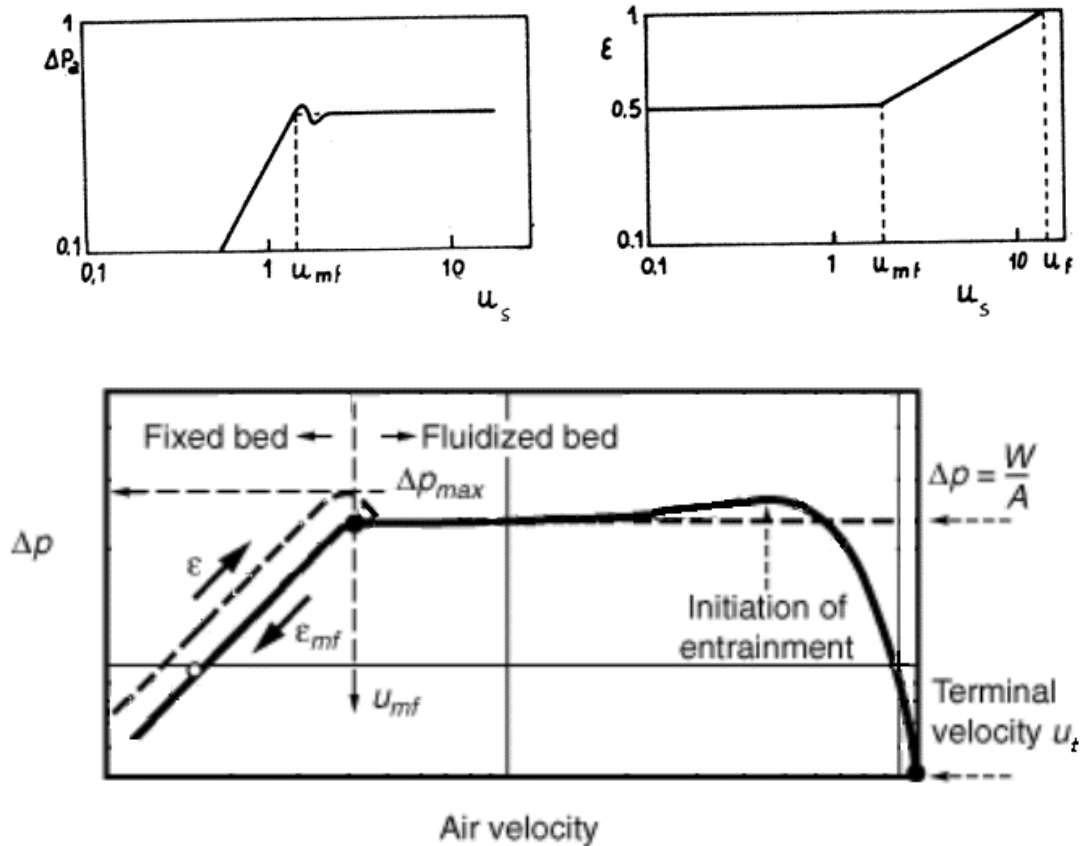


Fig. 4.2

At first, when the flow rate is zero, the pressure drop is zero, and the bed has a certain height. As we proceed along the right arrow (Fig. 4.2) in the direction of increasing superficial velocity, the pressure drop gradually increases while the bed height remains fixed.

This is a region where the Ergun equation for a packed bed can be used to relate the pressure drop to the velocity. When u_{mf} is reached, the bed starts expanding in height while the pressure drop levels off and no longer increases as the superficial velocity is increased. This is when the upward force exerted by the fluid on the particles is sufficient to balance the net weight of the bed (W) and the particles begin to separate from each other and float in the fluid.

As the velocity is increased further, the bed continues to expand in height, but the pressure drop stays constant; in fact, with reference to eq. 4.5, the increasing of h is compensated by a decreasing of ε .

It is possible to reach large superficial velocities without having the particles carried out with the fluid at the exit. This is because the settling velocities of the particles are typically much larger than the largest superficial velocities used.

Now, if we trace our path backward, gradually decreasing the superficial velocity, we find that the behaviour of the bed follows the reverse arrows in the figure.

A bed of particles, left alone for a sufficient length of time, becomes consolidated, but it is loosened when it is fluidized. After fluidization, it settles back into a more loosely packed state; this is why the constant bed height on the return loop is larger than the bed height in the initial state. This effect is called “*hysteresis effect*”.

If we now repeat the experiment by increasing the superficial velocity from zero, we'll follow the lower curve in both directions.

Procedure

A) DETERMINATION OF BED POROSITY

- Weight a small sample of materials (W_b)
- Place the sample in a dry graduated cylinder
- Measure the dry volume of the sample (V_b)
- The bed density (apparent density) is:

$$\rho_b = \frac{W_b}{V_b}$$

- The void fraction is

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p}$$

Where ρ_p is the density of the particle.

Sample	400÷600 μm glass microspheres, $\rho_p = 2.5 \text{ g/ml}$
W_b	105.13 g
V_b	70 ml
ρ_b	$105.13/70 = 1.5 \text{ g/ml}$
ε	$1 - 1.5/2.5 = 0.4$

Sample	250÷300 μm glass microspheres, $\rho_p = 2.5 \text{ g/ml}$
W_b	30 g
V_b	20 ml
ρ_b	$30/20 = 1.5 \text{ g/ml}$
ε	$1-1.5/2.5 = 0.4$

B) FLUIDISATION WITH WATER

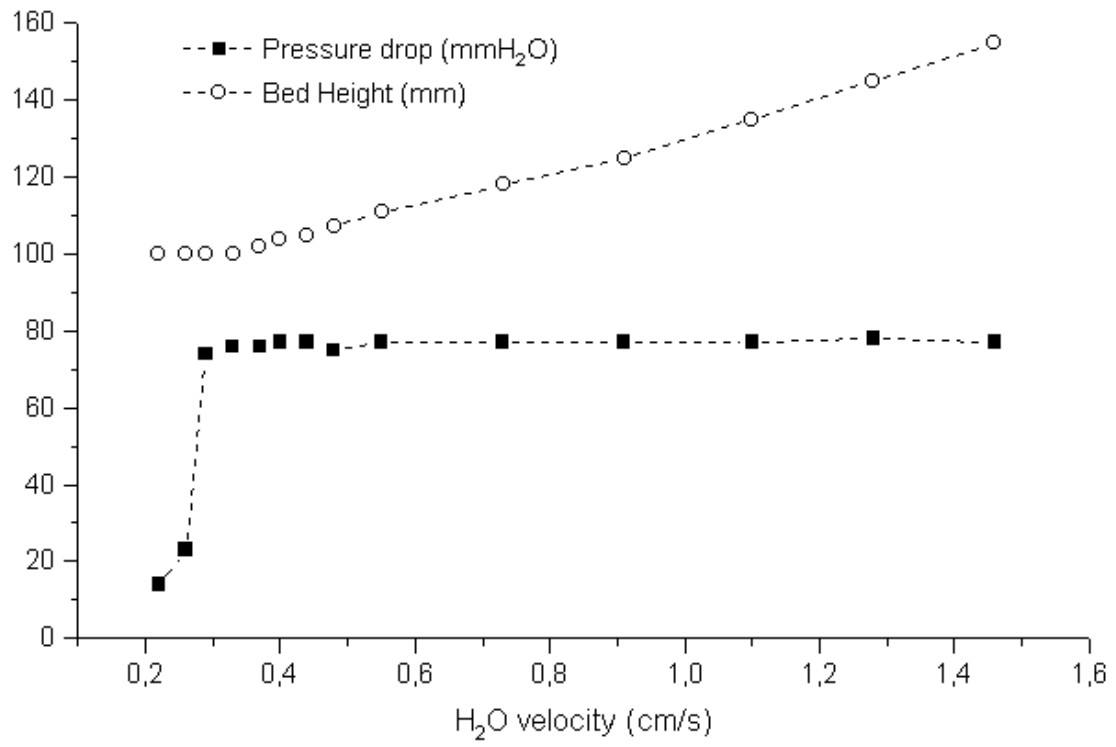
The minimum fluidization velocity U_{mf} can be found plotting the pressure drop through the bed versus the superficial velocity u corresponds to the point on the graph where the line drastically changes slope and begins to become horizontal.

The superficial velocity can be calculated from the flow rate and the cross-sectional area of the bed chamber ($A = 1.52 \cdot 10^{-3} \text{ m}^2$).

Estimation of minimum fluidization velocity gives a good indication of when to use small increments.

Sample	400÷600 μm glass microspheres, $\rho_p = 2.5 \text{ g/ml}$
ρ_b	1.5 g/ml
ε	0.4

Q (l/h)	u (cm/s)	dP (mmwc)	H (mm)	Observations
12				
14				
16				First movement of particles
18				Fluidization
20				
22				
24				
26				
30				
40				
50				
60				
70				
80				

*Fig. 4.3*

C) FLUIDISATION WITH AIR

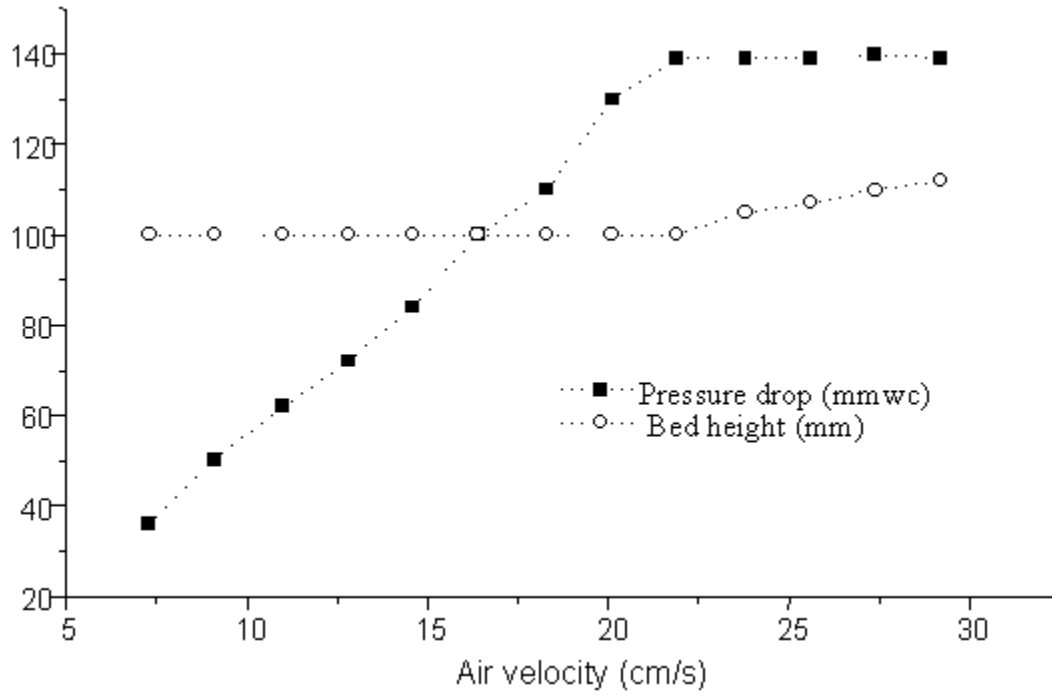
The minimum fluidization velocity u_{mf} can be found plotting the pressure drop through the bed versus the superficial velocity u corresponds to the point on the graph where the line drastically changes slope and begins to become horizontal.

