Experiment 5
Liquid Diffusion Coefficient

1.0 OBJECTIVES OF EXPERIMENT

➢ To determine the liquid diffusion coefficient of NaCl solution in distilled/de-ionized water.

2.0 INTRODUCTION

The knowledge of physical and chemical properties of certain materials is important because very often process engineering deal with the transformation and distribution of these materials in bulk. One such property is diffusivity. Mass transfer takes place when there is a concentration gradient of the diffusing component.

2.1 Theory

When a concentration gradient exists within a fluid consisting of two or more components, there is a tendency for each constituent to flow in such a direction as to reduce the concentration gradient. This is known as mass transfer. It takes place in either a gas or a liquid phase or in both phases simultaneously.

Rate of diffusion is given by

\[ J = -D \frac{\partial C}{\partial x} \tag{1} \]

where \( J \) = diffusion flux across unit area to the \( x \)-direction (right side), mol/cm\(^2\)s

\( D \) = diffusivity, cm\(^2\)/s

\( \frac{\partial C}{\partial x} \) = concentration gradient in the \( x \)-direction, mol/cm\(^3\)

The negative sign indicates that flow is from high to low concentration.

By expanding Eq. (1) using respective terms constitutive relations, we get

\[ \frac{V}{C_M} \frac{dk}{dt} = -D \left( \frac{\pi d^2}{4} \right) N \frac{M}{x} \tag{2} \]
Rearranging gives

\[
D = \frac{4Vx}{\pi d^2 NMC_m} \frac{dk}{dt}
\]  

where \( V \) = volume of water in diffusion vessel, L  
\( x \) = length of capillaries, cm  
\( d \) = capillaries diameter, cm  
\( N \) = number of capillaries  
\( M \) = molar concentration of NaCl solution, mol/L  
\( C_m \) = conductivity change per unit molar concentration change, \( \mu S/mol/L^{-1} \)  
\( \frac{dk}{dt} \) = rate of conductivity change over time

The slope obtained from the plot of conductivity as function of time can be used to calculate the diffusivity.

3.0 EQUIPMENT – LIQUID DIFFUSION COEFFICIENT APPARATUS

This liquid diffusion coefficient apparatus is used to determine the diffusivity of NaCl solution in distilled water. A known concentration of NaCl solution is placed in a diffusion cell immersed in distilled water. A magnetic stirrer and a conductivity meter are provided to monitor the progress of diffusion over time. A plot of conductivity against time will allow for the determination of the liquid diffusivity.

![Figure 2.1: Liquid Diffusion Coefficient Apparatus](image)

The concentration at the chosen lower ends is taken to be constant while the concentration at top end is effectively zero during experiment.
4.0 OPERATING PROCEDURE

4.1 Pre-experiment Procedures

1. Read and understand the theory of liquid diffusion.
2. Read and understand the equipment used in the experiment (liquid diffusion coefficient apparatus).
3. Read the safety precautions before conducting the experiment.
4. Prepare the following materials needed for the experiment:
   - Prepare 200 mL 1 M NaCl solution
   - Prepare 200 mL 2 M NaCl solution
   - Prepare 200 mL 4 M NaCl solution

4.2 General Start-Up Procedures

1. Fill the diffusion vessel with 1.1 L of distilled/de-ionized water.
2. Connect the conductivity probe BNC connector into the socket on the conductivity meter. Insert mini phono jack temperature sensor (built in) into the socket on conductivity meter.
3. Plug the magnetic stirrer main cable to electrical supply. Ensure that the supply voltage is correctly suit the equipment.
4. Press the ON button on the conductivity meter.
5. Switch on the magnetic stirrer and set the speed knob to 760 rpm.
6. Read the conductivity value. Distilled water shall give a very low reading.
7. The equipment is ready for the experiment.

4.3 General Shutdown Procedures

1. Switch off the magnetic stirrer and conductivity meter.
2. Disconnect both the BNC connector & mini phono jack from the conductivity meter.
3. Drain the solution in the diffusion vessel and cell and rinse them.
4. Salt solution may attack the metal part of the equipment. Wipe off any spillage from the unit immediately.
5.0 SAFETY PRECAUTIONS

- All operating instructions supplied with the unit must be carefully read and understood before attempting to operate the unit.
- Feed stock which severely affects PTFE, PFA, PVC and borosilicate glass are not to be used.
- The system should not be subjected to shock, sudden impact, vibration, additional load, or permanent external action of aggressive vapours.
- Always check and rectify any leak.
- Do not use any coarse or abrasive cleaners on glass and plastic components.
- Leaking couplings or fittings should be carefully retightened.

6.0 EXPERIMENT

6.1 Experiment Procedures

1. Start-up the unit according to procedures in Section 4.2.
2. Fill the diffusion cell with 1 M NaCl solution. Ensure that the capillary tubes are in place. Completely fill the cell and wipe off any excess solution.
3. Carefully immerse the cell into the distilled water and position the cell until the top of the capillaries is about 5 mm below the water level.
4. Switch on the conductivity meter and the magnetic stirrer.
5. After 10 minutes, record the conductivity reading. Take readings every 5 minute intervals for 40 minutes.
6. Repeat Step 1 to Step 6 for 2 M and 4 M NaCl solutions.

6.2 Experimental Datasheet

Sample data collection table is shown as follows:

<table>
<thead>
<tr>
<th>Volume of water, $V$</th>
<th>Length of capillaries, $x$</th>
<th>Diameter of capillaries, $d$</th>
<th>Number of capillaries, $N$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 M NaCl Solution</td>
<td>2 M NaCl Solution</td>
<td>4 M NaCl Solution</td>
<td></td>
</tr>
<tr>
<td>Conductivity, $k$ ($\mu$S)</td>
<td>Conductivity, $k$ ($\mu$S)</td>
<td>Conductivity, $k$ ($\mu$S)</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Time, $t$ (s)</th>
<th>1 M NaCl Solution</th>
<th>2 M NaCl Solution</th>
<th>4 M NaCl Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Conductivity, $k$ ($\mu$S)</td>
<td>Conductivity, $k$ ($\mu$S)</td>
<td>Conductivity, $k$ ($\mu$S)</td>
</tr>
</tbody>
</table>
7.0 RESULTS ANALYSIS AND DISCUSSIONS

Discuss all your results. The questions below only serve as a guideline. Your discussion should not only limited to these questions.

1. Plot a graph of conductivity versus time. Determine the liquid diffusivity of NaCl solution from the obtained slope.

2. Compare the experimental value with theoretical value predicted from empirical equations.

3. Comment on the diffusivity values obtained at different NaCl concentration.
Experiment 6
Batch Distillation Column

1.0 OBJECTIVES OF EXPERIMENT

- To understand the principles of distillation column.
- To determine the composition of a mixture by using refractive index.
- To determine the variation of top product against time at constant reflux ratio.
- To investigate the effects of constant reflux ratio and regulating reflux ratio on product composition.
- To determine the temperature profile along the height of the column.

2.0 INTRODUCTION

Distillation process is widely applied in industry for separation operations. It is a process in which a liquid or vapour mixture of two or more substances is separated into its component fractions of desired purity by the application and removal of heat. Distillation processes depends on the distribution of components between gas and liquid phase.

The more volatile, “light” or “low boiling”, components will concentrate in the vapour. The less volatile, “heavy” or “high boiling”, components will concentrate in the liquid phase.

Separation of components from a liquid mixture via distillation depends on the differences in boiling points of the individual components. Also, depending on the concentrations of the components present, the liquid mixture will have different boiling point characteristics.

The simplest distillation is the separation of binary mixtures. The typical binary equilibrium curves, usually plotted for the more volatile component are shown in the Figure 2.1. Curve A is the most common case where the more volatile component remains more volatile throughout the entire composition range.

Curve B is a system with homogeneous azeotrope, where the component which is more volatile at low values of $x_1$ and becomes less volatile at high values of $x_1$. The vapour and liquid compositions are equal when the curve crosses the $x = y$ line. The maximum overhead product composition achievable with a feed of $x_1 = 0.25$ is the azeotropic composition. Similarly, the maximum bottoms product composition can be no lower than the azeotrope composition for a feed with $x_1 = 0.9$.

Curve C is a system with heterogeneous azeotrope, or two liquid phases in equilibrium with one vapour phase. The azeotrope determines the maximum possible separation achievable by simple distillation.
2.1 Batch Distillation

Batch distillation is a process commonly carried out in small plants to recover volatile products from a liquid solution. The liquid mixture is charged into a still or reboiler, and heat is supplied through a heating coil or jacket to partially vaporise the mixture. The most basic method of operation is to directly take the vapours to a condenser. The vapour that leaves the still is always in equilibrium with the liquid in the still, but due to the high concentration of the more volatile component in the vapour, the compositions of liquid and vapour are not constant.

To determine how the compositions vary with time, consider the case where $n_0$ moles are charged into a batch distillation unit. Let $n$ be the moles of liquid remaining in the still at a given time, and $x$ and $y$ be the vapour and liquid compositions respectively. The total moles of a more volatile component $A$ left in the still, $n_A$, will be

$$n_A = xn \tag{1}$$

Differentiating Eq. (1) gives

$$dn_A = d(xn) = n \, dx + x \, dn \tag{2}$$

If a small amount of liquid, $dn$, is vaporised, the change in the moles of $A$ is $y \, dn$, or $dn_A$.

From Eq. (2),

$$y \, dn = n \, dx + x \, dn$$

$$\frac{dn}{n} = \frac{dx}{y - x} \tag{3}$$
By integrating Eq. (3) between the initial and final concentrations, we obtain

\[ \int_{n_0}^{n} \frac{dn}{n} = \int_{x_0}^{x} \frac{dx}{y - x} \]

or

\[ \ln \frac{n}{n_0} = \int_{x_0}^{x} \frac{dx}{y - x} \]  \hspace{1cm} (4)

Eq. (4) is known as the Rayleigh Equation. The term \( dx/(y - x) \) can be integrated graphically or numerically using equilibrium curve.

