CHAPTER 1
PRELIMINARY

1.1 Background

The reactor is the main tool in industry that used to chemical process that is to convert raw materials into products. Reactor can be classified on the basis of mode of operation, geometry, and reaction phase. Based on the method of operation, batch, semi batch and continuous reactors are known. If viewed from the geometry, it is divided into stirred tank reactors, column reactor, and fluidization reactor. Meanwhile, when viewed based on the phase reactions that occur in it, the reactor is classified into homogeneous reactor and heterogeneous reactor.

Heterogeneous reactors are reactors used to react a component consisting of at least two phases, such as a gas – liquid phase. The reactor that used for gas – liquid phase contacts, among which are known column reactors bubble column reactor and air – lift reaactor. This type of reactor widely used in chemical industrial processes with very slow reactions, production processes that use microbes (bioreactors), as well as biological waste treatment using activated sludge.

In reactor design, knowledge of reaction kinetics must be studied comprehensively with the event of mass transfer, heat and momentum to optimize reactor performance. Hydrodynamic phenomenon which includes gas and liquid hold – up, circulation rate is the most important factor related to the mass transfer rate. In this experiment will study the hydrodynamics of an air – lift reactor, especially regarding with the effect of air flow rate, viscosity, and density on hold up, the rate of circulation and mass transfer coefficient of gas – liquid in a quantitative system batches.

1.2 Experimental Objectives

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

1. Determine the effect of operating condition variables on hold – up gas (ε).
2. Determine the effect of the operating condition variable on the circulation rate (V_L).
3. Determine the effect of the operating condition variable on the transfer coefficient gas – liquid mass (K_{La}).

4. Determine the effect of residence time of Na_2SO_3 on K_{La}.

1.3 Trial Benefits

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

CHAPTER II
LITERATURE REVIEW

2.1 Buble Column Reactor and Air Lift

A reactor is a device where a chemical reaction takes place to converting one material into another material that has economic value higher. The air lift reactor is a column – shaped reactor with flow circulation. Column filled with liquid or slurry which is divided into two parts namely riser and downcomer. The riser is the column part that always sprayed with gas and has an upward flow. While the downcomers are an area that is not sprayed with gas and has a downward flow. On downcomer or riser zones allow there to be a filter plate on the walal, there are one or two baffles. So many possibillities reactor shapes with different uses and purposes (Widayat, 2004).

In general, air – lift reactors are grouped into two, namely air– lift reactor with internal loop and external loop (Christi, 1989; William, 2002). An air – lift reactor with an internal loop is a bubbly column that divided into two parts, riser and downcomer with internal baffle where the top and bottom of the riser and downcomer are connected. Air – lift reactor with external loop is a bubbly column where the riser and downcomer are two separate tubes connected horizontally between the top and bottom of the reactor. In addition, air – lift reactor are also grouped based on the sparger used, namely static and dynamic. In the air lift reactor with dynamic sparger, the sparger is placed on the riser and/or downcomer that can be changed (Christi, 1989 ; William, 2002).

Theoretically an air – lift reactor is used for several contact processes liquid gas or slurry. This reactor is often used for several fermentations aerobics, sewage treatment and similar operations.

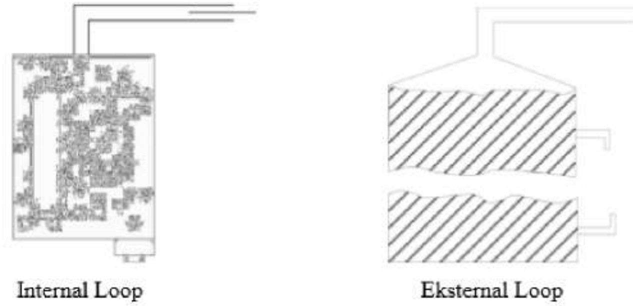

Figure 2.1 Type of Air-Lift Reactor

Advantages of using an air – lift reactor over conventional reaactors others, including :

1. The design is simple, without any moving parts.
2. Flow and stirring are easy to control.
3. The residence time in the reactor is uniform.
4. Wider contact area with low input.
5. Improve mass transfer.
6. Allows a large tank thereby increasing the product.

Weaknesses of the air – lift reactor include :

1. The initial investment cost is expensive especially on a large scale.
2. Requires high pressure of large process scale.
3. Low gas compression efficiency.
4. Separation of gas and liquid is not efficient due to foam (foaming)

In the application of the air – lift reactor, there are two things that underlie the mechanism the work of the reactor, namely hydrodynamics and gas – liquid transfer.

2.2 Reactor Hydrodynamics

In the design of bioreactors, the most influential factors are reactor hydrodynamics, gas – liquid mass transfer, process rheology and morphology organism productivity. Reactor hydrodynamics studies the changes in fluid dynamics in the reactor as a result of the flow rate entering the reactor and the characteristics of the liquid. The reactor hydrodynamics includes gas hold – up (gas fraction dissipation) and the rate of fluid circulation. Fluid circulation speed controlled by hold – up gas, while hold – up gas is affected by speed bubble rise. Circulation also affects turbulence, coefficient transfer of mass and heat and the resulting energy.