The superficial velocity can be calculated from the flow rate and the cross-sectional area of the bed chamber ($A = 1.52 \cdot 10^{-3} \text{ m}^2$)

Estimation of minimum fluidization velocity gives a good indication of when to use small increments.

Sample	400 ÷ 600 μm glass microspheres, $\rho_p = 2.5 \text{ g/ml}$
ρ_b	1.5 g/ml
ε	0.4

Q (l/h)	u (cm/s)	dP (mmwc)	H (mm)	Observations
400				
500				
600				
700				
800				
900				
1000				First movement of particles
1100				
1200				Fluidization
1300				Bubbling
1400				
1500				
1600				

*Fig. 4.4*

Data Sheet**Fluidization with water**

Q (l/h)	u (cm/s)	dP (mmwc)	H (mm)	Observations
12				
14				
16				First movement of particles
18				Fluidization
20				
22				
24				
26				
30				
40				
50				
60				
70				
80				

Fluidization with air

Q (l/h)	u (cm/s)	dP (mmwc)	H (mm)	Observations
400				
500				
600				
700				
800				
900				
1000				First movement of particles
1100				
1200				Fluidization
1300				Bubbling
1400				
1500				
1600				

EXPERIMENT 5

LINEAR HEAT CONDUCTION

Theory

Fourier's law states that, if two faces of a plane with thickness Δx and surface area A show a temperature difference ΔT , the conduction heat flow will be the result of the following equation

$$Q = k \cdot A \cdot \frac{\Delta T}{\Delta x}$$

where k is the thermal conductivity of the material of that plane. Fourier's equation can be used to correlate ΔT and Δx and to compare the theoretical results with the experimental data.

Apparatus

The accessory mod. TE6A/EV has been designed to demonstrate the application of Fourier's equation to a single-dimension system in steady-state conditions.

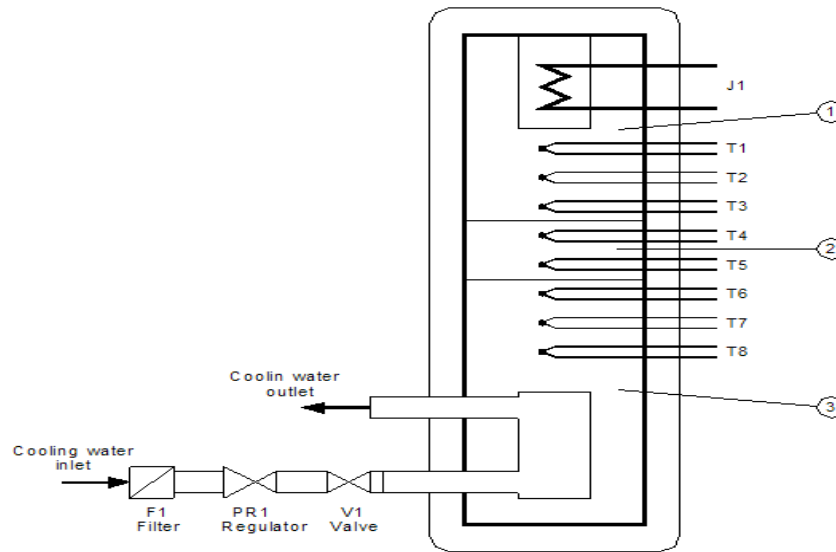


Fig. 5.1: linear heat conduction apparatus

This unit includes a heating section (1) and a cooling section (3) which can be simply clamped together or clamped with interchangeable intermediate sections (2).

The heating section consists of a brass cylinder with diameter of 25 mm with a cartridge-type electric heating element installed at one end; this heating element, powered at low voltage, outputs 60 W at 24 Vdc and it is equipped with a safety thermostat.

The power supplied to the heating element can be controlled by the service unit TE6/EV2. Three K thermocouples (T1, T2 and T3) are positioned along the heating section at uniform intervals of 15 mm to measure the temperature gradient along the section.

The cooling section consists of a brass cylinder with diameter of 25 mm cooled by water flowing through a hollow inside it. Three K thermocouples (T6, T7 and T8) are positioned along the cooling section at uniform intervals of 15 mm to measure the temperature gradient along the section. A pressure regulator (PR1) incorporating a filter will minimize the effect of pressure fluctuations in the cooling water. A valve (V1) allows the flow rate of cooling water to be varied over the operating range of 0-100 litres/h; measurement of this flow rate is not essential, but it can be carried out by an optional sensor connected with the service unit mod. TE6/EV. The heating section, cooling section and all intermediate sections are located co-axially inside plastic housings which minimize heat loss to the surroundings and prevent burns to the operator.

The intermediate sections can be inserted between the cooling and heating sections only one at a time. Using two metallic slides and two clamping knobs will enable to stack the two main sections, with or without intermediate section. The intermediate sections can include two thermocouples at a distance of 15 mm from each other.

This equipment also includes four intermediate sections with different metallic samples:

- Brass: 30mm-long brass cylinder with diameter of 25 mm, fitted with two thermocouples at intervals of 15 mm. The thermal conductivity of brass can be considered as equal to **121 W/m·°C** at the typical operating temperatures of the unit mod. TE6A/EV .
 - Stainless steel AISI 316: 30mm-long steel cylinder with diameter of 25 mm, fitted with two thermocouples at intervals of 15 mm, to demonstrate the effect of a change of thermal conductivity on heat transfer. The thermal conductivity of steel can be considered as equal to **25 W/m·°C** at the typical operating temperatures of the unit mod. TE6A/EV
 - Aluminium: 30mm-long aluminium cylinder with diameter of 25 mm, fitted with two thermocouples at intervals of 15 mm, to demonstrate the effect of a change of thermal conductivity on heat transfer. The thermal conductivity of aluminium can be considered as equal to **180 W/m·K** at the typical operating temperatures of the unit mod. TE6A/EV
-
- Brass: 30mm-long brass cylinder with diameter of 13 mm, fitted with two thermocouples at intervals of 15 mm. The thermal conductivity of brass can be considered as equal to **121 W/m·K** at the typical operating temperatures of the unit mod. TE6A/EV.

Procedure

I . Steady-state heat conduction

- Clamp the heating and cooling sections directly (insert between the two section the plastic ring supplied to reduce thermal dispersion) applying a thin film of thermal paste on the metal/metal interface
- Adjust the voltage to 8 V
- Wait until temperatures stabilize and record their values; a criterion for the steady state could be $d(T1)/dt \leq 0.2 \text{ } ^\circ\text{C}$
- Increase the voltage to 10 V
- Wait until temperatures stabilize and record their values
- Increase the voltage to 12 V
- Wait until temperatures stabilize and record their values

Volt			
Ampere			
T1 (° C)			
T2 (° C)			
T3 (° C)			
T4 (° C)			
T5 (° C)			
T6(° C)			
T7 (° C)			
T8 (° C)			

N.B.: the distance between thermocouples is of 0.015 m

· Calculate:

$$Q = V \cdot I \text{ (W)}$$

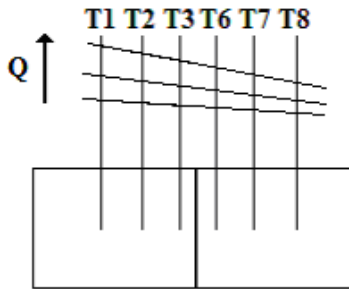
$$\Delta T_{\text{hot}} = T1 - T3$$

$$\Delta T_{\text{cold}} = T6 - T8$$

$$\Delta T_{\text{Intermediate}} = T4 - T5$$

For every steady-state condition, applying Fourier's law, calculates the theoretical values of Δx and compare with the experimental data.

- Compare the values of ΔT_{hot} and ΔT_{cold} for the same value of heat flow
- Compare the values of ΔT_{hot} and ΔT_{cold} for different values of heat flow
- Plot a chart with the temperature values versus the position and carry out a linear interpolation of data



- Observe that temperature profiles are straight lines with angular coefficient directly proportional to the heat flow Q ; calculate the angular coefficient (m) of every straight line and check that:

$$\frac{Q}{m} = \text{constant}$$

II. Demonstrating Fourier's law

- Clamp the heating and cooling sections directly (insert between the two section the plastic ring supplied to reduce thermal dispersion) applying a thin film of thermal paste on the metal/metal interface
- Adjust the voltage to 8 V
- Wait until temperatures stabilize and record their values; a criterion for the steady state could be $d(T1)/dt \leq 0.2 \text{ } ^\circ\text{C}$
- Increase the voltage to 10 V
- Wait until temperatures stabilize and record their values
- Increase the voltage to 12 V
- Wait until temperatures stabilize and record their values.