The Rayleigh equation can also be derived for an ideal mixture based on relative volatility. Although the temperature in the distillation column increases during a batch distillation process, the relative volatility, or the ratio of vapour pressures, does not change drastically. Hence, an average value for relative volatility is used and assumed to be constant.

\[ \frac{y_A}{y_B} = \alpha_{AB} \frac{x_A}{x_B} \]  \hspace{1cm} (5)

Consider a binary mixture consisting of \( n_A \) mol of A and \( n_B \) mol of B. The ratio \( n_A/n_B \) is equal to \( x_A/x_B \). When \( dn \) mol is vaporised, the change in component A is \( y_Adn \) or \( dn_A \), and the change in component B is \( y_Bdn \) or \( dn_B \). Substituting these terms into equation (4) yields

\[ \frac{dn_A}{dn_B} = \frac{\alpha_{AB} \frac{n_A}{n_B}}{dn_B} \]  \hspace{1cm} (6)

Integrating Eq. (6) between the initial and final condition yields

\[ \ln \frac{n_A}{n_{0A}} = \alpha_{AB} \ln \frac{n_B}{n_{0B}} \]  \hspace{1cm} (7)

Eq. (7) above can be plotted as a straight line on a logarithmic scale. If the recovery of either component is specified, then Eq. (7) can be used directly to determine the recovery of the remaining component.
3.0 EQUIPMENT – BATCH DISTILLATION COLUMN

The batch distillation column is designed to demonstrate the separation of mixture with different boiling points. This unit comprised of a sieve plate column and a packed column which is interchangeable. The internal (plate or packings) of the column can be seen clearly via the borosilicate glass section in the middle.

The overhead condenser at the top of the column is used to condense the product vapour which is then collected at the reflux drum. The amount of top product to return back to the column or directed to the top product tank can be controlled by adjusting the reflux ratio. The range of reflux ratio can be adjusted from 0% to 100% via electronic control valves at the control panel. The reboiler at the base of the column is used to evaporate feed. A jet vacuum pump fitted at the top product line allows vacuum distillation.

![Batch Distillation Column Diagram](image)

Figure 3.1: Batch Distillation Column
4.0 OPERATING PROCEDURES

4.1 Pre-experiment Procedures

1. Read and understand the theory of distillation.
2. Read and understand the equipment used in the experiment (batch distillation column).
3. Read the safety precautions and chemical hazards before conducting the experiment.
4. Read the Material Safety Data Sheet (MSDS) for the chemicals used in the experiment in Appendix A – ethanol, 2-propanol.
5. Prepare the following apparatus and materials needed for the experiment:
   - Refractometer
   - Beakers
   - Syringe / Dripper
   - 0%, 10%, 20%, ..., 100% of ethanol-water or 2-propanol-water mixture
   - Prepare 10 L of 25 v/v% ethanol-water or 2-propanol-water mixture

4.2 General Start-Up Procedures

1. Ensure that all valves on the unit are closed.
2. Attach the inlet hose from the condenser unit to a water supply and the outlet hose to a drain.
3. Connect the 3-pin plug to 240VAC main power supply. Turn on the power supply and main power switch at the front of the control panel.
5. Open the cooling water supply and set the flow rate to maximum. The vacuum pump will now draw air from the column. Allow the vacuum pump to run for at least 10 minutes.
6. Close valves V5 and V6. Open valve V4 to allow cold water to circulate inside the condenser. (Note: Do not open valves V5 and V6 for the vacuum pump during the experiment)

4.3 General Shutdown Procedures

1. After the experiment, drain off the contents in the reflux drum and top product tank.
2. Dispose all chemicals into the container provided.
3. Switch off the main power switch at the control panel and power supply.
4. Turn off the cooling water supply.
5.0 CHEMICAL HAZARDS, SAFETY AND PRECAUTIONS

5.1 Chemical Hazards (refer MSDS in Appendix A for more details)

- Ethanol is very flammable, so constitutes a fire risk. Ethanol contact with the eyes can cause considerable irritation.
- Propanol is very flammable. It evaporates readily, so it is possible for dangerous levels of vapour to build up, perhaps reaching a point at which an explosion is possible if a source of ignition is present.
- If propanol is in contact with oxygen over a long period, explosive peroxides may be formed. These typically have a higher boiling point than propanol, so may become concentrated in the liquid if propanol is distilled. Therefore, bottles of propanol, once opened, should not be stored indefinitely, in order to avoid any risk of peroxide formation.
- 2-propanol is very flammable. It can be ignited by flames, but also by contact with items such as hot plates or hot air guns.

5.2 Safety Precautions

- Always wear safety glasses, mask and gloves when handling chemicals. Do not allow the solution to come into contact with your skin or eyes.
- Should any chemicals come into contact with the body, rinse off immediately with plenty of water and inform the laboratory instructor/officer. Seek medical treatment if symptoms persist.
- Ensure that there is no source of ignition, such as a Bunsen burner, gas flame, hot plate, hot air gun or hot water pipe near the working area.
- Propanol releases irritating vapours; avoid inhalation and work in a well-ventilated area.
- Good ventilation is essential so that it is not possible for high concentrations of alcohol vapour to form.
- Dispose of all unused chemicals in an appropriate manner after the experiment. Under no circumstances should the chemicals be allowed to flow into sinks or drains.
- Wash your hands thoroughly with soap after the experiment.
- Pour slowly and be very careful when pouring the mixture into the reboiler. The inlet nozzle is quite small so if the solution is poured too fast, the nozzle will choke and the solution will spill out.
6.0 EXPERIMENTS

6.1 Experiment 1A: Batch Distillation with Constant Reflux

1. Start-up the unit according to procedures in Section 4.2.

2. Fill 10 L of 25 v/v% ethanol-water or 2-propanol-water mixture into the reboiler slowly. The inlet nozzle is quite small so if the solution is poured too fast, the nozzle will choke and the solution will spill out. *(Caution: The person who pours the mixture should wear a face shield.)*

3. Collect a sample from the feed and measure its refractive index (see Experiment 2).

   *Note: Do not open the valves V5 and V6 for the vacuum pump during the experiment. Operation of the vacuum pump after the mixture is partially vaporised will cause the fluids to gush upwards and damage the column.*

4. Set the temperature of the reboiler, T13 to 85°C using the temperature controller on the control panel.

5. Set the reflux ratio to 100%.

6. Once the top product appears in the reflux drum, run the column at total reflux for another 10 minutes.

7. Record down all the temperatures, T1 to T13, by turning the temperature selector knob on the control panel.

8. Set the reflux ratio to 80%.

9. At intervals of 10 minutes, record all temperatures and collect a sample from both the top product tank and reboiler. Measure the refractive index of each sample. *(Note: the feed sample has to be cooled down to room temperature for accurate result)*

10. Repeat Step 9 until 4 sets of samples are collected. The system should stabilise after 40 minutes.

11. Empty the top product tank at the end of the 4th set of sample and measure its volume.

12. Tabulate all the data obtained and determine the composition of the feed and product using the calibrated data from Experiment 2.

13. Shutdown the unit according to procedures in Section 4.3.

6.2 Experiment 1B: Batch Distillation with Regulating Reflux

By using the same setup and chemicals as Experiment 1, design and run an experiment to determine the product composition with regulating reflux ratio.
6.3 **Experiment 2: Calibration of Refractometer**

1. Prepare 10 samples of ethanol-water or propanol-water mixture for 0%, 10%, 20%, ..., 100% concentration.

2. Open the secondary prism of the refractometer and drip 2 or 3 drops of 0% mixture onto the centre part of the main prism surface. Gently close the secondary prism. The sample will spreads between the main prism and secondary prim as a thin film.

3. While observing through the eyepiece, gently turn the measurement knob until the boundary line appears in the field of refraction view. At that time, the boundary line may be coloured and unclear because it is not yet achromatised.

4. Turn the measurement knob in a wide range so as to set it at a point where the field of refraction view clearly changes from bright field to dark field or vice versa. The brightness of the field can be adjusted with the illumination selector switch in the upper part of the digital thermometer.

5. Turn the colour compensator knob to achromatise the boundary line so that the line appears clearly in the field. Turn the measurement knob again to set the boundary line at the intersection of the cross hairs.

6. The upper scale is for reading refractive index ($n_D$) while the lower scale is for Brix percent. Record down the refractive index of the sample.

7. Repeat Step 2 to Step 6 for all the 10 samples mixture.

**CAUTION!**

- Measured value for liquid sample changes depending on the change of temperature. Therefore, samples' temperature must be kept constant.

- Do not wet the refractometer with sample liquid unreasonably. If the refractometer gets wet excessively, it may cause malfunction and breakdown of the refractometer.

- Do not tap or pick at the prism surface with a metal spoon, tweezers, etc. because the prism surface is made of optical glass. If the prism surface is scratched, the refractometer may fail in measurement.

- When measurement is complete, wipe up sample from the prism surface and its surroundings with soft tissues moistened with water first, and then wipe the wet parts with dry tissues to dry them up.

- After measurement of a sample of a high molecular compound (e.g. oil, fat etc) wipe the prism surface with tissues moistened with alcohol or neutral detergent first and then wipe it up again with dry tissues to dry up.

- On ending a day's measurement, turn off the power switch.
7.0 RESULTS ANALYSIS AND DISCUSSION

Discuss all your results. *The questions below only serve as a guideline. Your discussion should not only limited to these questions.*

1. Sketch a schematic diagram, including all valves and sensors/controllers, for the distillation unit used in the experiment. Label all valves and instruments.
2. Plot the calibration curve and use it to determine the composition of a mixture.
3. Discuss the variation of top product against time at constant reflux ratio.
4. Discuss the effects of constant reflux ratio and regulating reflux ratio on product composition.
Experiment 7

Liquid-Liquid Extraction

1.0 OBJECTIVES OF EXPERIMENT

- To understand the principles of liquid-liquid extraction.
- To determine the amount of solute extracted from feed.
- To determine the efficiency of extraction.
- To investigate the effects of feed and solvent flow rates on extraction.

2.0 INTRODUCTION

When separation by distillation is ineffective or very difficult, liquid extraction is one of the main alternatives to consider. Close-boiling mixtures or substances that cannot withstand the temperature of distillation, even under a vacuum, may also be separated by extraction. Extraction utilises chemical differences instead of vapour pressure differences for separation.

Liquid-liquid extraction, also known as solvent extraction, is a method to withdraw solute in a liquid mixture using a liquid solvent, in which the solvent is immiscible or nearly immiscible with the liquid. The simplest liquid-liquid extraction involves only a ternary system. The feed consists of two miscible components, the carrier, C and the solute, A. Solvent, S is a pure component. C and S are immiscible or only partly miscible. A is soluble in C and completely or partially soluble in S. During extraction, solute A is transferred from the feed to solvent S, with less transfer of carrier C to S, or S to feed.

