CHAPTER I

PRELIMINARY

1.1 Background

Reactors are fundamental units in industrial chemical engineering processes, serving to convert raw materials into products through chemical reactions. Reactors can be classified based on their mode of operation, geometry, and reaction phase. Based on their mode of operation, reactors are known as batch, semi-batch, and continuous reactors. In terms of their geometry, they are classified as stirred tank reactors, column reactors, and fluidised bed reactors. Meanwhile, based on the reaction phase that occurs inside them, reactors are classified as homogeneous reactors and heterogeneous reactors.

A heterogeneous reactor is a reactor used to react components consisting of at least two phases, such as the gas-liquid phase. Reactors used for gas-liquid phase contact include bubble column reactors and air-lift reactors. These types of reactors are widely used in chemical industry processes with very slow reactions, production processes that use microbes (bioreactors), and in biological waste treatment units using activated sludge.

In reactor design, reaction kinetics must be studied comprehensively with mass, heat, and momentum transfer events to optimise reactor performance. Hydrodynamic phenomena, including gas and liquid hold-up and circulation rate, are important factors related to mass transfer rate. This experiment will study hydrodynamics in an air-lift reactor, particularly in relation to the effects of air flow rate, viscosity, and density on hold-up, circulation rate, and gas-liquid mass transfer coefficient in a sequential batch system.

1.2 Statement of the Problem

(Students determine their own statement of the problem in reactor hydrodynamics practicums).

1.3 Experimental Objectives

After conducting this experiment, students are expected to be able to.

1. Determining the effect of operating conditions variables on gas hold-up (ε).

2. Determining the effect of operating conditions variables on circulation rate (U_L).
3. Determining the effect of operating conditions variables on the gas-liquid mass transfer coefficient (K_{La}).
4. Determining the effect of Na_2SO_3 residence time on the gas-liquid mass transfer coefficient (K_{La}).

1.4 Benefits of the Experiment

1. Students can determine the effect of operating conditions variables on gas hold-up (ε).
2. Students can determine the effect of operating conditions variables on circulation rate (U_L).
3. Students can determine the effect of operating conditions variables on the gas-liquid mass transfer coefficient (K_{La}).
4. Students can determine the effect of Na_2SO_3 residence time on the gas-liquid mass transfer coefficient (K_{La}).

CHAPTER II
LITERATURE REVIEW

2.1 Bubble Column and Air Lift Reactor

A reactor is a device where chemical reactions take place to transform a substance into another with higher economic value. A bubble column reactor is a heterogeneous reactor that facilitates reactions between two substances in different phases, namely liquid–gas. The air-lift reactor is a specific type of bubble column reactor that features liquid circulation induced by the airflow entering the reactor (Im et al., 2019). The bubble column reactor has a very simple design, consisting of a single zone with a sparger installed at the bottom. The presence of the sparger generates fine gas bubbles, enabling mixing and aeration. This simple design makes the bubble column reactor advantageous in terms of lower cost. Unlike the bubble column reactor, the air-lift reactor consists of two interconnected zones. These zones include the riser, which contains the sparger where the gas mixture is dispersed and flows upward, and the downcomer, which does not receive gas and has a downward flow. The circulation in the air-lift reactor provides superior mixing efficiency without the need for mechanical agitation (Uyar et al., 2023). In the downcomer or riser zones, filter plates may be installed on the walls, along with one or two baffles. Thus, there are many possible reactor configurations, each offering distinct advantages and serving different purposes (Widayat, 2004).