Volt			
Ampere			
T1 (° C)			
T2 (° C)			
T3 (° C)			
T4 (° C)			
T5 (° C)			
T6(° C)			
T7 (° C)			
T8 (° C)			

N.B.: the distance between thermocouples is of 0.015 m

· Calculate:

$$Q = V \cdot I \text{ (W)}$$

Heat flow

$$A = \frac{\pi D^2}{4}$$

Cross section of the sample

$$\Delta T_{\text{hot}} = T1 - T3$$

Temperature gradient in the heating section

$$k_{\text{hot}} = \frac{\Delta x_{13} \cdot Q}{\Delta T_{\text{hot}} \cdot A_{\text{hot}}}$$

Conductivity in the heating section

$$\Delta T_{\text{int}} = T4 - T5$$

Temperature gradient in the intermediate section

$$k_{\text{int}} = \frac{\Delta x_{45} \cdot Q}{\Delta T_{\text{int}} \cdot A_{\text{int}}}$$

Conductivity in the intermediate section

$$\Delta T_{\text{cold}} = T6 - T8$$

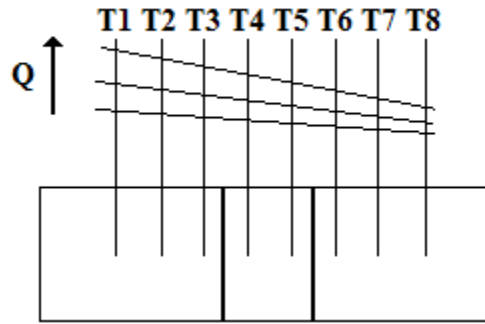
Temperature gradient in the cooling section

$$k_{\text{hot}} = \frac{\Delta x_{68} \cdot Q}{\Delta T_{\text{int}} \cdot A_{\text{int}}}$$

Conductivity in the cooling section

For every steady-state condition

- Compare the values of thermal conductivity of brass for the same value of heat flow
- Compare the values of thermal conductivity of brass for different values of heat flow
- Plot a chart with the temperature values versus the position and carry out a linear interpolation of data.



- Observe that temperature profiles are straight lines with angular coefficient directly proportional to the heat flow Q
- Calculate the average thermal conductivity using the angular coefficient (m) of straight lines and the heat flow Q .

$$k = \frac{Q}{A \cdot m}$$

- Compare the value obtained now with those obtained before for each section

Data Sheet

Volt			
Ampere			
T1 (° C)			
T2 (° C)			
T3 (° C)			
T4 (° C)			
T5 (° C)			
T6(° C)			
T7 (° C)			
T8 (° C)			

Volt			
Ampere			
T1 (° C)			
T2 (° C)			
T3 (° C)			
T4 (° C)			
T5 (° C)			
T6(° C)			
T7 (° C)			
T8 (° C)			

EXPERIMENT 6

RADIAL HEAT CONDUCTION

Theory

The unit mod. TE6B/EV has been designed to study the radial heat conduction in steady-state conditions; it mainly consists of a metallic disk with temperature probes mounted radially from the centre to the periphery.

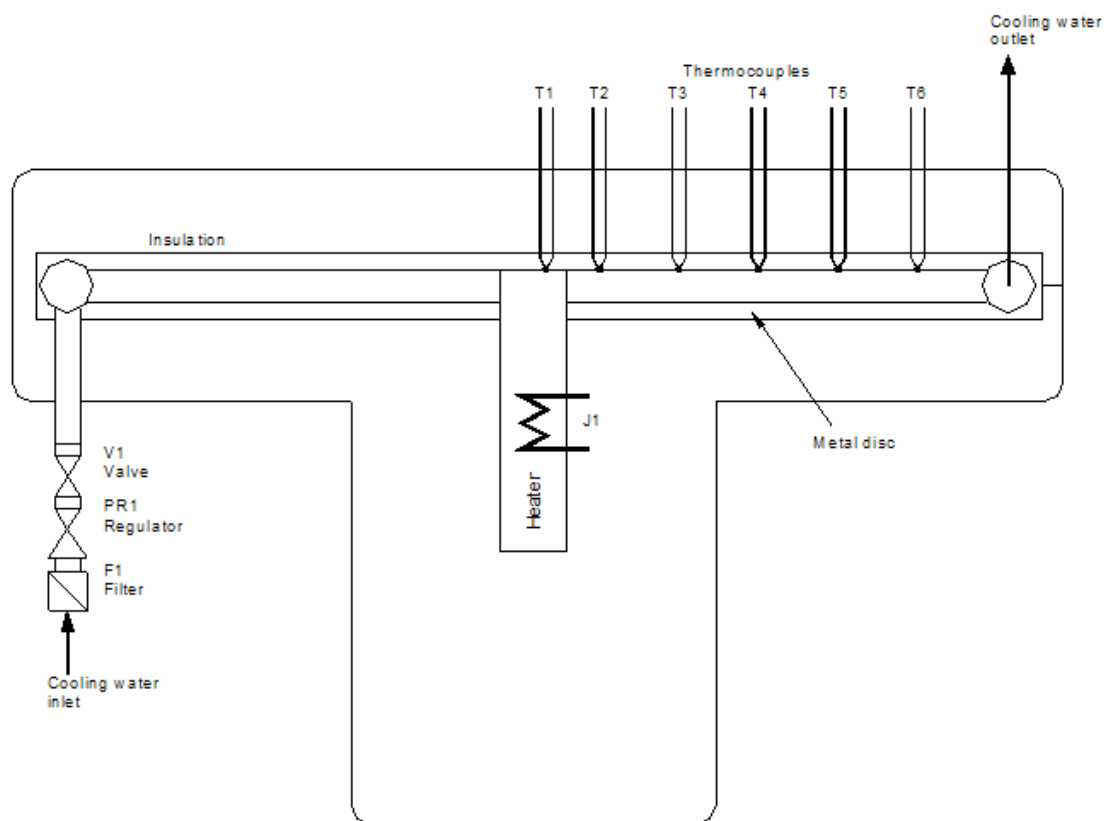


Fig. 1.1: radial heat conduction apparatus

The metallic disk is heated at the centre and cooled at the periphery to create a radial flow of heat by conduction.

The disk is manufactured from brass, is 3.0 mm-thick and it has a diameter of 110 mm and a central copper core (diameter of 14 mm).

The whole specimen is located inside a plastic enclosure to minimize heat loss to the surroundings and prevent burns to the operator.

The cartridge-type electric heating element, powered at low voltage, outputs 100 W at 24 Vdc and it is equipped with a safety thermostat.

The power supplied to the heating element can be controlled by the service unit TE6/EV.

The periphery of the disk is cooled by tap water flowing through a copper tube which is welded to the circumference of the disk.

Six K-type thermocouples (T1, T2, T3, T4, T5 and T6) are positioned at different radii in the heated disk: T1 is placed at a distance of 7 mm from the centre; then the other thermocouples are placed at uniform intervals of 10 mm.

All the thermocouples are positioned on a tangent to the copper cylinder so that disturbances to the heat transfer between thermocouples are minimized.

The distance of thermocouples from the centre is indicated here below:

T1 = 7 mm

T2 = 10 mm

T3 = 20 mm

T4 = 30 mm

T5 = 40 mm

T6 = 50 mm

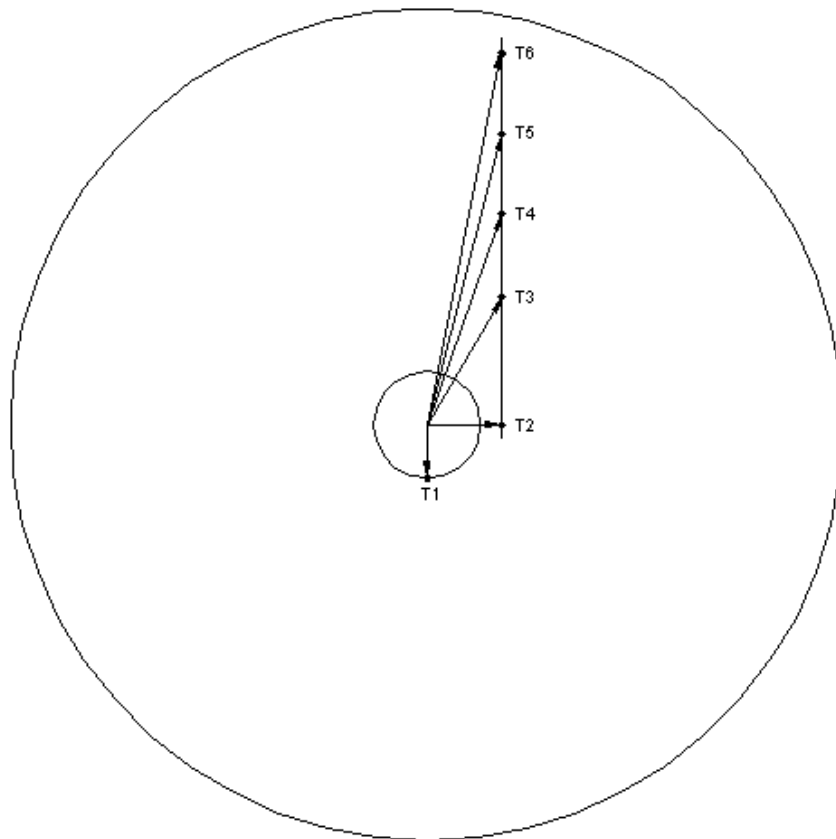


Fig. 1.2: distance of thermocouples from the centre.

A pressure regulator (PR1) incorporating a filter (F1) will minimize the effect of pressure fluctuations in the cooling water.

A valve (V1) allows the flow rate of cooling water to be varied over the operating range of 0-100 litres/H; measurement of this flow rate is not essential, but it can be carried out by an optional sensor connected with the service unit mod. TE6/EV.

The heating section incorporates a safety thermostat; if the voltage is set too high and temperature T1 reaches about 70 °C, the current to the heater will be disconnected until the temperature falls. In this situation, the display shows voltage but no current.

The voltage supplied to the heater should be adjusted so that the temperature T1 is maximum 65°C.

2. OPERATING INSTRUCTIONS

The accessory mod. TE6B/EV can be connected immediately with the unit TE6/E.