![Figure 2.1: A Typical Liquid-Liquid Extraction Column](image-url)
A general extraction column consists of two inlets and two outlets, as shown in Figure 2.1. Usually, the inlets consist of a feed stream from the top of column, which contains the solute to be extracted, and a solvent feed from the bottom which extracts the solute from the feed. After mixing the feed and the solvent, the two phases are separated either by gravity or centrifugal forces. The solvent that contains the extracted solute leaves the top of the column and is referred to as the extract stream. The solution that exits from the bottom of the column which contains only a small amount of solute is referred as raffinate.

In liquid-liquid extraction, as in gas absorption and distillation, two phases must be brought into good contact to permit transfer of material between the phases. In extraction the two phases have comparable densities, so the energy available for mixing and separation is small. The viscosities of both phases are also relatively high and linear velocities through most extraction equipment are low. Therefore, in some cases, mechanical mixing and separation are necessary.

### 2.1 Extraction Equipment

Generally, industrial extraction equipment can be divided into 4 types: mixer-settler, columns with agitator, columns without agitator and centrifugal extractors. The table below shows the advantages and disadvantages of these extractors.

<table>
<thead>
<tr>
<th>Types</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixer-Settler</td>
<td>➢ high efficiency</td>
<td>➢ high set-up cost</td>
</tr>
<tr>
<td></td>
<td>➢ low head room</td>
<td>➢ large scale</td>
</tr>
<tr>
<td></td>
<td>➢ induces good contacting</td>
<td>➢ high operation cost</td>
</tr>
<tr>
<td></td>
<td>➢ can handle any number of stages</td>
<td></td>
</tr>
<tr>
<td>Column without Agitator</td>
<td>➢ low investment costs</td>
<td>➢ high head room</td>
</tr>
<tr>
<td></td>
<td>➢ low operating costs</td>
<td>➢ difficult to scale up from lab</td>
</tr>
<tr>
<td></td>
<td></td>
<td>➢ less efficient than mixer-settler</td>
</tr>
<tr>
<td>Column with Agitator</td>
<td>➢ good dispersion</td>
<td>➢ difficult to separate small density differences</td>
</tr>
<tr>
<td></td>
<td>➢ low investment costs</td>
<td>➢ does not tolerate high flow ratios</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Centrifugal Extractors</td>
<td>➢ can separate small density differences</td>
<td>➢ high set-up, operating and maintenance costs</td>
</tr>
<tr>
<td></td>
<td>➢ short holding time</td>
<td>➢ cannot handle many stages</td>
</tr>
<tr>
<td></td>
<td>➢ small liquid inventory</td>
<td></td>
</tr>
</tbody>
</table>
2.2 Theory – Material Balance on Solute

To obtain the simplest result, it is preferable to express compositions of the solute as mass/mole ratios instead of mass/mole fraction.

Let  \( F_A \) = feed rate of carrier A  
\( S \) = flow rate of solvent C  
\( X_B = \frac{\text{mass/moles of solute B}}{\text{mass/moles of other component in the feed (F), extract (E) or raffinate (R)}} \)

Material balance for Solute B,

\[ B \text{ in } F = B \text{ in } E + B \text{ in } R \]

\[ X_B^{(F)} F_A = X_B^{(E)} S + X_B^{(R)} F_A \]  

(1)

The distribution of solute at equilibrium,

\[ X_B^{(E)} = K'_{DB} X_B^{(R)} \]  

(2)

where \( K'_{DB} \) is the distribution coefficient defined in terms of mass or mole ratios.

Substitute Eq. (2) into (1),

\[ X_B^{(R)} = \frac{X_B^{(F)} F_A}{F_A + K'_{DA} S} \]  

(3)

Extraction factor, \( E_B \), for solute B,

\[ E_B = \frac{S}{F_A} \]  

(4)

The larger is the value of \( E_B \), the greater extent to which the solute is extracted. Large values of \( E_B \) result from large values of \( K'_{DB} \) or large ratios of solvent to carrier, \( S/F_A \). Fraction of B that is not extracted,

\[ \frac{X_B^{(R)}}{X_B^{(F)}} = \frac{1}{1 + E_B} \]  

(5)

This clearly shows that the larger the value of \( E_B \), the smaller the fraction of B that is not extracted.
\( X \) (mass/mole ratios) are related to \( x \) (mass/mole fractions) by

\[
X_i = \frac{x_i}{(1 - x_i)}
\]  

\( K'_D \), in terms of ratio, is related to \( K_D \), in terms of fractions by

\[
K'_D = \frac{x_i^{(1)}/(1 - x_i^{(1)})}{x_i^{(2)}/(1 - x_i^{(2)})} = K_D \left( \frac{1 - x_i^{(2)}}{1 - x_i^{(1)}} \right)
\]

where \( K_D = \frac{x_i^{(1)}}{x_i^{(2)}} \). When \( x_i \) are small, \( K'_D \) approaches \( K_D \).

The efficiency of the extraction process is given by

\[
\text{Separation Efficiency} = \frac{\text{amount of solute B in extract}}{\text{amount of solute B in feed}}
\]

### 3.0 EQUIPMENT – LIQUID-LIQUID EXTRACTION UNIT

The liquid-liquid extraction unit is designed to demonstrate liquid mixture separation by solvent extraction. This unit comprised of a glass extraction column, a distillation column for recovery of solvent (both are packed with Raschig rings), tanks, pumps, water jet vacuum and all the necessary instrumentation.

The liquid mixture to be separated is delivered from the feed tank to the bottom of the extraction column via a gear pump. The solvent is then delivered via a diaphragm pump to the top of the extraction unit to contact with the feed in counter-current flow. The feed mixture to be separated comprises of a transition component (solute) and a carrier liquid. A phase boundary is established in the column because the carrier liquid and solvent are insoluble in one another. The transition component transfers from the carrier liquid into the solvent in the unit. The system is fitted with two three-way valves to enable the unit to operate as a closed or open process.

Separation of the transition component and the solvent is done by the distillation unit. The distillation unit is made up of a glass packed column with a bottom boiler and an overhead condenser. The mixture of the transition component and the solvent is heated up to the desired evaporation temperature by using the boiler with a digital temperature controller.

A thermocouple with digital display is provided for measurement of the column top temperature. Distillation removes the solvent from the transition component, where both are individually collected as top and bottom products. The recovered solvent is then reused for the next extraction process. A pH and conductivity meter is provided to monitor the separation process efficiency.
Figure 3.1: Liquid-Liquid Extraction Column with Solvent Recovery

4.0 OPERATING PROCEDURES

4.1 Pre-experiment Procedures

1. Read and understand the theory of liquid-liquid extraction.
2. Read and understand the equipment used in the experiment (liquid-liquid extraction unit).
3. Read the safety precautions and chemical hazards before conducting the experiment.
4. Read the Material Safety Data Sheet (MSDS) for the chemicals used in the experiment in Appendix A – acetic acid, ethyl-acetate
5. Prepare the following apparatus and materials needed for the experiment:
   - pH meter
   - Burette
   - Erlenmeyer flasks
   - Beakers
   - 1.0 M NaOH solution
   - Phenolphthalein
   - Prepare 20 L of 20 v/v% acetic acid solution
5.0 CHEMICAL HAZARDS, SAFETY AND PRECAUTIONS

5.1 Chemical Hazards (refer MSDS in Appendix A for more details)

- Acetic acid contact with the eyes can cause serious long-term damage. The pure material and its solutions are corrosive; concentrated solutions can cause serious burns.
- Ethyl acetate is very flammable, so constitutes a fire risk. It can be ignited by flames, but also by contact with items such as hot plates or hot air guns.

5.2 Safety Precautions

- Always wear safety glasses, mask and gloves when handling chemicals. Do not allow the solution to come into contact with your skin or eyes.
- Should any chemicals come into contact with the body, rinse off immediately with plenty of water and inform the laboratory instructor/officer. Seek medical treatment if symptoms persist.
- Ensure that there is no source of ignition, such as a Bunsen burner, gas flame, hot plate, hot air gun or hot water pipe near the working area.
- Ensure the heating coil is fully immersed in liquid before switching on the heater.
- Do not touch the boiler and distillation column when conducting the experiment.
- Be careful when using the handheld pH and conductivity meter.
- Do not attempt to change the setting of the pumps.
- Dispose of all unused chemicals in an appropriate manner after the experiment. Under no circumstances should the chemicals be allowed to flow into sinks or drains.
- Wash your hands thoroughly with soap after the experiment.

6.0 EXPERIMENTS

6.1 Experiment 1: Determination of Efficiency of Extraction

1. Connect the 3-pin plug to 240VAC main power supply. Turn on the power supply and main power switch at the front of the control panel.
2. Make sure all the valves are closed.
3. Fill 20 L of 20 v/v% acetic acid solution into the feed tank.
4. Fill 10 L of ethyl-acetate into the solvent tank.
5. Switch on the solvent pump and set the flow rate to 15 L/hr to feed the solvent into the bottom of the column. *(Note: press the up/down soft button to adjust the flow rate, do not turn the turning knob)*
6. Once the solvent level reaches about a third of the height of the column, switch on the feed pump and set the flow rate to 15 L/hr.

7. As the liquid level reaches the top of column reduce the feed and solvent flow rates to 10 L/hr.

8. Open the Gate Valve at the bottom of the extraction column to direct the bottom product to the raffinate tank.

9. Keep an eye at the liquid level; ensure that the liquid level is maintained near the top of the column by adjusting the feed and solvent flow rates.

10. The boundary between the two immiscible liquids (the feed and solvent) is maintained around the middle of the column by adjusting the Gate Valve.

11. After 10 minutes, obtained sample from extract tank and raffinate tank respectively and determine their pH values.

12. Collect 5 ml of sample, from both the extract and raffinate tank, into a Erlenmeyer flasks and determine the amount of acetic acid by titration method with 1.0 M NaOH solution:
   (a) Take the initial NaOH volume.
   (b) Drop a few droplet of phenolphthalein into the sample in Erlenmeyer flask.
   (c) Titrate the solution with NaOH until the sample turn to pink colour.
   (d) Record the NaOH volume after titration.