The hold – up gas or gas void fraction is the volume fraction of the gas phase in gas – liquid dispresions or slurries. Overall gas hold – up (ε).

$$\varepsilon = \frac{V_\varepsilon}{V_L - V_\varepsilon} \quad \ldots (1)$$

where : ε = hold up gas

V_ε = volume of gas (cc/s)

V_L = volume of fluid (cc/s)

Hold up gas is used to determine the residence time of gas in fluid. Gas hold – up and bubble size affect gas surface area liquid required for mass transfer. Hold up gas depends on bubble rise rate, bubble area and flow patern. Inverted manometer is a manometer used to determine the difference in height liquid due to gas flow, which is then used in the calculation of gas hold – up (ε) on the riser and downcomer. The amount of hold up gas on the riser and downcomer can be calculated by the equation :

$$\varepsilon = \frac{\rho_L}{\rho_L - \rho_\varepsilon} \times \frac{\Delta h}{z} \quad \dots (2)$$

$$\varepsilon_r = \frac{\rho_L}{\rho_L - \rho_g} \times \frac{\Delta h_r}{z} \quad \dots (3)$$

$$\varepsilon_d = \frac{\rho_L}{\rho_L - \rho_g} \times \frac{\Delta h_d}{z} \quad \dots (4)$$

where :

ε = hold up gas

ε_r = hold up gas riser

ε_d = hold up gas downcomer

ρ_L = density of liquid (gr/cc)

ρ_g = gas density (gr/cc)

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 high state dispersion when the gas flow enters the reactor has reached a steady state. The equation to calculate the total gas hold up is as following :

$$\varepsilon = \frac{h_0 - h_i}{h_0} \quad \dots (5)$$

where : ε = hold up gas

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

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

The relationship between hold up gas riser (ε_r) and downcomer (ε_d) can be expressed by equation 6 :

$$\varepsilon = \frac{A_r . \varepsilon_r + A_d . \varepsilon_d}{A_r + A_d} \quad ...(6)$$

where : A_r = area of riser zone (cm^2)

A_d = area of downcomer zone (cm^2)

The circulation of the liquid in the air lift reactor is caused by the difference in hold up riser and downcomer gases. This fluid circulation can be seen from changes in fluid that is increasing fluid flow in the riser and decreasing flow in the downcomer. The magnitude of the fluid circulation rate in the downcomer (U_{Ld}) is shown by equation 7 and the rate of fluid circulation in the riser is given by equation 8 :

$$U_{ld} = \frac{L_C}{t_C} \quad ...(7)$$

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)

Due to the high and volumetric liquid flow in the riser and downcomer is the same, then the relationship between the fluid flow rate in the riser and downcomers are :

$$U_{lr} . A_r = U_{ld} . A_d \quad ...(8)$$

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 reisdence time of t_{ld} and t_{lr} of circulation liquid in the downcomer and riser depends on the hold up gas as shown in the following equation :

$$\frac{t_{lr}}{t_{ld}} = \frac{A_d}{A_r} \frac{1 - \varepsilon_r}{1 - \varepsilon_d} \quad ...(9)$$

where :

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 = hold up gas riser

ε_d = hold up gas downcomer

2.3 Mass Transfer

Mass transfer between gas – liquid phases occurs because of the difference in concentration between the two phases. The mass transfer that occurs is oxygen from the gas phase to the liquid phase. This mass transfer rate can be determined with the mass transfer coefficient.

The volumetric mass transfer coefficient (K_{La}) is the spesific velocity of mass transfer (gas adsorbed per unit time, per unit contact area, per different concentration). K_{La} depends on the physical properties of the system and its dynamics fluid. There are two terms regarding the volumetric mass transfer coefficient, namely :

1. The mass transfer coefficient of K_{La}, which depends on the physical properties of fluids and fluid dynamics close to the liquid surface.
2. Area of bubbles per unit volume of the reactor.
3. The dependence of K_{La} on the incoming energy is small, where the contact area is a function of the physical properties of the geometric and hydrodynamic design.

The contact area is a bubble parameter that cannot be set. In the other hand, the mass transfer coefficient is in fact a very important factor proportional to the mass flux and the substrate (or chemical transferred), N_s and the gradient that affects the phenomenon of different concentrations. This can formulated with equation 10 :

$$N = K_{La}(C_1 - C_2) \quad \ldots(10)$$

where : N = mass flux

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

C_1 = O_2 intake concentration (gr/L)

C_2 = concentration of O_2 out (gr/L)

For mass transfer of oxygen into a liquid can be formulated as process kinetics, as in equation 11 :

$$\frac{dC}{dt} = K_{La}(C_1 - C_2) \quad \ldots(11)$$

where : C = air concentration (gr/L)

The gas-liquid transfer coefficient is a function of the air flow rate or gas superficial velocity, viscosity, and riser area and downcomer/geometric tools. The gas-liquid mass transfer constant can be measured with the following method :

1. OTR-Cd method

   The basic of this method is the mass transfer equation (equation 11) all variables expect K_0A 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 air-flow. Deoxygenation can obtained by passing oxygen through the liquid or stopping the air-flow, 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-C0i

   This method is basically the same as the OTR-Cd method. However, as is well known, some sulfites are continuously added to the liquid as long as the reaction conditions are maintained in the region where the value of C0i can be known. C0i can be measured from the addition of sulfite. Also consumption reaction other oxygen can be used.