- Position the accessory mod. TE6B/EV beside the unit TE6/E on a suitable table
- Insert the thermocouples of unit TE6/EV in the holes along the plate of mod. E6A/EV
- Reduce the output voltage of the unit TE6/EV to zero
- Connect the plug of the heating section with the socket OUTPUT3 available on the rear side of the unit mod. TE6/EV
- Connect the pressure reducer with the water distribution network using the rubber hose and the metallic clamps supplied with the equipment: a flow rate of 100 l/h at a max. pressure of 1 bar is required
- Connect the outlet of the cooling water with a proper drain or with a tank, using the rubber hose and the metallic clamps supplied with the equipment
- Switch on the main LED switch of the unit TE6/EV
- Adjust the flow rate of the cooling water to approximately 100 l/h acting on the pressure reducer (PR1) and valve V1. Using a vessel of known volume and a chronometer will enable to calculate the flow rate:

$$\text{Flow rate} = \text{volume collected (litres)} / \text{time (minutes)}$$

- Shift the temperature selector to the position T1 to display the nearest temperature of the heating section
- Shift the other switch to the position V and adjust the voltage to 8 V, for instance, with the respective knob (voltage control)
- Temperature T1 will rise gradually together with the other temperatures; the behaviour of temperatures in steady-state conditions will be: T1>T2>T3>T4>T5>T6>
- At the end of the test set the voltage control knob to zero

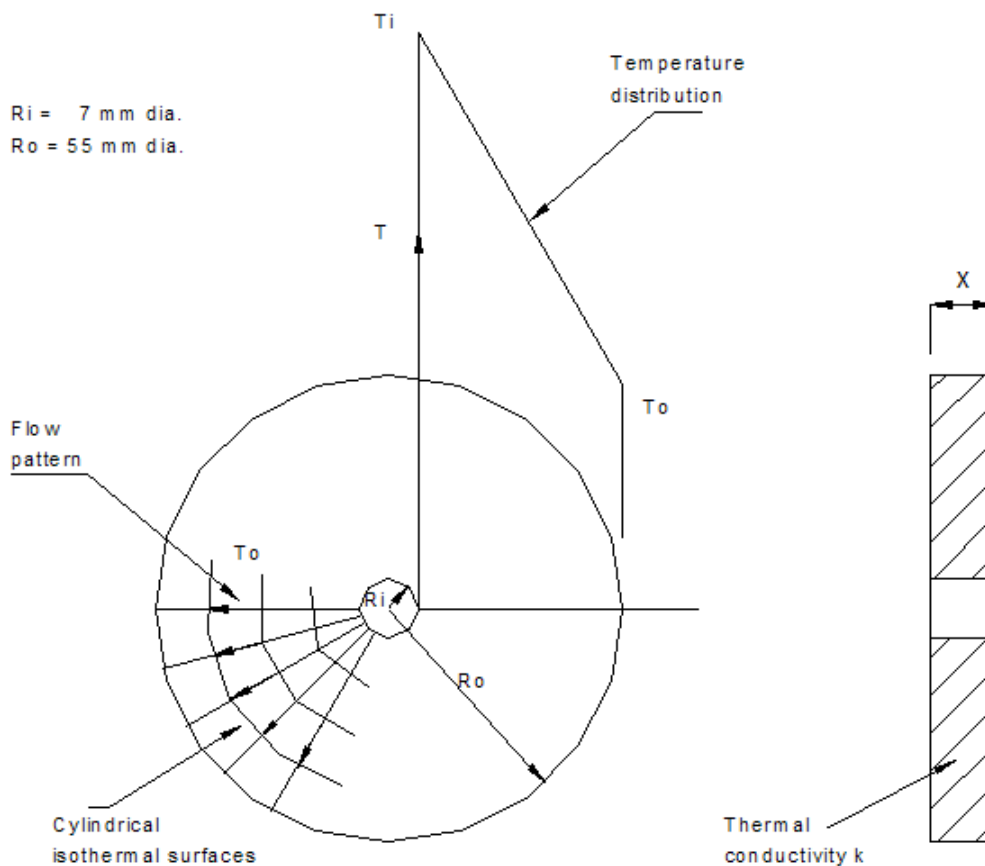
- Wait until the temperature T1 decreases down to the ambient temperature, then close the cooling water
- Disconnect the accessory mod. TE6B/EV from the water distribution network and drain the water from the cooling section

3. EXERCISES

3.1 Radial heat conduction

When the outer and inner surfaces of a cylinder are at different and uniform temperatures, heat flow with radial direction is created.

If the copper disk of the unit mod. TE6B/EV consists of a set of consecutive layers, the radial heat flow crossing each layer must be constant. As the surface area of these layers increases if the radius increases, the temperature gradient will decrease with it.



The surface area of the heat flow in a layer with thickness dR , height L and distance R from the centre, will be: $A = 2 \pi RL$ The radial temperature gradient (perpendicular to the axis of the cylinder) is dt/dR ; applying Fourier's law will obtain:

$$Q = -k(2\pi \cdot R \cdot L) \frac{dT}{dR}$$

As Q does not depend on R , integrating between the position of the thermocouple $T1$, $R1$, and the position of the thermocouple $T6$, $R6$, will obtain:

$$Q \cdot \ln\left(\frac{R6}{R1}\right) = -k(2\pi \cdot L)(T6 - T1)$$

Considering a cylinder of length x and rearranging will lead to the following formula:

$$Q = (2\pi \cdot k \cdot x) \frac{T1 - T6}{\ln\left(\frac{R6}{R1}\right)}$$

or:

$$k = \frac{Q \ln\left(\frac{R6}{R1}\right)}{(2\pi \cdot x)(T1 - T6)}$$

In the case of the unit mod. TE6B/EV, the length of the cylinder is: $x = 3$ mm, with an inner radius $R1 = 7$ mm (position of the thermocouple $T1$) and an outer radius $R6 = 50$ mm (position of the thermocouple $T1$).

The temperature distribution can also be written as:

$$T(R) = T1 - \frac{(T1 - T6)}{\ln\left(\frac{R6}{R1}\right)} \ln\left(\frac{R}{R1}\right)$$

Or

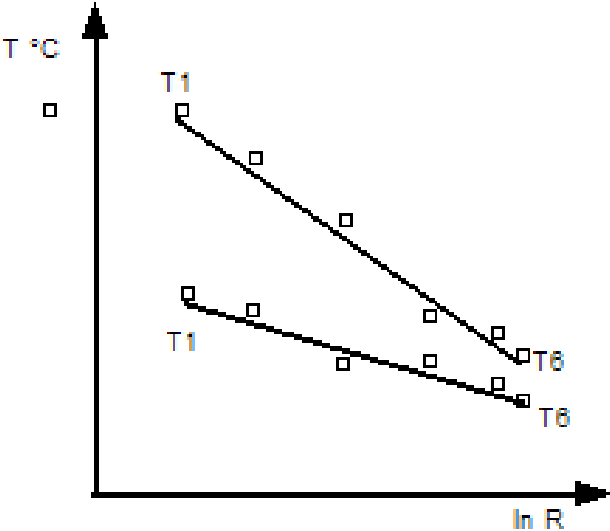
$$T(R) = T1 - \frac{Q}{2\pi \cdot k \cdot x} \ln\left(\frac{R}{R1}\right)$$

- Carry out the operations described in the Chapter 2 and adjust the voltage to 8 V
- Wait until temperatures stabilize and record their values; a criterion for the steady state could be $d(T1)/dt \leq 0.2$ °C
- Increase the voltage to 10 V
- Wait until temperatures stabilize and record their values

- Increase the voltage to 12 V
- Wait until temperatures stabilize and record their values

Volt			
Ampere			
T1 (°C)			
T2 (°C)			
T3 (°C)			
T4 (°C)			
T5 (°C)			
T6 (°C)			
F (l/h)			

- Record the temperature versus \ln of radius for each steady-state condition and extrapolate the temperature at disk circumference ($R = 55 \text{ mm}$) for each value of Q :
 $Q = V \cdot I \text{ (W)}$
- Using the equation 5.4 calculate the thermal conductivity for each value of Q
 The accuracy of the value of k increases when the values of $R1/T1$ and $R6/T6$ are used because the temperature difference is maximum between the extremes of the radius
- Plot a chart of temperature versus \ln of the radius for the various values of Q ; starting from the angular coefficient of the straight line T vs. $\ln(R)$, determine the value of k for each value of Q and compare the resulting values (they would be approximately of $121 \text{ W/m}^\circ\text{C}$)
- Observe that temperature profile is logarithmic and the slope of temperature profiles increases as the heat flow increases.



Conclusions: the test has demonstrated how Fourier’s equation can be applied to a radial heat flow and how the thermal conductivity of a material can be determined if the flow of heat, the thickness of the disk and the temperature in two different radii of the disk are known.

Data Sheet

Volt			
Ampere			
T1 (°C)			
T2 (°C)			
T3 (°C)			
T4 (°C)			
T5 (°C)			
T6 (°C)			
F (l/h)			

Volt			
Ampere			
T1 (°C)			
T2 (°C)			
T3 (°C)			
T4 (°C)			
T5 (°C)			
T6 (°C)			
F (l/h)			

EXPERIMENT 7

COOLING TOWER

THEORY OF WATER COOLING

1.1 Introduction

Many industrial processes use water as cooling medium in the heat exchangers to lower the temperature of process streams; because the costs involved in its provision and disposal, the cooling water is recycled but its temperature must be lowered.

The most common unit used to accomplish this goal is a cooling tower.

1.2 Cooling tower

The general idea of a cooling tower is simple: warm water trickles down the packing in a column and encounters air flowing upwards. A small portion of the water evaporates, requiring latent heat, which causes the decreasing of the temperature of the water (although, when the air temperature is low, there is also some sensible heat transfer to the air).

This process also known as humidification involves the simultaneous transfer of mass and heat.

The packing are used to produce a large water surface area for heat and mass transfer.

Cooling towers fall into two main sub-divisions: “natural draught” and “mechanical draught”.

Natural draught towers depend upon natural convection, due to the difference of density between ambient air and the heated air.