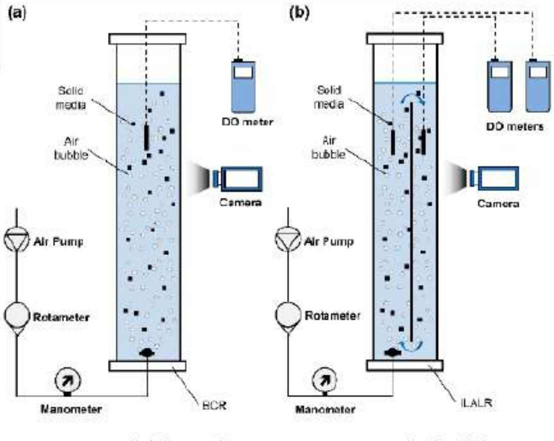

Figure 2.1 Bubble column reactor and air-lift reactor

In general, air-lift reactors are categorized into two main types: internal-loop air-lift reactors and external-loop air-lift reactors (Christi, 1989; William, 2002). The internal-loop air-lift reactor is essentially a bubble column divided into two sections, namely the riser and the downcomer, which are interconnected through internal baffles at both the top and bottom. In contrast, the external-loop air-lift reactor consists of a bubble column in which the riser and downcomer are two separate tubes, horizontally connected at the upper and lower ends of the reactor. Moreover, air-lift reactors can also be classified based on the type of sparger employed, which may be static or dynamic. In dynamic-sparger air-lift reactors, the sparger is positioned within the riser and/or downcomer, and its location can be adjusted as required (Christi, 1989; William, 2002).

Theoretically, air-lift reactors are employed for various gas–liquid or slurry contact processes. These reactors are frequently utilized in aerobic fermentations, wastewater treatment, and other related operations.

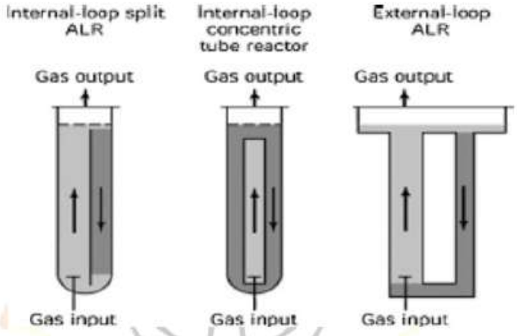

Figure 2.2 Types of air-lift reactor

The advantages of employing an air-lift reactor compared to other conventional reactors include:

1. Its design is simple, with no moving parts.
2. Flow and mixing are easily controlled.
3. Residence time within the reactor is uniform.
4. It provides a larger contact area with minimal input.
5. It enhances mass transfer.
6. It allows for the use of large tanks, thereby increasing product yield.

The disadvantages of air-lift reactors include:

1. High initial investment costs, particularly at large scale.
2. The requirement for high pressure in large-scale processes.
3. Low gas compression efficiency.
4. Inefficient gas–liquid separation due to foam formation.

In the application of air-lift reactors, two fundamental aspects underlie their operating mechanism, namely hydrodynamics and gas–liquid transfer.

2.2 Reactor Hydrodynamics

In the design of bioreactors, several factors exert significant influence, including reactor hydrodynamics, gas–liquid mass transfer, process rheology, and the morphology associated with organism productivity. Reactor hydrodynamics examines the changes in fluid dynamics within the reactor as a consequence of the inlet flow rate and the characteristics of the liquid. Hydrodynamics encompasses gas hold-up (the fraction of gas during dispersion) and liquid circulation rate. The circulation velocity of the liquid is governed by gas hold-up, whereas gas hold-up is affected by the rising velocity of bubbles. Circulation also influences turbulence, the coefficients of mass and heat transfer, as well as the energy generated.

Gas hold-up, or gas void fraction, refers to the volumetric fraction of the gas phase in a gas–liquid dispersion or slurry. The overall gas hold-up (ε) represents the total fraction of gas present within the system.

$$\varepsilon = \frac{V_G}{V_G + V_L} \tag{2.1}$$

Where:

ε = gas hold up
V_G = volume of gas (cm^3/s)
V_L = volume of liquid (cm^3/s)

Gas hold-up is employed to determine the residence time of gas within a liquid. Both gas hold-up and bubble size influence the interfacial area between gas and liquid that is required for mass transfer. Gas hold-up depends on bubble rise velocity, bubble surface area, and flow pattern. An inverted manometer is utilized to measure the liquid level difference caused by gas flow, which is subsequently applied in the calculation of gas hold-up (ε) in the riser and downcomer. The magnitude of gas hold-up in the riser and downcomer can be determined using the following equation.