13. Repeat Step 11 and Step 12 every 10 minutes until 4 sets of samples are collected.

14. Switch off the main power switch at the control panel and power supply after the experiment.

6.2 Experiment 2: Investigate the Effect of Feed and Solvent Flow Rates on Extraction

By using the same setup and chemicals as Experiment 1, design and run an experiment to investigate the effect of feed and solvent flow rates on the efficiency of extraction.

7.0 RESULT ANALYSIS AND DISCUSSION

Discuss all your results. The questions below only serve as a guideline. Your discussion should not only limited to these questions.

1. Sketch a schematic diagram, including all valves and sensors/controllers, for the extraction unit used in the experiment. Label all valves and instruments.

2. Calculate the amount of solute (acid acetic) in the feed, extract and raffinate.

3. Calculate the efficiency of extraction.

4. Discuss the effects of feed and solvent flow rates on the efficiency of extraction based on your experiment. Compared the results with theory.
Experiment 8
Tray Dryer

1.0 OBJECTIVES OF EXPERIMENT

➢ To perform drying test on solids.
➢ To investigate the effects of air velocity on drying rate.
➢ To perform heat and mass transfer analysis of a drying process.

2.0 INTRODUCTION

Drying is one of the food preserving methods beside canning and freezing. It prevents deterioration by reducing the moisture content of food. In the absence of sufficient water, the bacteria which cause food spoilage and decay are unable to grow and multiply. Many of the enzymes which promote undesired chemical changes in the food cannot function without water. Drying of foods is also used to reduce the weight and bulk of food for cheaper storage and transport.

Supervision is required toward the end of the drying process. Each fruit and vegetable has a critical temperature above which a scorched taste develops. The temperature should be high enough to evaporate moisture from the food, but not too high to cook the food.

For effective drying, air should be hot, dry and moving continuously. The water contained in the food will be vaporised by the hot dry air. For this to happen, latent heat of vaporization must be supplied to the process.

There are two important process-controlling factors in the operation of drying: (1) transfer of heat to provide the necessary latent heat of vaporisation and (2) movement of water or water vapour through the food material and then away from to separate the water from the food.

The size of pieces, relative moisture, flow rate and temperature of the moving air will affect the time required for drying. All these factors are inter-related.

Drying processes fall into three categories:

1. Air and contact drying under atmospheric pressure: In air and contact drying, heat is transferred through the foodstuff either from heated air or from heated surfaces. The water vapour is removed with the air.

2. Vacuum drying: In vacuum drying, advantage is taken of the fact that evaporation of water occurs more readily at lower pressures than at higher ones. Heat transfer in vacuum drying is generally by conduction, sometimes by radiation.
3. **Freeze drying**: In freeze drying, the water vapour is sublimed off frozen food. The food structure is better maintained under these conditions. Suitable temperatures and pressures must be established in the dryer to ensure that sublimation occurs.

### 2.1 Process Parameters for Drying

#### 2.1.1 Speed of Process

Foods should be dried rapidly, but not too fast that the outside harden before the moisture inside has a chance to evaporate. Drying must not be interrupted. Once a drying process started, it should not be cooled down and restart again later. Moulds and other spoilage organisms can grow on partly dried food.

#### 2.1.2 Process Temperature

**Dry Bulb Temperature** ($T_{DB}$)
The temperature of the air, measured by a thermometer bulb, is termed as the dry bulb temperature.

**Wet Bulb Temperature** ($T_{WB}$)
If the thermometer bulb is surrounded by a wet cloth, heat is removed by evaporation of the water from the cloth and the temperature falls. The temperature measured by this method is termed as the wet bulb temperature.

The temperature of the air affects the humidity. Higher temperatures reduce the humidity and allow the air to carry more water vapour. The difference between the dry bulb temperature and wet bulb temperature is used to find the relative humidity ($H_R$) of air from the Psychrometric Chart.

**Dew Point Temperature** ($T_{DP}$)
The dew point is the temperature at which air becomes saturated with moisture (100% RH) and any further cooling from this point results in condensation of the water from the air. This happens at night when air cools and water vapour forms as dew on the ground.

During the early stage of the drying process, the air temperature can be relatively high so that moisture can evaporate quickly from the surface of food. Because food loses heat during rapid evaporation, the air temperature can be high without increasing the temperature of the food. But as soon as surface moisture is lost (the outside begins to feel dry) and the rate of evaporation slows down, the food warms up. The air temperature must then be reduced so that the water vapour inside the food can evaporate.


2.1.3 **Humidity and Ventilation**

The dryness of air is termed as humidity. The lower the humidity, the drier is the air.

\[
\text{Humidity, } H = \frac{p_w}{P - p_w \left( \frac{M_w}{M_A} \right)}
\]

(1)

where \( M_w \) = molecular weight of water vapour \( M_A \) = molecular weight of air \( p_w \) = partial pressure of water vapour, Pa \( P \) = total pressure, Pa

Relative Humidity, \( H_R \) is defined as the ratio of partial pressure of water vapour to the vapour pressure of water at the gas temperature,

\[
H_R = \frac{p_w}{P} \times 100\%
\]

(2)

Zero percentage (0%) relative humidity means dry air while hundred percent (100%) relative humidity means saturated air.

In food drying, low \( H_R \) air must be blown over foods since it has the capacity to pick up water vapour from the food and remove it. If high \( H_R \) air is used, it will become saturated rather quickly and therefore unable to pick up further water vapour from the food, thus providing poor efficiency.

Trapped air becomes saturated once it picks up sufficient amount of water vapour from the food. In this case, drying process no longer take place. Thus, moving air must be supplied to ensure that the ventilation in tray dryer is adequate.

**Psychrometric Chart**

Psychrometric Chart is a chart of humidity against temperature for normal temperature and high temperature. The two main axes are temperature (dry bulb) and humidity (humidity ratio). The saturation curve is plotted, dividing the whole area into an unsaturated and a two-phase region.

Any constant temperature line running between the saturation curve and the zero humidity axes can be divided evenly into fractional humidity which will correspond to fractional relative humidity. This chart can be used as the basis of many calculations. It can be used to calculate relative humidity and other properties.

2.1.4 **Uniform Drying**

Food drying takes a little extra effort and attention. Stirring the pieces of food frequently and shifting the racks in the oven or dryer are essential because heat is not uniform in all parts of the dryer. For best results, thin layers of uniformly-sized pieces of food should be spread on the drying racks.
2.2 Mass Balance

The mass going into the tray drier must balance with the mass coming out,

\[ \text{mass in} = \text{mass out} + \text{mass stored} \]  
\[ \text{raw materials} = \text{products} + \text{wastes} + \text{stored materials} \]

If there are no chemical changes occurring in the process, the law of conservation of mass will apply also to each component. For component A,

\[ m_A \text{ in entering materials} = m_A \text{ in the exit materials} + m_A \text{ stored in food} \]

For example, in a process that is producing sugar, if the total quantity of sugar going into the process in the form of sugar cane or sugar beet is not equalled by the total of the purified sugar and the sugar in the waste liquors, then there is something wrong. Sugar is either being burned (chemically changed) or accumulating in the process or else it is going unnoticed down the drain somewhere. In this case,

\[ m_A = m_{AP} + m_{AW} + m_{AS} + m_{AU} \]

where
- \( m_{AP} \) = component A in product
- \( m_{AW} \) = component A in waste
- \( m_{AS} \) = component A stored
- \( m_{AU} \) = component A lost

2.3 Energy (Heat) Balance

Energy is also conserved in food processing operations just like mass balance. The energy going into tray drier must be balanced with the energy coming out and the energy stored.

\[ \text{total energy in} = \text{total energy out} + \text{total energy stored} \]

Energy balances are often complicated because energy can be converted to another form of energy, for example mechanical energy to heat energy, but the total quantities must be balanced.

Heat energy is the most common form of energy. The conservation of heat energy can be illustrated by considering operations such as heating and drying. In these cases, enthalpy (total heat) is conserved. Similar with mass balances, enthalpy balances can be written with respect to the various items of equipment or process stages.

Assuming no appreciable heat is converted to other forms of energy such as work, Figure 2.3 illustrates the heat balance,
Enthalpy, $H$ is referred to a reference level or datum, thus all the quantities are relative to this datum. Energy balances is a matter of considering the various quantities of materials involved, their specific heats, and their changes in temperature or state.

Heat is absorbed or evolved by reactions in food processing but usually the quantities are small when compared with other forms of energy entering into food processing such as sensible heat and latent heat.

**Latent Heat**: the heat required to change the physical state of materials from solid to liquid, liquid to gas, or solid to gas at constant temperature. The units of latent heat are J/kg and total latent heat change is calculated by multiplying the mass of the material, which changes its phase, by the latent heat.

**Sensible Heat**: the heat which is added or subtracted from food materials when foods change their temperature. Sensible heat change is calculated by multiplying the mass with the specific heat and the change in temperature: $mC\Delta T$. The unit for sensible heat is joule, J.

### 2.4 Rate of Drying

![Figure 2.1: Drying Rate](image)
Figure 2.1 shows two distinct phases of drying: (1) the constant rate period and (2) the falling rate period. In the constant rate period the surface of the food remains wet and it can therefore be spoiled by moulds and bacteria. In the falling rate period the surface is dry and the risk of spoilage is much smaller. The food should therefore be dried to a weight that corresponds to the end of the constant rate period as quickly as possible.

\[
\text{Drying rate} = \frac{\text{initial weight} - \text{final weight}}{\text{time interval}}
\]  

(3)

By calculating the drying rate for each period, graph for drying rate versus moisture content can be plotted as shown in Figure 2.2:

![Drying Rate vs. Moisture Content](image)

**Figure 2.2: Drying Rate vs. Moisture Content**

Figure 2.2 gives two important pieces of information: (1) the actual drying rate during the constant rate period which shows the efficiency of the drier, (2) the final moisture content of the dried food which shows whether it will be stable during storage.

The moisture content of both the fresh food and the final dried food can be found by weighing the fresh food, then heating it at 100°C in an oven for 24 hours and reweighing. The moisture content is found as follows:

\[
\text{Moisture Content (\%)} = \frac{\text{initial weight} - \text{final weight}}{\text{initial weight}} \times 100\%
\]  

(4)

Other values of moisture content during the drying period can be found by relating these two results to the weights of food recorded during the drying experiment and applying similar factors to intermediate weights.
2.5 Heat Transfer in Drying

The rates of drying are generally determined by the rates at which heat energy can be transferred to the water in order to provide the latent heats, though under some circumstances the rate of mass transfer (removal of the water) can be limiting. All three of the mechanisms by which heat is transferred – conduction, radiation and convection – may enter into drying. The relative importance of the mechanisms varies from one drying process to another and very often one mode of heat transfer predominates to such an extent that it governs the overall process.