In a mechanical draught tower (the much more widely used), a fan serves to provide a controlled flow of air through the packing; the fan can be located at the top of the tower or at the bottom. Since the air flow is controlled and bigger than in the natural draught towers, this type of towers are considerably more compact.

For a good understanding of water cooling in cooling tower, it is necessary some fundamental concepts of psychrometry reported in the following chapter.

2.PSYCHROMETRY

2.1 Introduction

The capacity of air for moisture removal depends on its humidity and its temperature. The study of relationships between air and its associated water is called psychrometry.

Humidity (Y) is the measure of the water content of the air. The absolute humidity is the mass of water vapour per unit mass of dry air and the units are therefore kg kg⁻¹, and this will be subsequently termed just the humidity.

Air is said to be saturated with water vapour at a given temperature and pressure if its humidity is a maximum under these conditions. If further water is added to saturated air, it must appear as liquid water in the form of a mist or droplets. Under conditions of saturation, the partial pressure of the water vapour in the air is equal to the saturation vapour pressure of water at that temperature.

The total pressure of a gaseous mixture, such as air and water vapour, is made up from the sum of the pressures of its constituents, which are called the partial pressures. Each partial pressure arises from the molecular concentration of the constituent and the pressure exerted is that which corresponds to the number of moles present and the total volume of the system. The partial pressures are added to obtain the total pressure.

The **relative humidity (RH)** is defined as the ratio of the partial pressure of the water vapour in the air (p) to the partial pressure of saturated water vapour at the same temperature (p_s). Therefore:

$$RH = \frac{p}{p_s}$$

The humidity Y can therefore be related to the partial pressure p_w of the water in air vapour by the equation:

$$Y = \frac{18 p_w}{29(P - p_w)} \quad (2.1)$$

where P is the total pressure. In circumstances where p_w is small compared with P, and this is approximately the case in air/water systems at room temperatures, $Y \approx 18p_w/29P$.

Corresponding to the specific heat, C_p , of gases, is the humid heat, C_s of moist air. It is used in the same way as a specific heat, the enthalpy change being the mass of dry air multiplied by the temperature difference and by the humid heat. The units are J .kg⁻¹ °C⁻¹ and the numerical values can be read off a psychrometric chart. It differs from specific heat at constant pressure in that it is based only on the mass of the dry air. The specific heat of the water it contains is effectively incorporated into the humid heat which therefore is numerically a little larger than the specific heat to allow for this.

Wet-bulb Temperatures

A useful concept in psychrometry is the wet-bulb temperature, as compared with the ordinary temperature, which is called the dry-bulb temperature.

The wet-bulb temperature is the temperature reached by a water surface, such as that registered by a thermometer bulb surrounded by a wet wick, when exposed to air passing over it.

The wick and therefore the thermometer bulb decreases in temperature below the dry-bulb temperature until the rate of heat transfer from the warmer air to the wick is just equal to the rate of heat transfer needed to provide for the evaporation of water from the wick into the air stream. Equating these two rates of heat transfer gives:

$$h_c \times A \times (T_a - T_s) = l \times k'gA(Y_s - Y_a)$$

where a and s denote actual and saturation temperatures and humidities; h_c is the heat-transfer coefficient and $k'g$ the mass transfer coefficient from the air to the wick surface; l is the latent heat of evaporation of water.

As the relative humidity of the air decreases, so the difference between the wet-bulb and dry-bulb temperatures, called the wet-bulb depression, increases and a line connecting wet-bulb temperature and relative humidity can be plotted on a suitable chart. When the air is saturated, the wet-bulb temperature and the dry-bulb temperature are identical.

Therefore if $(T_a - T_s)$ is plotted against $(Y_s - Y_a)$ remembering that the point (T_s, Y_s) must correspond to a dew-point condition, we then have a wet-bulb straight line on a temperature/humidity chart sloping down from the point (T_s, Y_s) with a slope of:
 $-(l k'g/hc)$

A further important concept is that of the adiabatic saturation condition. This is the situation reached by a stream of water, in contact with the humid air. Both ultimately reach a temperature at which the heat lost by the humid air on cooling is equal to the heat of evaporation of the water leaving the stream of water by evaporation.

Under this condition with no heat exchange to the surroundings, the total enthalpy change

$$\Delta H = c_s(T_a - T_s) + l(Y_s - Y_a) = 0$$

where c_s is the humid heat of the air.

Now it just so happens, for the water/air system at normal working temperatures and pressures that for practical purposes the numerical magnitude of the ratio:

$$hc/csk'g \text{ (known as the Lewis number)} \gg 1$$

This has a useful practical consequence. The wet bulb line and the adiabatic saturation line coincide when the Lewis number = 1.

It is now time to examine the chart we have spoken about. It is called a psychrometric chart.

2.2 Psychrometric charts

In the preceding discussion, we have been considering a chart of humidity against temperature, and such a chart is given in Fig.2.2.1.

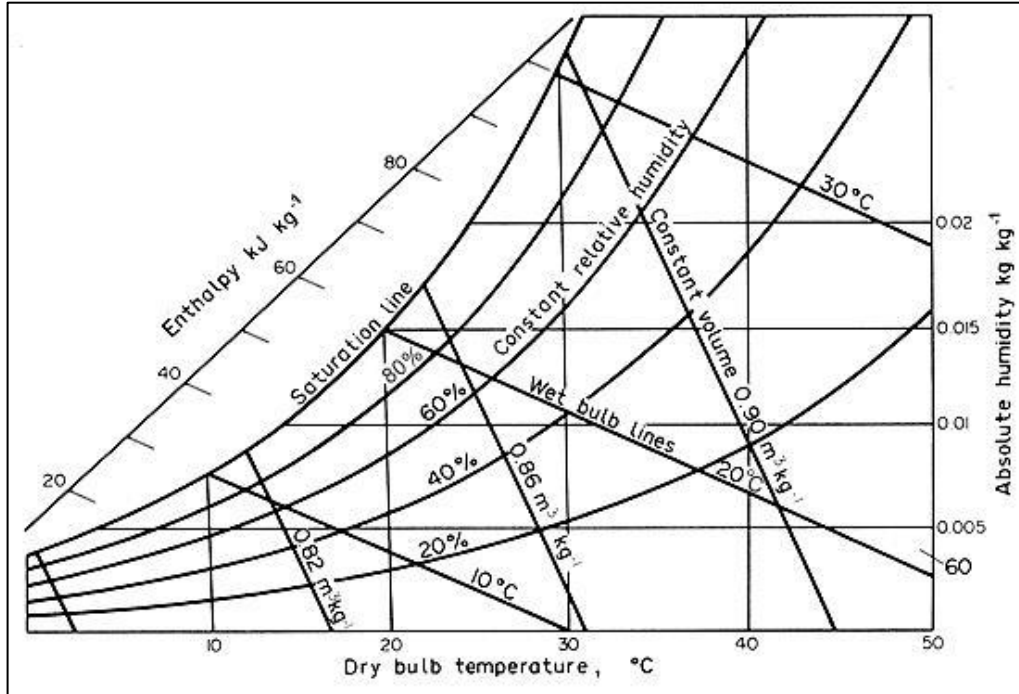


Fig. 2.2.1 Psychrometric chart

This chart can be used as the basis of many calculations. It can be used to calculate relative humidities and other properties.

3. THEORY OF COOLING TOWER

The humidification process can be represented graphically plotting humidity vs. gas temperature. There are several different situations that can occur during the countercurrent contact of air and water (see Fig. 3.1).

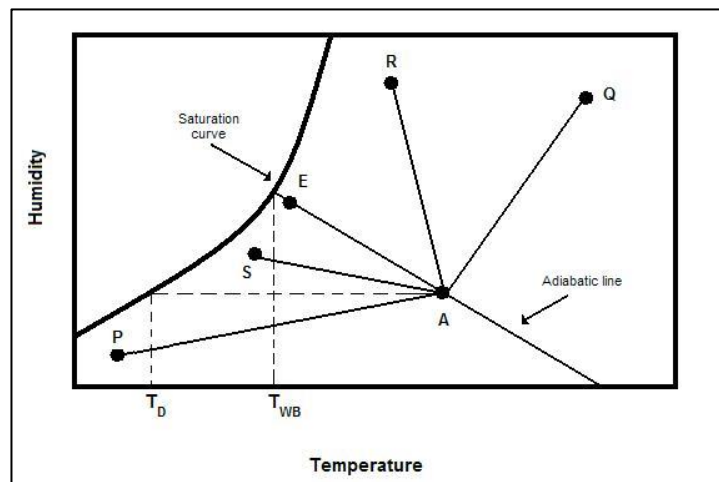


Fig. 3.1

The curves of the diagram illustrate the conditions of the air as it flows from the bottom of the tower (A) to the top of the tower (Q, R, E, S, or P depending on the condition).

Curve AQ: liquid cooling with air heating and humidification

Curve AR: liquid cooling with air cooling and humidification

Curve AE: adiabatic liquid cooling with air humidification

Curve AS: air cooling and humidification but less humidification than in adiabatic cooling

Curve AP: liquid heating and gas dehumidification

Consider the cooling tower illustrated in Fig. 3.2 operating under steady state counter current flow conditions.

G is the mass flow rate of the dry air per unit cross-section ($\text{kg}/\text{m}^2\cdot\text{h}$) and remains constant throughout the tower. Assume that the water rate per unit cross-section ($\text{kg}/\text{m}^2\cdot\text{h}$) is also constant at L (only a small fraction of water evaporates - 1-5% - compared to the total amount of liquid feed into the tower).

A mass balance on a differential element of column gives:

$$dL = G \cdot dX \quad (1)$$

where dX is the increment of humidity ($\text{kgH}_2\text{O}/\text{kg}$ dry air) of air.

An enthalpy balance on the same differential element of column gives:

$$G \times dH_G = L \times dH_L \quad (2)$$

where H_G is the enthalpy of air (kcal/kg) and H_L of the liquid (kcal/kg).