$$\varepsilon = \frac{\rho_L}{\rho_L - \rho_\varepsilon} \times \frac{\Delta h}{z} \tag{2.2}$$

$$\varepsilon_r = \frac{\rho_L}{\rho_L - \rho_g} \times \frac{\Delta h_r}{z} \tag{2.3}$$

$$\varepsilon_d = \frac{\rho_L}{\rho_L - \rho_g} \times \frac{\Delta h_d}{z} \tag{2.4}$$

Where:

ε = gas hold up
ε_r = hold up gas riser
ε_d = hold up gas downcomer

ρ_L = densiy of liquid (gr/cm^3)

ρ_g = density of gas (gr/cm^3)

Δh_r = riser manometer height difference (cm)

Δh_d = downcomer manometer height difference (cm)

z = difference between pressure taps

The total gas hold-up in the reactor can be calculated from the dispersion height once the gas flow entering the reactor has reached a steady-state condition. The equation for determining the total gas hold-up is expressed as follows.

$$\varepsilon = \frac{h_0 - h_i}{h_0} \tag{2.5}$$

Where:

ε = gas hold up

h_0 = height of gas mixture after steady state (cm)

h_i = height of the initial liquid in the reactor (cm)

The relationship between gas hold-up in the riser (ε_r) and in the downcomer (ε_d) can be expressed by Equation 2.6.

$$\varepsilon = \frac{A_r . \varepsilon_r + A_d . \varepsilon_d}{A_r + A_d} \tag{2.6}$$

Where:

A_r = area of riser zone (cm^2)

A_d = area pf downcomer zone (cm^2)

Liquid circulation in an air-lift reactor is induced by the difference in gas hold-up between the riser and the downcomer. This fluid circulation can be observed from the movement of the liquid, namely the upward flow in the riser and the downward flow in the downcomer. The liquid circulation velocity in the downcomer (U_{Ld}) is represented by Equation 2.7, while the liquid circulation velocity in the riser is represented by Equation 2.8.

$$U_{Ld} = \frac{L_C}{t_C} \tag{2.7}$$

Where:

U_{Ld} = fluid circulation rate at the downcomer (cm/s)

L_C = length of the path in the reactor (cm)

t_C = time (s)

Since the height and volumetric liquid flow in the riser and downcomer are identical, the relationship between the liquid flow rates in the riser and the downcomer is given as follows.

$$U_{Lr}.A_r = U_{Ld}.A_d \tag{2.8}$$

Where:

U_{Lr} = riser fluid circulation rate (cm/s)

U_{Ld} = downcomer fluid circulation rate (cm/s)

A_r = area of riser zone (cm^2)

A_d = area of downcomer zone (cm^2)

The residence times of liquid circulation in the downcomer (t_{Ld}) and in the riser (t_{Lr}) depend on the gas hold-up, as shown in the following equation.

$$\frac{t_{Lr}}{t_{Ld}} = \frac{A_d}{A_r}\frac{1-\varepsilon_r}{1-\varepsilon_d} \tag{2.9}$$

Dimana:

t_{Lr} = residence time of circulating liquid on the riser (s)

t_{Ld} = residence time of circulation liquid on the downcomer (s)

A_r = area of riser zone (cm^2)

A_d = area of downcomer zone (cm^2)

ε_r = gas hold up in the riser zone (cm^2)

ε_d = gas hold up in the downcomer zone (cm^2)

2.3 Mass Transfer

Mass transfer between the gas and liquid phases occurs due to the concentration difference between the two phases. The mass transfer involved is the transport of oxygen from the gas phase to the liquid phase. The rate of this mass transfer can be determined by the mass transfer coefficient.