As an example, in air drying the rate of heat transfer is given by:

\[ q = h_s A (T_a - T_s) \]  

(5)

where
- \( q \) = heat transfer rate, J/s
- \( h_s \) = surface heat-transfer coefficient, J/m²·s·°C
- \( A \) = area through which heat flow is taking place, m²
- \( T_a \) = air temperature, °C
- \( T_s \) = temperature of drying surface, °C

To take another example, in a roller dryer where moist material is spread over the surface of a heated drum, heat transfer occurs by conduction from the drum to the foodstuff, thus the equation is

\[ q = UA(T_i - T_s) \]  

(6)

where
- \( U \) = overall heat-transfer coefficient, J/m²·s·°C
- \( T_i \) = drum temperature, °C
- \( T_s \) = temperature of drying surface, °C

The value of \( U \) can be estimated from the conductivity of the drum material and of the layer of foodstuff. Values of \( U \) have been quoted as high as 1800 J/m²·s·°C under very good conditions and down to about 60 J/m²·s·°C under poor conditions.

In cases where substantial quantities of heat are transferred by radiation, the surface temperature of the food may be higher than the air temperature. Estimates of surface temperature can be made using the relationships developed for radiant heat transfer although the actual effect of combined radiation and evaporative cooling is complex. Convection coefficients also can be estimated using the standard equations.

As drying proceeds, the character of the heat transfer situation changes. Dry material begins to occupy the surface layers and conduction will take place through these dry surface layers. These layers are poor heat conductors, thus heat transfer to the drying region progressively becomes more slowly.
2.6 Dryer Efficiency

Energy efficiency in drying process is very important. Energy consumed for drying is a large component of the overall costs. Energy efficiency is defined as the ratio of the minimum energy needed to the energy actually consumed.

Efficiency calculations are useful when assessing the performance of a dryer, looking for improvements, and in making comparisons between the various classes of dryers.

A useful measure for drying efficiency is to look at a heat balance over the air, treating the dryer as adiabatic with no exchange of heat with the surroundings. The heat transferred to the food for drying corresponds to the drop in temperature in the drying air, and the heat which has to be supplied corresponds to the rise of temperature of the air in the air heater. So this adiabatic air-drying efficiency, η can be defined by

\[ η = \frac{T_1 - T_2}{T_1 - T_a} \] (7)

where
- \( T_1 \) = inlet (high) air temperature into the dryer, °C
- \( T_2 \) = outlet air temperature from the dryer, °C
- \( T_a \) = ambient air temperature, °C

The numerator, the gap between \( T_1 \) and \( T_2 \) is a major factor in the efficiency.

3.0 EQUIPMENT – TRAY DRYER

The tray dryer unit is designed to demonstrate the theoretical and practical aspects of solids drying. It consists of a uniform flow tunnel with a variable speed air handler, variable electric heater, drying compartment, instrumentation, and mobile carrier.

The demonstration capabilities of this unit include demonstration of drying rate regime, heat and mass transfer analysis, drying tests on solids, effects of air velocity and temperature on the drying rate etc.

Experiments capabilities include drying test on solids for commercial and industrial use, determination of drying rates, heat transfer analogy and mass transfer analogy.

3.1 Features

- Floor standing unit with variable speed axial fan mounted on one end.
- Electrical heating elements downstream of the fan with regulator.
- Chamber with transparent access door containing rack of trays suspended from a balance mounted on top of the drier; trays total capacity is approximately 4 kg of solids.
- Control panel mounted at the fan end for air speed variation and heater temperature regulation to vary the temperature in the drier.
Measurements of wet and dry bulb temperature and humidity connected to ports before and after the drying compartment.

![Tray Dryer Unit](image)

**Figure 3.1: Tray Dryer Unit**

### 3.2 Temperature Sensors

<table>
<thead>
<tr>
<th>Sensor</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>Dry Bulb Temperature before Tray</td>
</tr>
<tr>
<td>T2</td>
<td>Wet Bulb Temperature before Tray</td>
</tr>
<tr>
<td>T3</td>
<td>Dry Bulb Temperature after Tray</td>
</tr>
<tr>
<td>T4</td>
<td>Wet Bulb Temperature after Tray</td>
</tr>
</tbody>
</table>

### 4.0 OPERATING PROCEDURES

#### 4.1 Pre-experiment Procedures

1. Read and understand the theory of drying.
2. Read and understand the equipment used in the experiment (tray dryer).
3. Read the safety precautions before conducting the experiment.
4. Prepare the following materials needed for the experiment:
   - 1 small packet of rice or sand / soils
   - cloths (to remove the hot trays)
5.0 SAFETY PRECAUTIONS

- Do not touch the chamber when running the experiment. The surface is hot.
- Do not switch ON the heater if the fan is OFF.
- Do not place weight of more than 6 kg on the scale.
- Keep away from the front and rear side of the chamber.

6.0 EXPERIMENTS

6.1 Experiment 1: Drying of Rice / Wet Sand (Low Air Flow Rate)

1. Insert the empty trays into the tray holder.
2. Attach the tray holder to a digital balance to measure the initial weight of trays.
3. Add roughly 80 g of water to 1 kg of rice / dry sand on the tray.
4. Measure the weight of the rice / wet sand to obtain the actual weight of water.
5. The initial moisture content should be more than 20%.
6. Measure the surface area of the sand that is exposed to air.
7. Connect the tray drier to 415VAC 3-phase power supply. Turn on the power supply and main power switch at the front of the control panel.
8. Turn on the heater power and set the heater temperature to 65°C.
9. Turn on the axial fan and set the frequency to approximately 8 Hz.
10. Record the initial values for T1, T2, T3 and T4.
11. At intervals of 10 minutes, record all temperatures and the total weight of rice/sand in the trays.
12. Refer to psychrometric charts for the values of relative humidity at different time interval.
13. The experiment is complete when the total mass of the rice / sand becomes constant.

6.2 Experiment 2: Investigate the Effect of Air Velocity on Drying Rate

By using the same setup from Experiment 1, design and run an experiment to investigate the effect of air velocity on drying rate.
6.3 Experimental Datasheet

Initial mass of rice / dry sand =
Amount of water added =
Total mass (wet sand + trays + tray holder) =

Moisture content percentage = \( \frac{M_{\text{water}}}{M_{\text{sand}} + M_{\text{water}}} \times 100\% \)

Cross sectional area of trays, m\(^2\) =
Axial fan frequency, Hz =
Heater temperature, °C =

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Mass, ( m ) (g)</th>
<th>( T_{1\text{DB}} ) (°C)</th>
<th>( T_{2\text{WB}} ) (°C)</th>
<th>( T_{3\text{DB}} ) (°C)</th>
<th>( T_{4\text{WB}} ) (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
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</table>

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Mass of Evaporated Water (g)</th>
<th>Product Moisture Content (%)</th>
<th>Air Humidity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td></td>
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</tr>
</tbody>
</table>

Useful information:

\( \Delta H^{\text{vap}} \) for water @ 1 atm = 2257 kJ/kg

\( C_p \) for water @ 1 atm = 4.186 kJ/kg°C

\( C_p \) for rice/sand @ 1 atm = ___ kJ/kg°C
7.0 RESULTS ANALYSIS AND DISCUSSION

Discuss all your results. *The questions below only serve as a guideline. Your discussion should not only limited to these questions.*

1. Plot a graph of moisture content (%) versus time.
2. Plot a graph of drying rate versus moisture content (%).
3. From the graphs, discuss on the drying rate.
4. Perform a mass balance around the drying unit.
5. Calculate amount of heat transferred from the air to the rice / dried sand.
6. Calculate the drier efficiency.
Experiment 9
Fluid Mixing

1.0 OBJECTIVES OF EXPERIMENT

- To study the mixing and flow pattern in agitation process
- To determine the speed characteristic and Power/Reynolds numbers for different types of impellers.
- To determine the mixing characteristic and Power/Reynolds numbers for different types of liquid.

2.0 INTRODUCTION

Mixing is the random distribution of particles in separate phases, into and through one another. Mixing also defined as reduction of inhomogeneity such as concentration, temperature and phase to obtain the end result.

Generally in the industry, fluid mixing operations are carried out in batch and are often associated with other processes such as separations, absorptions and chemical reactions. There are different type of mixing mechanisms depending on the degree of mixing needed and the nature of material used. The common mixing mechanisms include dispersion, molecular diffusion, eddy diffusion, convection and Taylor dispersion.

- Dispersion (also known as diffusion) is the act of spreading out of compounds within other compounds.
- Molecular diffusion is defined as the diffusion caused by relative molecular motion, and is characterised by the molecular diffusivity.
- Eddy diffusion or turbulent diffusion is diffusion caused by turbulent flows where eddies created by movement of large groups of molecules take place, and is characterised by the turbulent diffusivity.
- The convection mechanism, also bulk diffusion is the dispersion caused by bulk motion of molecules.
- Taylor dispersion is related and slightly similar to convection, where dispersion is created by a mean velocity gradient.

2.1 Theory

The mixing of liquid are carried out for several reasons:

- to provide suspension of solid particles (solid -liquid dispersion)
- to dissolve gas in small bubbles (gas-liquid dispersion)
- to blend miscible liquids (blending)
to disperse other immiscible liquid (immiscible liquid-liquid dispersion)

to provide and promote heat transfer to another medium

The liquid mixers are differentiated based on several factors:

- The process operation is either a batch or continuous process.
- The degree of mixing required. This determines the size, power and capability of the mixing equipment.
- The nature of the process such as miscibility of solutions, preparation of liquid and the liquids physical properties such as viscosity and acidity.

### 2.2 Stirred / Agitation Vessels

There are few ways to perform mixing in vessels. Most mixers contain an agitator to produce better mixing; mechanical agitation, jets and gas sparging are often used. The different mixer geometries differ with the variety of processing objectives, needs and application, depending on the capacity of tank, properties of fluid and the degree of mixing required.

The mixing in an agitated vessel can be accomplished in either a continuous, batch or fed-batch mode. Mixing can be done under two main flow conditions, i.e. in laminar flow or turbulent flow, depending on the impeller Reynolds number. For process with laminar flow, the Reynolds number is less than 10, while fully turbulent conditions occur at Reynolds number higher than 104.