With $T_0 = 0^\circ\text{C}$ as reference temperature for the enthalpy, we have:

$$H_L = C_{p,L} \cdot (T_L - T_0) \quad (3)$$

$$H_G = C_{p,G} \cdot (T_G - T_0) + \lambda_L \cdot X \quad (4)$$

Where:

$C_{p,L}$ = average specific heat of liquid ($\text{kcal}/\text{kg}\cdot^\circ\text{C}$) in the range $T_L - T_0$

$C_{p,G}$ = average specific heat of gas ($\text{kcal}/\text{kg}\cdot^\circ\text{C}$) in the range $T_G - T_0$

λ_L = average evaporation enthalpy (kcal/kg)

X = gas humidity ($\text{kgH}_2\text{O}/\text{kg}$ dry air)

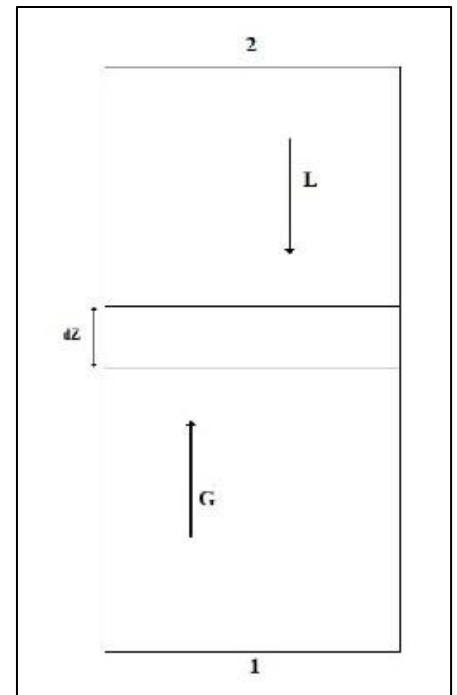
Substituting the differential of eq. 3 in eq. 2, we obtain:

$$G \times dH_G = L \times C_{p,L} \times dT_L \quad (5)$$

The integral of eq. 5 between the bottom and the top of the column is:

$$G (H_{G2} - H_{G1}) = L c_{p,L} (T_{L2} - T_{L1}) \quad (6)$$

Where the subscript "1" denotes "at the tower inlet" and the subscript 2 denotes "at the tower outlet".



The eq. 6 is the operating line and can be rewrite as follow:

$$(HG_2 - HG_1) = (L/G) c_{p,L} (T_{L2} - T_{L1}) \quad (6.1 \text{ operating line})$$

Energy (Heat) Balance

$$Q_{\text{load}} = Q_{\text{air}} + Q_{\text{losses}}$$

Q_{load} (cooling load) is the amount of heat released from the hot stream (hot water)

$$\text{Cooling load} = \dot{m}_{\text{hot water}} * c_p * \Delta T = \dot{m}_{\text{hot water}} * c_p * (T_{\text{water, inlet}} - T_{\text{water, outlet}})$$

$$Q_{\text{air}} = \dot{m}_{\text{dry air}} (\text{Enthalpy}_{\text{Air, out}} - \text{Enthalpy}_{\text{Air, in}})$$

$$Q_{\text{load}} = Q_{\text{air}} + Q_{\text{losses}}$$

$$\dot{m}_{\text{hot water}} * c_p * (T_{\text{water, inlet}} - T_{\text{water, outlet}}) = \dot{m}_{\text{dry air}} (\text{Enthalpy}_{\text{Air, out}} - \text{Enthalpy}_{\text{Air, in}}) + Q_{\text{losses}}$$

$$\% \text{ losses} = \frac{Q_{\text{losses}}}{Q_{\text{load}}} * 100\%$$

Mass Balance

Dry air flow rate ($\dot{m}_{\text{dry air}}$) In (Kg/s)

$$\dot{m}_{\text{dry air}} = 0.0165 \sqrt{\frac{\Delta P}{(1+wB) \cdot v_{AB}}}$$

where:

ΔP : orifice differential pressure (mmH₂O)

w_B : specific humidity of outlet dry air ($\frac{kg}{kg}$)

v_{AB} : specific volume of outlet dry air ($\frac{m^3}{kg}$)

$$\dot{m}_{\text{Evaporation, Experimental}} = \frac{\text{make up quantity (kg)}}{\text{sampling time (sec)}}$$

$$\dot{m}_{\text{Evaporation, Theoretical}} = \dot{m}_{\text{dry air}} (\text{Humidity}_{\text{out}} - \text{Humidity}_{\text{in}})$$

$$\% \text{ Error} = \frac{|\dot{m}_{\text{Evaporation, Experimental}} - \dot{m}_{\text{Evaporation, Theoretical}}|}{\dot{m}_{\text{Evaporation, Theoretical}}}$$

Procedure

• Start up

-Close valve V3

-Open partially valves V1 and V2

-Fill the reservoirs of the wet bulb thermometers TI3 and TI5 with water and ensure that the gauze covering each of the wet bulb thermometers is thoroughly wetted

-Fill the hot water reservoir D2 with distilled water (necessary about 10 liters) through the make-up tank D1 up to the mark

Keep attention: the water level in the make-up reservoir D1 must be minimum 80 mm; if there is not water in D, do not switch on the heating elements J1, J2 and J3.

- Connect the unit with the electrical supply: single-phase + G
- To measure the pressure drop on the packing, open the two relative valves on the differential manometer and close the others (see the labels)
- To measure the pressure drop on the orifice, open the two relative valves on the differential manometer and close the others (see the labels)
- Switch on the E.L.C.B
- Set the thermostat TW2 at 40°C .
- Switch on the three electrical heating elements J1, J2 and J3
- Switch on the pump G1 .
- Adjust the water flow rate using the valves V1 and V2 (for example at 120 l/h)
- Switch on the fan P1 and by means of the damper (Louver) adjust the air flow rate (for example at 50% louver opening)
- Allow about 10 minutes for conditions to reach steady state
- Measure and record the process parameters

- **Shut down**

- Switch of the heaters J1, J2 and J3
- After about 2-3 minutes, switch off the fan P1 and water pump G1
- Switch off the E.L.C.B.
- If the unit is idle for long period (more than 4-5 days), it should be completely drained trough the valve V3 .

Useful data

$$\text{Orifice formula: } m = 0.0165 \sqrt{\frac{\Delta P}{v_B}} = 0.0165 \sqrt{\frac{\Delta P}{(1+w_B) \cdot v_{AB}}}$$

Where:

m = dry air mass flow rate (kg/s)

ΔP = orifice differential pressure (mmH₂O)

v_B = specific volume of outlet humid air

w_B = specific humidity of outlet air

v_{AB} = specific volume of outlet dry air

Dimension of column: 150 · 150 · 600 (h) mm

Packing data:

	A	B	C
Decks number	8	8	8
Plates number	7	10	18
Total surface m²	1.1	1.56	2.8
Packing height m	0.53	0.53	0.53
Packing specific surface m²/m³	92	131	235

Constant

Water specific heat	1 kcal/kg/K
Air specific heat (range -50 40 °C)	0.24 kcal/kg/K
1 kW	860 kcal/h

Heating elements

The heating elements are rated at 230 V (subject to manufacturing tolerance) and the nominal rating, at the local voltage, should be multiplied by $(V_{\text{local}} / 230)^2$.

In the following experiments the local voltage was 212 V and then the effective rating is 425 V for each element.

Experiment

Objective: determination of overall mass and energy balance

-Stabilize the column on the following suggested condition:

Louver opening	50%
Water flow rate	120 l/h
Cooling load	???? Must be calculated

-Fill the make up tank up to the mark with distilled water

-Note temperatures and flows at regular intervals of 5 minutes

-At the end of this period, refill the make up tank with distilled water using a measuring cylinder. -

-By difference, calculate the make up water supplied in the time interval

Data Sheet

Date:	Atm. Pressure:					
Test n°	1	2	3	4	5	6
Packing type						
Packing density m^{-1}						
Air inlet Dry bulb (°C)						
Air inlet Wet bulb (°C)						
Air outlet Dry bulb (°C)						
Air outlet Wet bulb						
Water inlet temperature (°C)						
Water outlet temperature (°C)						
Water make up temperature (°C)						
Orifice ΔP mmH ₂ O						
Water flow rate m_w (kg/h)						
Cooling load Q (kW)						
Make up quantity m_{mu} (kg)						
Sampling time t (sec)						
Packing ΔP mmH ₂ O						

EXPERIMENT 8

REVERSE OSMOSIS

Theory

To be useful for separation of different species, a membrane must allow passage of certain molecules and exclude or greatly restrict passage of other molecules. In osmosis, a spontaneous transport of solvent occurs from a dilute solute or salt solution to a concentrated solute or salt solution across a semipermeable membrane which allows passage of the solvent but impedes passage of the salt solutes. (In Fig. 1-a) the solvent water normally flows through the semipermeable membrane to the salt solution. The levels of both liquids are the same as shown. The solvent flow can be reduced by exerting a pressure on the salt-solution side and membrane, as shown in (Fig. 1-b), until at a certain pressure, called the osmotic pressure π of the salt solution, equilibrium is reached and the amount of the solvent passing in opposite directions is equal. The chemical potentials of the solvent on both sides of the membrane are equal. The property of the solution determines only the value of the osmotic pressure, not the membrane, provided that it is truly semipermeable. To reverse the flow of the water so that it flows from the salt solution to the fresh solvent as in (Fig. 1-c), the pressure is increased above the osmotic pressure on the solution side.

This phenomenon, called reverse osmosis, is used in a number of processes. An important commercial use is in the desalination of seawater or brackish water to produce fresh water. Unlike distillation and freezing processes used to remove solvents, reverse osmosis can operate at ambient temperature without phase change. This process is quite useful for processing of thermally and chemically unstable products. Applications include concentration of fruit juices and milk, recovery of protein and sugar from cheese whey, and concentration of enzymes.