The volumetric mass transfer coefficient (K_{La}) represents the specific rate of mass transfer (gas absorbed per unit time, per unit interfacial area, per concentration difference). K_{La} depends on the physical properties of the system and fluid dynamics. There are three aspects related to the volumetric mass transfer coefficient:

1. The mass transfer coefficient (K_{La}), which is influenced by the physical properties of the liquid and the fluid dynamics near the liquid surface.
2. The bubble surface area per unit volume of the reactor.
3. The dependence of K_{La} on-energy input is relatively small, as the interfacial area is primarily a function of physical properties, geometric design, and hydrodynamics.

The interfacial area is a bubble parameter that cannot be directly specified. On the other hand, the mass transfer coefficient is essentially a proportional factor between the mass flux and the substrate (or chemical species being transferred), N_s, and the gradient that governs the concentration difference phenomenon. This relationship can be formulated by Equation 2.10.

$$N = K_{La} (C_1 - C_2) \tag{2.10}$$

Where:

N = mass fluks

K_{La} = gas-liquid mass transfer coefficient (1/s)

C_1 = O_2 intake concentration (gr/L)

C_2 = concentration of O_2 out (gr/L)

The transfer of oxygen into the liquid can be formulated as a kinetic process, as presented in Equation 2.11.

$$\frac{dC}{dt} = K_{La}(C_1 - C_2) \tag{2.11}$$

Where:

C = air concentration (gr/L)

The gas–liquid mass transfer coefficient is a function of the air flow rate or the superficial gas velocity, the viscosity, and the cross-sectional area of the riser and downcomer, as well as the reactor geometry.

The measurement of the gas–liquid mass transfer constant can be carried out using the following methods:

1. OTR-Cd Method

   The basic of this method is the mass transfer equation (equation 2.11) all variables expect KoA can be measured. This means that can be used in the oxygen demand system, the oxygen concentration of the gas phase entering and leaving the bioreactor can be analyzed.

2. Dynamic Method

   The method is based on the measurement of the C0i of the liquid, deoxygentaed as a function of time, after the incoming airflow. Deoxygenation can be obtained by passing oxygen through the liquid or stopping the airflow, in this case the need for oxygen in fermentation.

3. Chemical Absorption Method

   This method is based on the chemical reaction of the absroption of gas (O_2, CO_2) with the addition of chemicals in the liquid phase (Na_2SO_3, KOH). This reaction often used in reaction sections where the bulk concentration of the liquid in gas component = 0 and absroption can entrance chemical transfer.

4. Chemical Method OTR-Coi

This method is basically the same as the OTR-Cd method. However, as is well known, some sulfites are continuously added to the liquid if the reaction conditions are maintained in the region where the value of COi can be known. COi can be measured from the addition of sulfite. Also, consumption reaction another oxygen can be used.

5. Sulfite Method

This method is founded upon the reduction reaction of sodium sulfite. The underlying reaction mechanism can be described as follows:

Reaction in reactor:

$$Na_2SO_3 + 0{,}5O_2 \rightarrow Na_2SO_4 + Na_2SO_3 \text{ (unreacted)}$$

Reaction during analysis:

$$Na_2SO_3 \text{ (unreacted)} + KI + KIO_3 \rightarrow Na_2SO_4 + 2KIO_2 + I_2 \text{ (unreacted)}$$

$$I_2 \text{ (sisa)} + 2Na_2S_2O_3 \rightarrow Na_2S_4O_6 + 2NaI$$

Initial moles Na_2SO_3 (a)

$$= \frac{\mathrm{N\ Na_2SO_3}}{\mathrm{eq}} \times \text{reactor volume} \tag{2.12}$$

Mol I_2 *excess* (b)

$$= \frac{\mathrm{N\ KI}}{\mathrm{eq}} \times \text{volume of KI} \tag{2.13}$$

Moles of unreacted Na_2SO_3 (c)

$$= \mathrm{b} - \frac{1}{2}\left(\frac{\mathrm{N\ Na_2S_2O_3}}{\mathrm{eq}} \times \mathrm{V\ Na_2S_2O_3}\right) \tag{2.14}$$