### 2.3 Agitators / Impellers

Agitator will create a certain flow pattern in the mixer vessel. The flow pattern is highly influential over the mixing process. The impeller/agitator can be classified into 2 types according to the direction of flow generated.

The axial flow impellers, including propellers, pitched blade turbines (PBT) and hydrofoils, generate flow currents that are parallel to the axis of impeller shaft, as a single stage. It is usually used for blending, solids suspension, solids incorporation or draw down, gas inducement and heat transfer.

![Axial Flow Impellers](image)

**Figure 2.1: Axial Flow Impellers**
The radial flow impellers such as flat-blade impeller, disk turbine and hollow-blade turbine create flow in radial path, producing circulating loops above and below the impellers. It is usually used for low to medium viscosity fluids. These impellers provide higher shear and turbulence levels with lower pumping compared to the axial flow impellers. Thus, they are most effective in gas-liquid and liquid-liquid dispersion compared to other single or multiple phase mixing duties.

![Figure 2.2: Radial Flow Impellers](image)

Impellers cause liquid in the vessel to circulate throughout and end at its starting point. These flow patterns difference cause variations in energy dissipation rate and shear rate distribution within the vessel. For example, the axial flow impellers creates higher efficiency in liquid blending through the single circulation loop, while the radial flow impellers is better for dispersion of gas bubbles with dual circulation loops.

A propeller is a high speed impeller with an axial flow. It is more effective in creating persistence current flow, especially when operating in big tanks. A turbine impeller, also known as paddles, pushes the liquid in circles with no vertical motion involved. This creates flow current that travels towards the vessel wall and then downwards or upwards along the wall.

![Figure 2.3: Agitator Arrangements and Flow Patterns](image)
2.4 Agitation Power Consumption

The agitator is driven by the shaft power, which can be estimated by the following equation. The energy supplied to the fluid by agitation system is eventually dissipated as heat.

$$N_p = K \text{Re}^b \text{Fr}^c$$  \hspace{1cm} (1)

where

- $N_p$ = power number = \( \frac{P}{D^2 N \rho} \)
- $P$ = shaft power, W
- $D$ = diameter of agitator impeller, m
- $N$ = speed of agitator, rps
- $\rho$ = liquid density, kg/m$^3$
- $\text{Re}$ = Reynold number = \( \frac{D^2 N \rho}{\mu} \)
- $\mu$ = liquid viscosity, N-s/m$^2$
- $\text{Fr}$ = Froude number = \( \frac{DN^2}{g} \)
- $K$ = constant (depends on agitator type, size & tank geometry)
- $g$ = gravity acceleration, m/s$^2$

The power number, $N_p$, is also a function of impeller blade width, number of blades, blade angel, baffle configuration & impeller elevation. Figure 2.4 and 2.5 are the power curves for single propellers and turbine impellers that can be used to determine the respective power number. Hence, functionality between $N_p$ and Re differs accordingly.

![Figure 2.4: Power Correlation for Single Three Bladed Propellers Baffled](image)
In laminar flow regime (Re < 10), the $N_p$ value is inversely proportional to Re and power depends significantly on viscosity. In turbulent regime (Re > 10000), $N_p$ value is constant and independent of liquid viscosity. In transitional regime (10 < Re < 10000), $N_p$ value only changes slightly.

![Figure 2.5: Power Correlation for Baffled Turbine Impellers, for Tank with 4 Baffles](image)

Density of a mixture may be computed by addition of component densities:

$$\rho_m = \rho_1 V_1 + \rho_2 V_2 + ...$$  \hspace{1cm} (2)

where $V_n$ is the volume fraction of the components.

The viscosity of a mixture may be computed from the viscosities of the ingredients using the following equations for baffled & unbaffled mixers:

Baffled:

$$\mu_m = \mu_1 V_1 + \mu_2 V_2$$

Unbaffled:

$$\mu_m = \mu_m \left(1 + 1.5\frac{\mu_1 V_2}{V_1[\mu_1 + \mu_2]}\right)$$

3.0 EQUIPMENT – FLUID MIXING APPARATUS

The Fluid Mixing Apparatus consists of a plexiglass cylindrical mixing vessel mounted on an epoxy coated mild steel framework. The mixing vessel incorporates a drain tap and can be completely removable to facilitate cleaning/flushing purposes.
An AC motor with gearbox is used to drive the impellers. The speed of the motor can be varied using a solid state speed controller. The speed controller gives a direct reading of the drive shaft speed in Hertz. A load cell is attached to the motor to measure the torque reading, which is shown digitally on a digital torque meter.

Control panel is mounted next to the plexiglass mixing vessel. The meter, controller and other electrical components are placed on the control panel. AC circuit breaker is provided as a safety feature.

A total of six impellers are available for experimental studies. These impellers can be easily fitted to the drive shaft by clamping them to the locking collar.

![Fluid Mixing Apparatus](image)

**Figure 3.1: Fluid Mixing Apparatus**

4.0 OPERATING PROCEDURES

4.1 Pre-experiment Procedures

1. Read and understand the theory of fluid mixing.
2. Read and understand the equipment used in the experiment (fluid mixing apparatus).
3. Read the safety precautions before conducting the experiment.
4. Prepare the following apparatus and materials needed for the experiment:
   - Different agitator shapes / types
   - Measuring tape / ruler
   - 10 L of water
   - 10 L of cooking oil
5.0 SAFETY PRECAUTIONS

- Ensure the set screw at the agitator shaft is fully tightened before starting the experiment.
- Do not touch any rotating parts of the apparatus when running the experiment.
- Switch OFF the apparatus immediately if there is water leakage.
- Stop the motor if the water is split out from the product tank.
- Do not impact the load cell.
- Do not change the setting of the digital meter and motor controller.

6.0 EXPERIMENTS

6.1 Experiment 1: Investigate the Effect of Agitator Frequencies on Mixing

1. *Ensure the main power supply of the apparatus is OFF.*

2. Loosen the set screw at the agitator shaft (I) and remove the agitator shaft from the motor shaft (A).

3. Choose the desired agitator shape. Measure its diameter and width. Fix the agitator to the end of the agitator shaft (I).

4. Insert the agitator shaft to the motor shaft. Tighten it using the set screw. *(Note: Ensure that the set screw is properly tightened.)*

5. Measure the diameter of the tank. Fill the product tank (C) with test fluid. *(Note: The level of the test fluid must be equal to the diameter of the product tank.)*

6. Ensure that there is no leakage from the product tank, fittings and valves.

7. Connect the 3-pin plug to 240VAC main power supply. Turn on the power supply and main power switch at the front of the control panel (D).

8. Tare zero the digital torque meter (E) by pressing the “UP” soft button.

9. Tare zero the maximum and minimum value by pressing the “MAX/MIN” soft button for about 2 second.

10. Start the geared motor by pressing the “pu/EXT” soft button at the motor speed controller (F). Turn the turning knob of the motor speed controller to 5 Hz.

11. Press the “set” soft button follows by the “RUN” soft button.

12. Allow the motor and agitator to run for 20 seconds.

13. Then, tare zero the maximum and minimum value by pressing the “MAX/MIN” soft button for about 2 second.
14. Press on the “MAX/MIN” soft button and record the maximum torque reading. \textit{(Reminder: You must always tare zero the max/min reading before recording the maximum torque reading.)}

15. Repeat the experiment to obtain an average torque reading.

16. Repeat Step 10 to Step 15 using different frequency values.

17. Repeat the experiment using cooking oil.

\textbf{6.2 Experiment 2: Investigate the Effect of Impeller Shapes on Mixing}

1. \textit{Switch OFF the main power supply of the apparatus.}

2. By using the same setup as Experiment 1, repeat the experiment with different agitators for water and cooking oil.

3. Wash the product tank after the experiment.

\textbf{6.3 Experimental Datasheet}

<table>
<thead>
<tr>
<th>Test Fluid =</th>
<th>Impeller Width and Diameter =</th>
<th>Impeller Type =</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>Average Torque (Nm)</th>
<th>Speed (rps)</th>
<th>Re</th>
<th>P (W)</th>
<th>( N_p )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

Useful Information and Equation:

- Gearbox ratio = 5
- Motor Maximum Speed = 2710 rpm
- Motor Speed (rps) = \( \frac{\text{Current Frequency}}{50} \times \frac{\text{Motor Max Speed}}{60} \)
- Actual Speed (rps) = \( \frac{\text{Motor Speed (rps)}}{\text{gear box ratio}} \)
- Reynolds number = \( \frac{D^2N\rho}{\mu} \)
\[ P = \text{torque (Nm)} \times \text{speed (rps)} \]

\[ N_p = \frac{P}{D^5 N^3 \rho} \]

### 7.0 RESULTS ANALYSIS AND DISCUSSION

Discuss all your results. **The questions below only serve as a guideline. Your discussion should not only limited to these questions.**

1. Plot a graph of power versus speed for all the results.
2. Plot a graph of power number versus Reynold number for all the results.
3. From the graphs, discuss the findings.
Experiment 10
Fluidised Bed

1.0 OBJECTIVES OF EXPERIMENT

- To determine the pressure drop across fluidised bed.
- To verify the Carman-Kozeny equation.
- To observed the differences between particulate and aggregative fluidisation.

2.0 INTRODUCTION

Fluidisation is a process in which solid particles or granular materials are transformed from static solid-like state into dynamic fluid-like state through suspension in a gas or liquid. This is accomplished by introducing a gas or liquid flow through a bed of solid particles from the bottom. The fluid will progress upwards through the bed via empty spaces between particles.

There exist a relationship between the bed pressure drop and the fluid velocity, $U$. The bed pressure drop increases as the velocity increases. This is partly due to frictional forces between the moving fluid and the solid particles. With higher velocities, the frictional resistance becomes greater and hence the bed pressure drop increases.

The stages of fluidised particles from fixed bed to fluidized bed as velocities increases are shown in Figure 2.1. As gas velocity increases, the fluidisation of sand particles become more aggressive and it goes through different stages from incipient (minimum), particulate (smooth), aggregative (bubbling), slugging to lean phase with pneumatic transport.