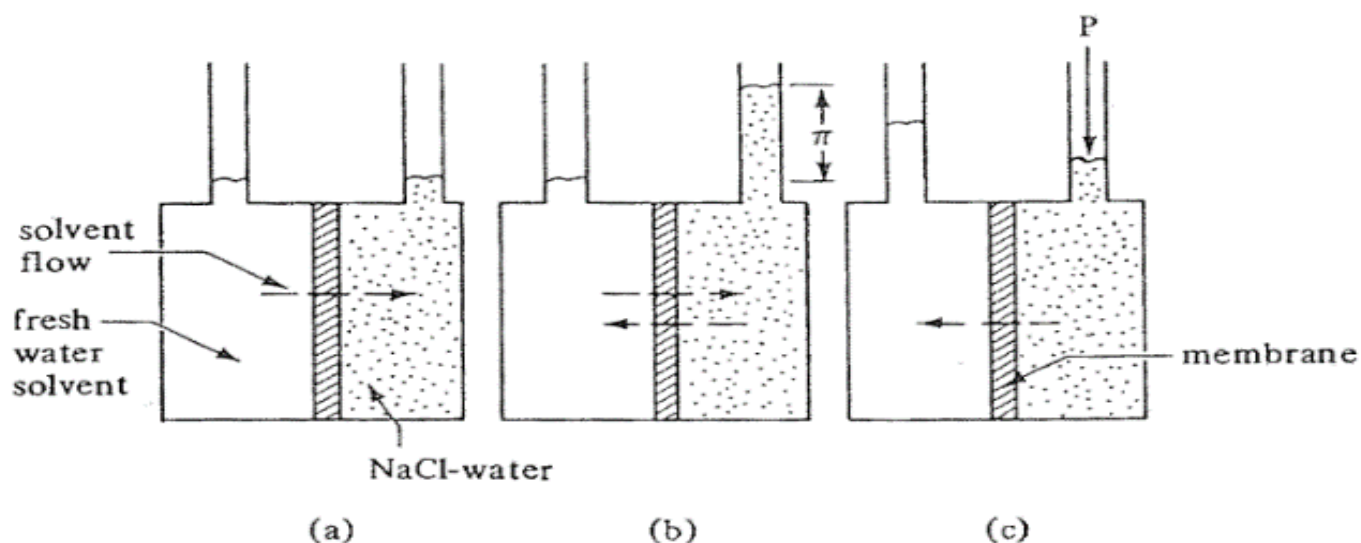


Figure 1: Osmosis and reverse osmosis: (a) osmosis, (b) osmotic equilibrium, (c) reverse osmosis.

Reverse Osmosis (RO) is a separation technique that is suitable for a wide range of applications, especially when salt and/or dissolved solids need to be removed from a solution. Accordingly, RO can be used for seawater and brackish water desalination, to produce both water for industrial application, and drinking water. It can also be applied for the production of ultrapure water (e.g. semiconductor, pharmaceutical industries) and boiler feed water. In addition, RO membrane systems are used for wastewater and water reuse treatments. RO is currently considered one of the most economic and effective process for water desalination. Accordingly, it is often the appropriate technique to treat solutions having salt concentrations from 100 to over 50,000 mg/liter. Solutions with salinity from surface water to sea water, and even brines, can be treated by RO membrane. Cross flow is the configuration applied for membrane separation using RO membrane.

Osmosis is a natural phenomenon which can be defined as the movement of pure water through a semi permeable membrane from a low to a high concentration solution (see Figure 2). The membrane is permeable to water and some ions but rejects almost all ions and dissolved solids. This process (movement of water) occurs until the osmotic equilibrium is reached, or until the chemical potential is equal on both sides of the membrane.

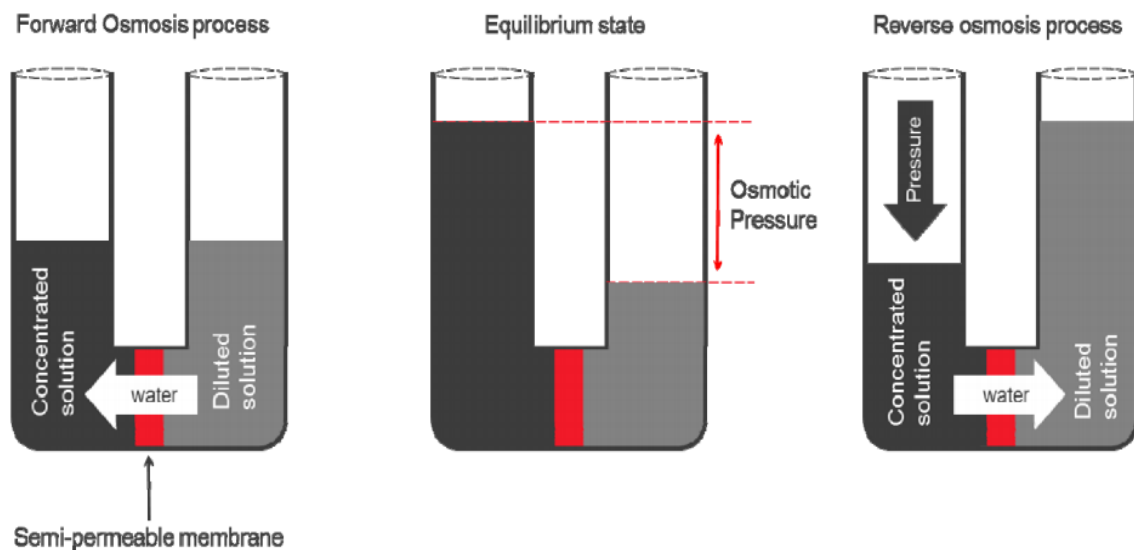


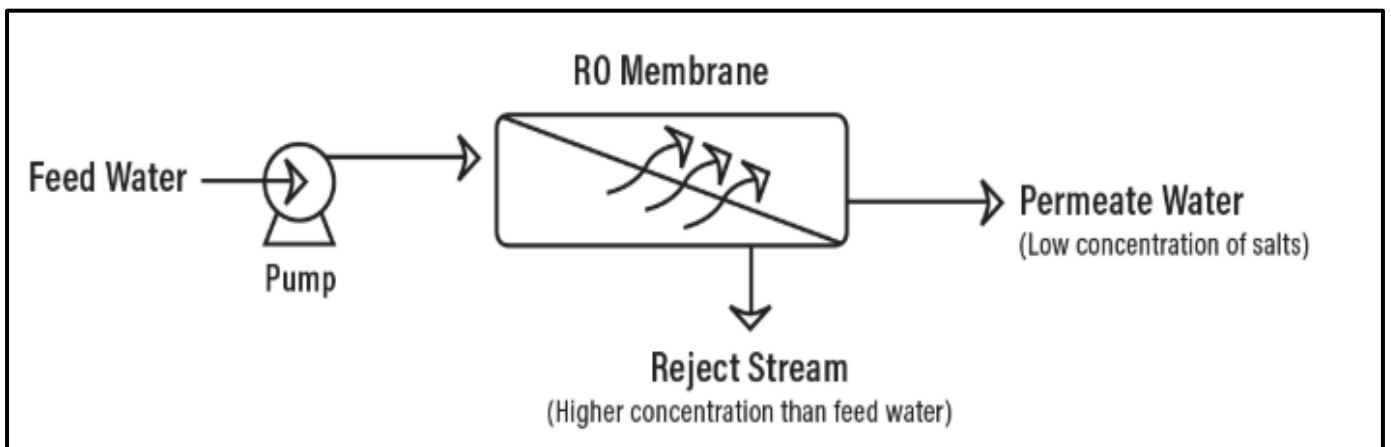
Figure 2: Principle Osmosis and of Reverse Osmosis (RO)

Obviously, the mass of water which moves across the membrane depends strongly on the concentration of salt on each side of the membrane. The more concentrated the salt solution, the greater the driving force for water diffusion to the concentrated side. Keep this in mind when thinking about reverse osmosis.

Reverse Osmosis is exactly that, the reverse of osmosis. A pressure is applied to the side of the membrane containing the salt solution, which is greater than the osmotic pressure. This forces the water across the membrane from the solution side to the "pure" side (reverse osmosis).

Reverse Osmosis works by using a high pressure pump to increase the pressure on the salt side of the RO and force the water across the semi-permeable RO membrane, leaving almost all (around 95% to 99%) of dissolved salts behind in the reject stream. The amount of pressure required depends on the salt concentration of the feed water. The more concentrated the feed water, the more pressure is required to overcome the osmotic pressure.

The desalinated water that is demineralized or deionized, is called permeate (or product) water. The water stream that carries the concentrated contaminants that did not pass through the RO membrane is called the reject (or concentrate) stream.



As the feed water enters the RO membrane under pressure (enough pressure to overcome osmotic pressure) the water molecules pass through the semi-permeable membrane and the salts and other contaminants are not allowed to pass and are discharged through the reject stream (also known as the concentrate or brine stream), which goes to drain or can be fed back into the feed water supply in some circumstances to be recycled through the RO system to save water. The water that makes it through the RO membrane is called permeate or product water and usually has around 95% to 99% of the dissolved salts removed from it.

Reverse osmosis process efficiency

- **SALT REJECTION %**

This equation tells you how effective the RO membranes are removing contaminants. It does not tell you how each individual membrane is performing, but rather how the system overall on average is performing. A well-designed RO system with properly functioning RO membranes will reject 95% to 99% of most feed water contaminants (that are of a certain size and charge). You can determine how effective the RO membranes are removing contaminants by using the following equation:

$$\text{Salt Rejection \%} = \frac{\text{Conductivity of Feed Water} - \text{Conductivity of Permeate Water}}{\text{Conductivity of Feed Water}} \times 100\%$$

The higher the salt rejection, the better the system is performing. A low salt rejection can mean that the membranes require cleaning or replacement.

- **SALT PASSAGE %**

This is simply the inverse of salt rejection described in the previous equation. This is the amount of salts expressed as a percentage that are passing through the RO system. The lower the salt passage, the better the system is performing. A high salt passage can mean that the membranes require cleaning or replacement.

$$\text{Salt Passage \%} = (1 - \text{Salt Rejection \%})$$

- **RECOVERY %**

Percent Recovery is the amount of water that is being 'recovered' as good permeate water. Another way to think of Percent Recovery is the amount of water that is not sent to drain as concentrate, but rather collected as permeate or product water. The higher the recovery % means that you are sending less water to drain as concentrate and saving more permeate water. However, if the recovery % is too high for the RO design then it can lead to larger problems due to scaling and fouling. The % Recovery for an RO system is established with the help of design software taking into consideration numerous factors such as feed water chemistry and RO pre-treatment before the RO system. Therefore, the proper % Recovery at which an RO should operate at depends on what it was designed for. By calculating the % Recovery you can quickly determine if the system is operating outside of the intended design. The calculation for % Recovery is below:

$$\% \text{ Recovery} = \frac{\text{Permeate Flow Rate}}{\text{Feed Flow Rate}} \times 100 \%$$

For example, if the recovery rate is 75% then this means that for every 100 gallons of feed water that enter the RO system, you are recovering 75 gallons as usable permeate water and 25 gallons are going to drain as concentrate. Industrial RO systems typically run anywhere from 50% to 85% recovery depending the feed water characteristics and other design considerations.