Moles of reacted O_2 (d)

$$= \frac{1}{2} \times (\mathrm{a} - \mathrm{c}) \tag{2.15}$$

O_2 entering the reactor (e)

$$= \frac{\mathrm{d} \times \mathrm{BM\ O_2}}{\mathrm{t} \times 60} \tag{2.16}$$

Gas-liquid mass transfer coefficient (K_{La})

$$= \frac{\mathrm{e}}{0{,}008} \tag{2.17}$$

The constant value of 0.008 in the equation for KLa is obtained from the volumetric O2 transfer coefficient equation as follows:

$$K_{La} = \frac{nO_2}{\Delta C} \tag{2.18}$$

Where:

nO_2 = Mass transfer flux of O2

ΔC = Concentration driving force between the two phases

Reaction:

$$0{,}5\ O_2 + SO_3^{2-} \rightarrow SO_4^{2-}$$

The mass of Na2SO3 required for 1 gram of O2:

$$\frac{1\text{ mole }O_2}{32\text{ g }O_2} \times \frac{1\text{ mole }Na_2SO_3}{0{,}5\text{ mole }O_2} \times \frac{126\text{ g }Na_2SO_3}{\text{mole }Na_2SO_3} = \frac{7{,}875\text{ g }Na_2SO_3}{\text{mole }Na_2SO_3}$$

$$\Delta C = \frac{7{,}875\text{ g }Na_2SO_3}{L} = 0{,}007875\ \frac{\text{g }Na_2SO_3}{L} = 0{,}008\ \frac{\text{g }Na_2SO_3}{L}$$

so, the value KLa is:

$$K_{La} = \frac{nO_2}{\Delta C} = \frac{e}{0{,}008} \qquad (2.19)$$

CHAPTER III

EXPERIMENT METHOD

3.1 Experimental Design

3.1.1 Practicum Design

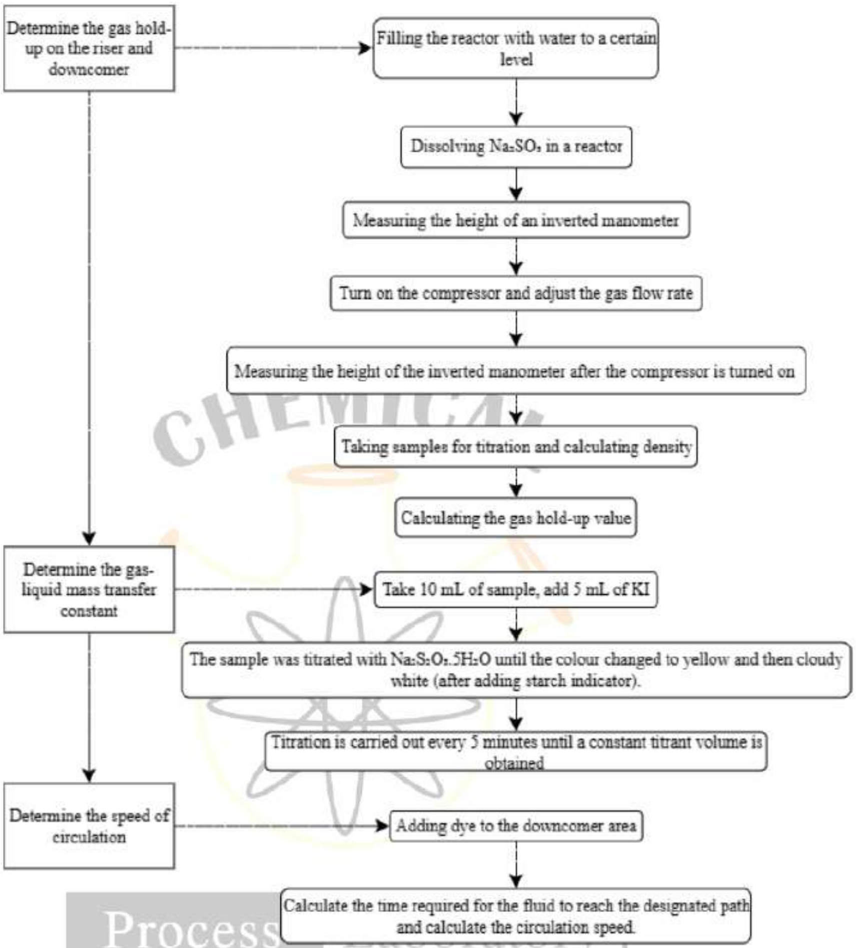

Figure 3.1 Experiment design scheme

3.1.2 Variabel Determination

1. Controlled variable :
2. Independent variable :

3.2 Materials and Tools Used

3.2.1 Materials

1. $Na_2S_2O_3.5H_2O$ 0,1 N
2. KI 0,1 N
3. Na_2SO_3
4. Amylum solution
5. Dyes
6. Aquadest

3.2.2 Tools

1. Burrette, stand, clamp
2. Watch glasses
3. Beaker glass
4. Rotameter
5. Erlenmeyer
6. Inverted manometer
7. Measuring cup
8. Sparger
9. Dropper pipe
10. Liquid tank
11. Compressor
12. Reactor
13. Reagent spoon
14. Picnometer

3.3 Main Toolkit

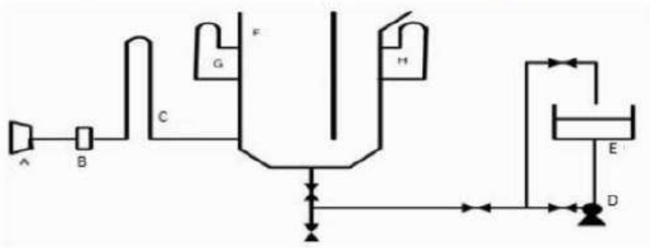

Figure 3.1 The hydrodynamics experimental toolkit

Information:

A. Compressor
B. Sparger
C. Rotameter of riser zone
D. Pump
E. Liquid holding tank
F. Reactor
G. inverted manometer in the riser area
H. Inverted manometer in the downcomer area

3.4 Experimental Procedure

1. Determine gas hold-up riser and downcomer
    - a. Fill the reactor with water and switch on the pump. Once the reactor is filled with water up to … cm, turn off the pump.
    - b. Add sodium sulfite (Na_2SO_3) of … N into the reactor, then wait for 5 minutes to allow the Na_2SO_3 solution to dissolve in water.
    - c. Observe the height of the inverted manometer.
    - d. Turn on the compressor and then check the height of the inverted manometer after the compressor is running.
    - e. Take a sample for titration and measure its density.
    - f. Calculate the gas hold-up.
    - g. Repeat the steps for other operating variables.
2. Determine the gas-liquid mass transfer constant
    - a. Take a 10 mL sample.
    - b. Add 5 mL of KI into the sample.
    - c. Titrate with $Na_2SO_3 \cdot 5H_2O$ … N until the color changes from dark brown to clear yellow.
    - d. Add 3 drops of starch indicator.
    - e. Continue titrating the sample with $Na_2SO_3 \cdot 5H_2O$ … N solution.
    - f. The end point is reached when a cloudy white color appears.
    - g. Record the volume of titrant used.
    - h. Repeat the titration every 5 minutes and stop once three consecutive titrations give constant values.
    - i. Repeat the steps for other operating variables.
3. Determine the speed of circulation
    - a. Assemble the equipment to be used.
    - b. Fill the reactor with water and Na_2SO_3 … N.
    - c. Switch on the compressor.
    - d. Add a sufficient amount of dye into the downcomer section of the reactor.
    - e. Measure the time required for the liquid, indicated by the dye, to travel along the designated path.
    - f. Calculate the circulation velocity.
    - g. Repeat the steps for other operating variables.

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