![Figure 2.1: Various Region of Contacting of a Batch of Solids by fluid and Gas](image)
For fluidisation to occur, the resistance of moving gas or liquid needs to exceed the weight of solid particle. As the gas or liquid passes upwards through bed of solids at low flow rate, it is simply percolates through void spaces between stationary particles. Hence, the separation of particles is constant, forming a fixed bed region. In this region, the pressure drop across bed is directly proportional to the velocity.

At higher velocity, a point is achieved where total frictional resistance is equal to apparent weight of particles in bed. The separation of particles increases creating a minimum fluidising velocity point. A fluidised bed region is then formed where the pressure drop becomes constant.

2.1 Theory

2.1.1 Fixed and Fluidised Bed of Water System

The fixed and fluidised bed for a water system is generally characterised by the regular expansion of the bed as the velocity increases from the minimum fluidisation velocity to the terminal failing velocity of the particles. The system is hydrodynamic. In fluidised bed the particles undergo no net movement and are maintained in suspension by the upward flow of water. A water system exhibit particulate fluidisation, the only important exceptions being those composed of fine particles of high density.

2.1.2 Fixed and Fluidised Bed of Air System

Fixed and fluidised bed for an air system is considerably more complex than a water system. It exhibits a gradual from fixed bed to fluidised bed followed by particle transport, without a series of transition regions. The bed expansion and pressure drop is close to the values calculated for ideal systems.

As air velocity increases, the circuit tends to go through various stages as below:

- **Fixed bed** in which the particles remain in contact with one another and the structure of the bed remains stable until the velocity is increased to the point where the pressure drop is equal to the weight per unit area of the particles.

- **Particulate** and regular predictable expansion over a limited range of gas velocities.

- A **bubbling** region characterised by a high proportion of the gas passing through the bed as bubbles which cause rapid mixing in the dense particulate phase.

- A **turbulent** chaotic region in which the gas bubbles tend to coalesce and lose their identity.

- A region where dominant pattern is one of the vertically upward transport of particle, essentially in air circuit. This condition sometimes referred to as **fast fluidisation**, which lies outside the range of true fluidisation.

2.1.3 General Expressions for Flow through Beds in Term of Carman-Kozeny Equation

General expressions for pressure drop and mean velocity for flow through packing in term of voidage and specific surface is given by
$$u = \frac{d_t^2 \frac{\Delta P}{l_t}}{32 \mu}$$  \hspace{1cm} (1)

where  
- \( u \) = mean velocity of the tube  
- \( \mu \) = viscosity of the fluid  
- \( d_t \) = diameter of the tube  
- \( l_t \) = length of the tube

If flow conditions within the bed are streamline, the relation between fluid velocity \( u_c \), pressure drop \((-\Delta P)\) and voidage \( e \) is given by (for fixed bed of spherical particles of diameter \( d \)),

$$u_c = 0.0055 \left( \frac{\rho_s}{\rho_f} \right) \frac{d^3 (1-e^2)}{\mu l} (-\Delta P d^2)$$  \hspace{1cm} (2)

For a fluidised bed, the buoyant weight of the particles is counterbalanced by the frictional drag. So Eq. (2) becomes

$$u_c = 0.0055 \left( \frac{\rho_s}{\rho_f} \right) \frac{d^3 (1-e^2)}{\mu l} \left( \frac{\rho_s - \rho_f}{\mu} g \right)$$  \hspace{1cm} (3)

where  
- \( g \) = acceleration due to gravity  
- \( \rho_s \) = density of particles  
- \( \rho_f \) = density of fluid  
- \( l_t \) = length of the tube

The Carman-Kozeny equation is not suitable when the flow regime is at the point of incipient fluidisation. Pressure gradient for laminar (Re < 10) and turbulent (Re > 2000) flows through a packed bed of mono-sized of spherical particle with diameter \( d_p \), is expressed in the Ergun equation:

$$\frac{(-\Delta P)}{H} = 150 \frac{\mu U}{d_p^2 \varepsilon^3} (1-\varepsilon)^2 + 1.75 \frac{\rho_f U^2}{d_p} \frac{(1-\varepsilon)}{\varepsilon^3}$$  \hspace{1cm} (4)

where  
- \( \frac{(-\Delta P)}{H} \) = pressure gradient  
- \( U \) = fluid velocity  
- \( \varepsilon \) = voidage  
- \( d_p \) = diameter of particle  
- \( \rho_f \) = density of fluid
Ergun equation is a combination of laminar and turbulent component of pressure gradient. Under laminar flow, the first term of the equation dominate and reduced to the Carmen-Kozeny equation. Due to the differences in shapes and packing of the particles, the constant is changed from 180 to 150. The second term dominates when the flow is turbulent. The pressure gradient increases linearly with superficial fluid velocity in laminar flow and is independent of fluid density.

The minimum fluidised velocity, $U_{mf}$ is required in order for fluidisation to occurred. $U_{mf}$ can be obtained by plotting the graph of bed pressure drop, $\Delta P$ versus fluid velocity, $U$ as shown in Figure 2.2, which is represented by point A. OA is the fixed region whereas BC is the fluidised bed region.

![Figure 2.2: Pressure Drop versus Fluid Velocity in Fixed and Fluidised Beds](image)

### 3.0 EQUIPMENT – FIXED AND FLUIDISED BED APPARATUS

The fixed and fluidised bed apparatus consists of two cylindrical acrylic columns, one for water circuit (right) and the other one for air circuit (left), mounted on epoxy coated mild-steel frame. The two circuits are independently controlled and can be operated separately or together according to the experiment requirements.

Water is circulated from a sump tank through a digital flow meter and control valve to the column by a water pump. Two valves are provided in the water circuit, i.e. a control valve to control the water flow rate and a bypass valve to prevent overloading the water pump. The water is returned to the sump tank via an overflow.

A diaphragm air pump is used to supply air to the second column. A control valve is provided to control the air flow rate before entering the column. Each column has tapping points and a digital manometer for measurement of the bed pressure drop.
4.0 OPERATING PROCEDURE

4.1 Pre-experiment Procedures

1. Read and understand the theory of fluidised bed.
2. Read and understand the equipment used in the experiment (fixed and fluidised bed apparatus).
3. Read the safety precautions before conducting the experiment.
4. Prepare the following apparatus and materials needed for the experiment:
   - Air compressor
   - Test materials: sand, carbon grain, acrylic

4.2 Changing Test Material and Cleaning Procedures

1. *Switch OFF the main power supply of the apparatus.*
2. Release the quick release coupling of the column.
3. Loosen the upper and lower removable coupling. *(Note: Be careful with the test material which may leak out from the column.)*
4. Clean and wash the column.
5. Place the column back to its original position and tighten the lower removable coupling.
6. Fill the column with another test material.
7. Tighten the upper removable coupling.
8. Connect the quick releasing coupling back to the pressure points.

5.0 SAFETY PRECAUTIONS

➢ Do not fill the test material more than half the column height.
➢ Do not start the water pump if there is no water in the water tank.
➢ Do not allow the air blower and water pump to run for long period of time if all the valves are fully closed.
➢ Do not attempt to change the setting of the digital meters.

6.0 EXPERIMENT

6.1 Experiment 1A: Determine the Pressure Drop across Fluidised Bed in Water System

1. Ensure the main power supply of the apparatus is OFF.
2. Fill the water tank (located at the back of the apparatus) with water up to 3/4 height.
3. Loosen the upper removable coupling and place the desired test material into the water column. (Reminder: Do not fill the test material more than half the column height.)
4. Tighten the upper removable coupling and the lower removable coupling and ensure that there is no water or air leakage.
5. Ensure the quick released couplings for the pressure drop points are connected properly for both of the water and air column.
6. Connect the 3-pin plug to 240VAC main power supply. Turn on the power supply and main power switch at the control panel. Ensure the MCB/ELCB is switched ON.
7. Switch on the water pump.
8. Set the water flow rate to 0.1 LPM by regulating the water flow control valve and the water bypass valve. Keep an eye on the digital water flow meter.
9. Record the differential pressure from the digital pressure drop meter.
10. Repeat the experiment with 0.1 LPM increment of water flow rate for at least 5 readings.
11. Drain off the water from the column after the experiment.
6.2 Experiment 1B: Investigate the Pressure Drop across Fluidised Bed for Different Materials in Water System

By using the same setup as Experiment 1A, repeat the experiment with different test material (refer to Section 4.2 for procedures to change the test material).

(Reminder: The height of the test material in the column must be the same as the previous experiment.)

6.3 Experiment 2A: Determine the Pressure Drop across Fluidised Bed in Air System

1. Repeat Step 1 to Step 6 in Experiment 1A.
2. Switch on the air blower.
3. Set the air flow rate to 8 LPM by regulating the air flow control valve. Keep an eye on the digital air flow meter.
4. Record the differential pressure from the digital pressure drop meter.
5. Repeat the experiment with 2 LPM increment of air flow rate until few sets of constant pressure drop readings are taken.

6.4 Experiment 2B: Investigate the Pressure Drop across Fluidised Bed for Different Materials in Air System

By using the same setup as Experiment 2A, repeat the experiment with different test material (refer to Section 4.2 for procedures to change the test material).

(Reminder: The height of the test material in the column must be the same as the previous experiment.)

6.5 Experimental Datasheet

Column Inner Diameter $= 46$ mm

<table>
<thead>
<tr>
<th>Test Material:</th>
<th>Flow Rate (LPM)</th>
<th>Differential Pressure (mBar)</th>
<th>Fluid Superficial Velocity (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>
7.0 RESULTS ANALYSIS AND DISCUSSION

Discuss all your results. *The questions below only serve as a guideline. Your discussion should not only limited to these questions.*

1. Sketch a schematic diagram, including all valves and sensors/controllers, for the fluidised bed unit used in the experiment.