Factors influencing the efficiency of the separation process

The two most important variables for describing the separation efficiency are:

- **Flow rate of the permeate**
- **Rejection**

These two variables are mainly influenced by the following parameters:

- **Pressure:** With increasing pressure in the raw water, the flow rate of the permeate increases. On the other hand, the concentration of the solute in the permeate declines with increasing pressure.
- **Temperature:** The flow rate of the permeate increases with increasing temperature, if all other parameters remain constant.
- **Recovery:** The flow rate of the permeate increase with increasing recovery
- **Concentration in the raw water:** Rejection and the flow rate of the permeate decrease with increasing concentration of solute in the raw water.

Experimental part

Apparatus

Complete device

CE 530 is designed for the desalination of NaCl solution of up to 4wt/wt % max. The pump pumps the solution out of the raw water tank to the membrane module. In the membrane module the salt solution (raw water) is separated into a high concentrated salt solution (retentate) and a low concentration salt solution (permeate).

Permeate and retentate are fed back into the raw water tank. Separation occurs according to the principle of reverse osmosis. The separation process requires a pressure that is higher than the osmotic pressure. The pump generates this pressure.

The pressure is set using the overflow valve V1 in the bypass to the pump and the valve V2 behind the membrane module in the retentate. The complete device consists of a supply unit and a trainer. The supply unit and trainer are connected to each other hydraulically and electrically.

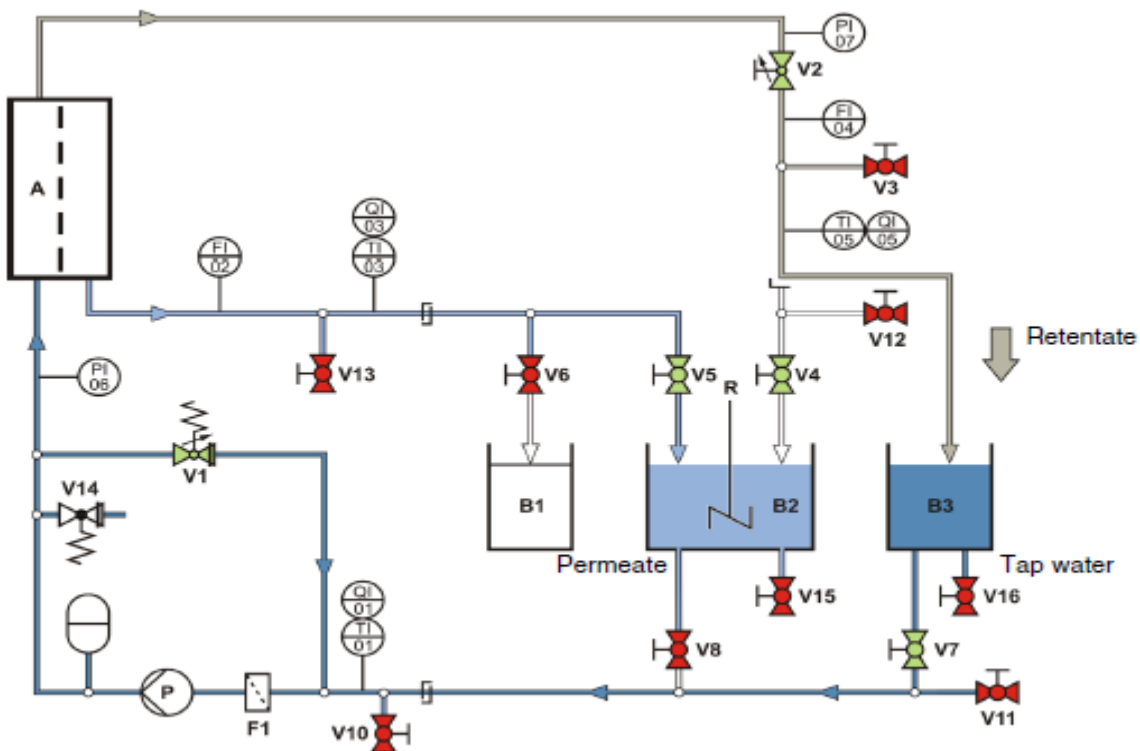
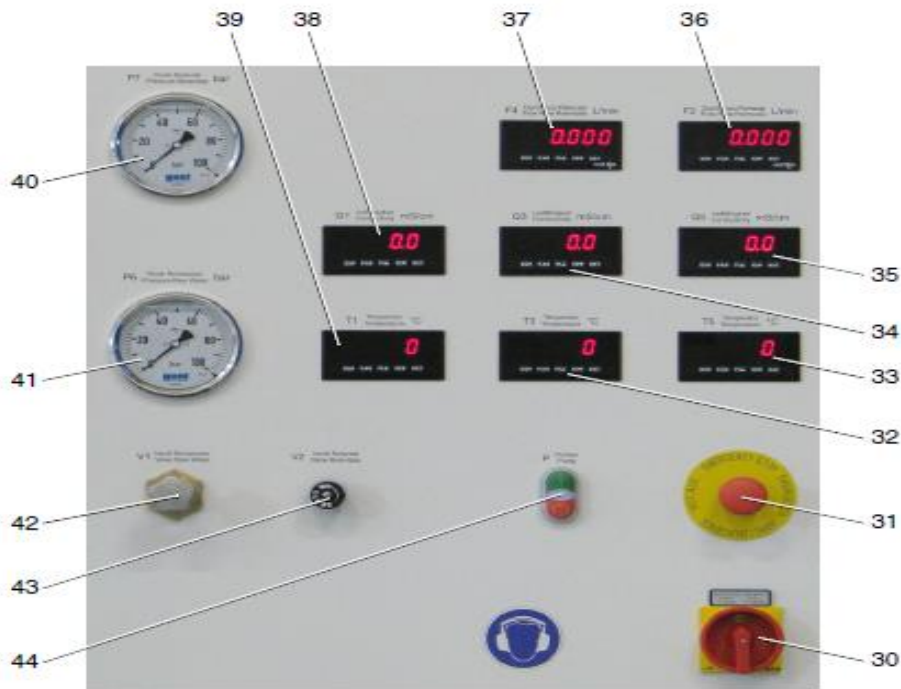


Fig. 3.18 Process diagram for the production of permeate



4.2

30	Main switch	38	Raw water conductivity display
31	Emergency off switch	39	Raw water temperature display
32	Permeate temperature display	40	Retentate manometer
33	Retentate temperature display	41	Raw water manometer
34	Permeate conductivity display	42	Bypass valve (V1)
35	Retentate conductivity display	43	Retentate valve (V2)
36	Permeate flow rate display	44	On/off switch for pump
37	Retentate flow rate display		

Fig. 3.13 Display and control elements

procedure

1. Weigh out 1kg NaCl in a tank.
2. Turn trainer on at the main switch.
3. Turn on stirring machine R.
4. Put NaCl into tank B2 while stirring.
5. Wait until the NaCl is completely dissolved.
6. Turn stirrer off.

7. Valves and taps at the beginning:

- V1 fully open
- V2 fully open
- V3 closed
- V4 open
- V5 open
- V6 closed
- V7 closed
- V8 closed
- V10 closed
- V11 closed
- V12 closed
- V13 closed
- V15 closed
- V16 closed

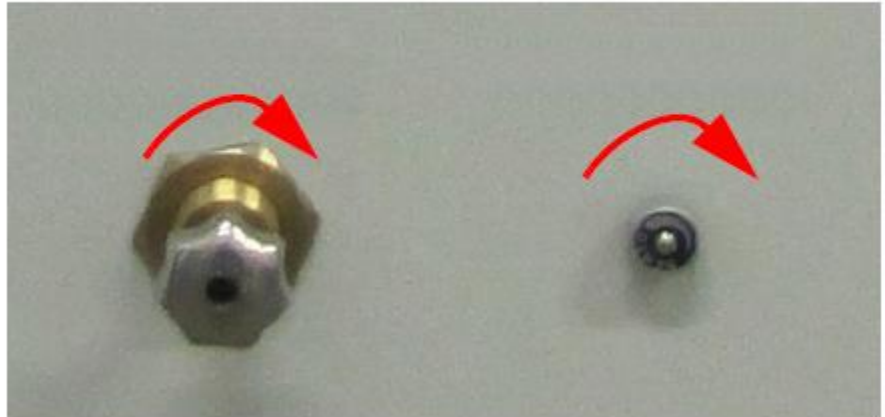


Fig. 5.4 Setting a higher pressure with the valves V1 (left) and V2 (right)

8. Open V8.

9. Switch on the pump.

Observations:

– *Liquid flows from the tap V4 into the tank B2.*

– *Little or no liquid flows from the tap V5.*

10. Put on ear defenders. Vibrations can occur in the transition area from the unpressurised state to the operating state.

11. Simultaneously turn valves V1 and V2 quickly clockwise, in order to quickly exceed the transition area (see Fig. 5.4). Set a pressure of about 40 bar.

Observation:

– *Liquid flows from the taps V4 and V5.*

12. By varying valves V1 and V2, set raw water pressure to 40 bar.

13. Wait about 5min.

14. From this point on, note down flow rates, conductivity and temperature in the raw water, permeate and retentate.

15. Repeat the experiment by increasing the pressure.

Data sheet

Pressure	Conductivity Of Raw water	Conductivity Of permeate	Conductivity Of retentate	Flow rate of permeate	Flow rate of retentate

Best of luck!