2. Plot a graph of pressure drop versus fluid velocity for all the results and discuss the findings.

3. Combine all the graphs into one graph for different test material and discuss the findings.
Appendix A
Material Safety Data Sheet (MSDS)

Ethanoic Acid (Acetic Acid)

<table>
<thead>
<tr>
<th>Common Synonyms</th>
<th>Acetic acid, glacial acetic acid, vinegar (when very dilute)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula</td>
<td>CH₃COOH</td>
</tr>
<tr>
<td>Physical Properties</td>
<td>Form: white semi-transparent solid, or colourless liquid when pure. Often used as a colourless solution in water.</td>
</tr>
<tr>
<td></td>
<td>Stability: stable</td>
</tr>
<tr>
<td></td>
<td>Melting point: 16.7°C</td>
</tr>
<tr>
<td></td>
<td>Boiling Point: 118.1°C</td>
</tr>
<tr>
<td></td>
<td>Water solubility: miscible</td>
</tr>
<tr>
<td></td>
<td>Specific gravity: 1.05</td>
</tr>
<tr>
<td></td>
<td>Flash point: 40°C</td>
</tr>
<tr>
<td>Principal Hazards</td>
<td>➢ Contact with the eyes can cause serious long-term damage.</td>
</tr>
<tr>
<td></td>
<td>➢ The pure material and its solutions are corrosive; concentrated solutions can cause serious burns.</td>
</tr>
<tr>
<td>Safe Handling</td>
<td>Always wear safety glasses. Do not allow the solution to come into contact with your skin.</td>
</tr>
<tr>
<td>Emergency</td>
<td>➢ Eye contact: Immediately flush the eye with plenty of water. Continue for at least ten minutes and call for immediate medical help.</td>
</tr>
<tr>
<td></td>
<td>➢ Skin contact: Wash off with plenty of water. Remove any contaminated clothing. If the skin reddens or appears damaged, call for medical aid.</td>
</tr>
<tr>
<td></td>
<td>➢ If swallowed: Drink plenty of water. Call for immediate medical help.</td>
</tr>
<tr>
<td>Disposal</td>
<td>Small amounts of dilute ethanoic acid can be flushed down a sink with a large quantity of water, unless local rules prohibit this. Larger amounts should be neutralised before disposal.</td>
</tr>
<tr>
<td>Protective Equipment</td>
<td>Always wear safety glasses when handling concentrated or moderately concentrated acid solutions. If you need gloves, butyl rubber or neoprene are suitable.</td>
</tr>
</tbody>
</table>

Source: [http://cartwright.chem.ox.ac.uk/hsci/chemicals/ethanoic_acid.html](http://cartwright.chem.ox.ac.uk/hsci/chemicals/ethanoic_acid.html), August 9, 2004
# Ethyl Acetate

<table>
<thead>
<tr>
<th>Common Synonyms</th>
<th>Ethyl ethanoate, acetic acid ethyl ester, ethanoic acid ethyl ester</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula</td>
<td>CH₃COOC₂H₅</td>
</tr>
<tr>
<td>Physical Properties</td>
<td></td>
</tr>
<tr>
<td>Form</td>
<td>colourless liquid with a sweet, fruity smell</td>
</tr>
<tr>
<td>Stability</td>
<td>stable, but highly flammable</td>
</tr>
<tr>
<td>Melting point</td>
<td>-84°C</td>
</tr>
<tr>
<td>Boiling point</td>
<td>77°C</td>
</tr>
<tr>
<td>Water solubility</td>
<td>soluble</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>0.9</td>
</tr>
<tr>
<td>Flash point</td>
<td>-4°C</td>
</tr>
<tr>
<td>Principal Hazards</td>
<td></td>
</tr>
<tr>
<td>Ethyl acetate is very flammable.</td>
<td></td>
</tr>
<tr>
<td>Safe Handling</td>
<td>Wear safety glasses. Make sure that there is no source of ignition near where you work. The vapour may be ignited by contact with a hot plate or hot water pipe - no naked flame is needed.</td>
</tr>
<tr>
<td>Emergency</td>
<td></td>
</tr>
<tr>
<td>Eye contact</td>
<td>Immediately flush the eye with plenty of water. If irritation persists call for medical help.</td>
</tr>
<tr>
<td>Skin contact</td>
<td>Wash off with water.</td>
</tr>
<tr>
<td>If swallowed</td>
<td>Flush the mouth out with water if the person is conscious. If the amount swallowed is substantial call for medical help.</td>
</tr>
<tr>
<td>Disposal</td>
<td>Small amounts of ethyl acetate can be flushed down a sink with a large quantity of water, unless local rules prohibit this. This material is very flammable, so care must be taken to avoid any build-up of vapour in sink or sewers.</td>
</tr>
<tr>
<td>Protective Equipment</td>
<td>Safety glasses. If gloves are required, use polyvinyl alcohol (PVA).</td>
</tr>
</tbody>
</table>

Source: [http://cartwright.chem.ox.ac.uk/hsci/chemicals/ethyl_acetate.html](http://cartwright.chem.ox.ac.uk/hsci/chemicals/ethyl_acetate.html), January 16, 2004
## Ethyl Alcohol (Ethanol)

<table>
<thead>
<tr>
<th>Common Synonyms</th>
<th>Ethanol, alcohol, grain alcohol, fermentation alcohol, fermentation ethanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula</td>
<td>C₂H₅OH</td>
</tr>
</tbody>
</table>
| Physical Properties | Form : colourless fragrant liquid  \  
 Stability : stable, but highly flammable  \  
 Melting point : -144°C  \  
 Boiling point : 78°C  \  
 Water solubility : miscible in all proportions  \  
 Specific gravity : 2.12  \  
 Explosion limits : 3.3 - 24.5% |
| Principal Hazards | Contact with the eyes can cause considerable irritation.  \  
 "One-off" consumption of small amounts of ethanol is not likely to be harmful, but consumption of large amounts can be (and has been) fatal.  \  
 Chronic (long-term) ingestion of ethanol may lead to damage to a variety of organs, such as the liver, and may increase the risk of cancer.  \  
 Ethanol is very flammable, so constitutes a fire risk. |
| Safe Handling | Wear safety glasses. Ensure that no sources of ignition, such as a gas flame, hot plate or hot air gun, are present in the working area. Check that ventilation is good; use a fume cupboard if possible. |
| Emergency | Eye contact : Flush the eye with plenty of water. If irritation persists call for medical help.  \  
 Skin contact : Wash off with water.  \  
 If swallowed : If the quantity swallowed is large, call for medical help |
| Disposal | Small amounts of ethanol can be flushed down a sink with a large quantity of water, unless local rules prohibit this. Do not forget that this material is very flammable, so precautions must be taken to ensure that flammable vapour does not build up in the sink or drains. |
| Protective Equipment | Safety glasses |

Source: [http://cartwright.chem.ox.ac.uk/hsci/chemicals/ethyl_alcohol.html](http://cartwright.chem.ox.ac.uk/hsci/chemicals/ethyl_alcohol.html), August 9, 2004
Common Synonyms  n-propanol, normal propanol, propan-1-ol, n-propyl alcohol

Formula  C$_3$H$_8$O

Physical Properties  
Form : colourless liquid
Stability : stable, but highly flammable
Melting point : $-127^\circ$C
Boiling point : 97$^\circ$C
Water solubility : high
Specific gravity : 0.8
Flash point : 15$^\circ$C
Explosion limits : 2.1% - 12%

Principal Hazards  
- Propanol is very flammable. It evaporates readily, so it is possible for dangerous levels of vapour to build up, perhaps reaching a point at which an explosion is possible if a source of ignition is present.
- If propanol is in contact with oxygen over a long period, explosive peroxides may be formed. These typically have a higher boiling point than propanol, so may become concentrated in the liquid if propanol is distilled. Therefore, bottles of propanol, once opened, should not be stored indefinitely, in order to avoid any risk of peroxide formation.

Safe Handling  
Wear safety glasses. Ensure that there is no source of ignition, such as a Bunsen burner, hot plate or hot air gun, near the working area. Good ventilation is essential; use of a fume cupboard is desirable.

Emergency  
- Eye contact : Immediately flush the eye with plenty of water. If irritation persists call for medical help.
- Skin contact : Wash off with water. Remove any contaminated clothing. Note that clothing soaked in propanol will present a significant fire risk, so should be removed immediately and placed in a safe location outside the building or in a fume cupboard.
- If swallowed : Call for medical help.

Disposal  
Small amounts of propanol can be flushed down a sink with a large quantity of water, unless local rules prohibit this. All traces of propanol should be washed from the sink, or it may evaporate and present a fire hazard. Larger amounts should be stored for disposal.

Protective Equipment  
Safety glasses. Gloves are not normally required when handling this chemical; if desired, neoprene, nitrile or butyl rubber are suitable.

Source: [http://cartwright.chem.ox.ac.uk/hsci/chemicals/1-propanol.html](http://cartwright.chem.ox.ac.uk/hsci/chemicals/1-propanol.html), September 30, 2004
# 2-propanol

<table>
<thead>
<tr>
<th>Common Synonyms</th>
<th>Isopropanol, isopropyl alcohol, rubbing alcohol</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Formula</strong></td>
<td>CH$_3$CHOHCH$_3$</td>
</tr>
</tbody>
</table>
| **Physical Properties** | Form : colourless liquid with an alcohol smell  
          Stability : stable, but highly flammable  
          Melting point : -89°C  
          Boiling point : 82°C  
          Water solubility : high  
          Specific gravity : 0.79  
          Flash point : 12°C |
| **Principal Hazards** | 2-propanol is very flammable. It can be ignited by flames, but also by contact with such items as hot plates or hot air guns. |
| **Safe Handling** | Wear safety glasses. Make sure that the area in which you work is well ventilated, so that it is not possible for high concentrations of the vapour to form. Work well away from possible sources of ignition, such as hot plates and Bunsen burners. |
| **Emergency** | ➢ **Eye contact**: Immediately flush the eye with plenty of water. If irritation persists, call for medical help.  
              ➢ **Skin contact**: Wash off with soap and water. Remove any contaminated clothing in a safe area. Be especially careful if a large volume of 2-propanol has been spilled on clothes, since there is the risk of very serious burns if the clothing catches fire. If the skin reddens or appears damaged, call for medical aid.  
              ➢ **If swallowed**: Call for medical help. |
| **Disposal** | Small amounts of 2-propanol can be flushed down a sink with a large quantity of water, unless local rules prohibit this. However, this material constitutes a fire risk; if large amounts of it are flushed down a sink dangerous concentrations may build up in the vapour phase in sewers. If any 2-propanol is poured into a sink, check that it is thoroughly flushed away. If some remains, a high concentration may build up in the air within the sink, presenting a fire risk. |
| **Protective Equipment** | Safety glasses. If you need to use protective gloves, neoprene, nitrile or butyl rubber are suitable. |

Source: [http://cartwright.chem.ox.ac.uk/hsci/chemicals/2-propanol.html](http://cartwright.chem.ox.ac.uk/hsci/chemicals/2-propanol.html), June 22, 2005