5. Sulfite method

   This method is based on the reduction reaction of sodium sulite. Mehcanism reactions that occur :

   Reaction in the reactor :

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

   Reaction during analysis :

   Na_2SO_3 (sisa) + KI + $KIO_3 \rightarrow Na_2SO_4 + 2KIO_2 + I_2$ (sisa)

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

Intial moles Na_2SO_3 (a)

$$= \frac{N\ Na_2SO_3}{eq} \times V\ reaktor$$

Mol I_2 excess (b)

$$= \frac{N\ KI}{eq} \times V\ KI$$

Moles of residual Na_2SO_3 (c)

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

Moles of reacted O_2 (d)

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

O_2 entering the reactor (e)

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

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

$$K_{La} = \frac{e}{0{,}008}$$

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

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

Where:

nO_2 = Mass transfer flux of O_2

ΔC = Concentration driving force between the two phases

Reaction:

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

The mass of Na_2SO_3 required for 1 gram of O_2:

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

$$\Delta C = \frac{7{,}875\ \text{g}\ Na_2SO_3}{L} = 0{,}0078\ \frac{\text{g}\ Na_2SO_3}{L} = 0{,}008\ \frac{\text{g}\ Na_2SO_3}{L}$$

so, the value K_{La} is:

$$K_{La} = \frac{nO_2}{\Delta C} = \frac{e}{0,008}$$

2.4 Uses of Reactor Hydrodynamics in Industry

The following are some of the basic processes in the design and its operation uses the principle of reactory hydrodynamics :

1. Bubble colum reactor

   Examples of bubble column reactor applications include :

   a. Absorption of pollutants with certain substances (eg CO_2 with KOH)

   b. For bioreactor

2. Air lift reactor

   Examples of applications for air-lift reactors include :

   a. The process of producing lactase (an analytical lignin enzyme that can degrade lignin) with microbes.

   b. The process of producing glucan (a polysaccharide composed of monomers) glucose with 1,3 bonds used as raw material for medicine cancer and tumors using microbes.

   c. Water treatment in drinking water treatment.

   d. Biological waster treatment.

CHAPTER III
EXPERIMENT METHOD

3.1 Experimental Design

3.1.1 Practicum Design

3.1.2 Variable Determination

1. Fixed Variable
2. Changing 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

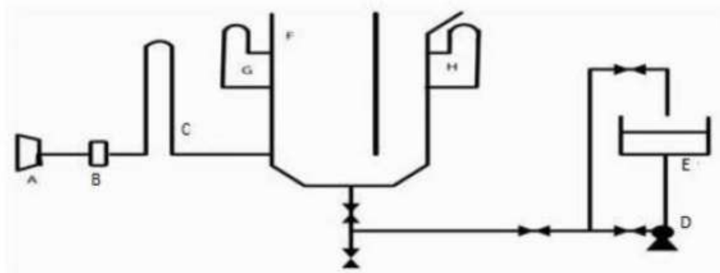

Figure 3.1 The hydrodynamics experimental toolkit

Information :

A. Compressor

B. Sparger

C. Rotameter of riser area

D. Pump

E. Liquid holding tank

F. Reactor

G. Inverted manometer

H. Inverted manometer

3.4 Experimental Procedure

1. Determine hold up riser and downcomer
   a. Fill the reactor with water and turn on the pump. After the reactor filled with water …. cm, turn off the pump.
   b. Add the Na_2SO_3 …. N into the reactor, wait five minutes for the Na_2SO_3 solution is soluble in water.
   c. Look at the height of the inverted manometer.
   d. Turn on the compressor then look at the height of the inverted manometer after the compressor is turned on.
   e. Take a sample for titration and calculate its density.
   f. Calculate the amount of holdup gas
   g. Repeat these steps for the other operating variables.

2. Determine the gas liquid mass transfer constant
   a. Take a sample of 10 ml.
   b. Add 5 ml of KI to the sample.
   c. Titrate with $Na_2SO_3.5H_2O$ …. N until there is a color change from dark brown to clear yellow.
   d. Add 3 drops of starch.
   e. Titrate the sample again with $Na_2SO_3.5H_2O$ …. N solution.
   f. TAT is obtained after the white colour is cloudy.
   g. Record the need for titrant.
   h. Repeat until the titrant volume every 5 minutes is constant.
3. Determine the speed of circulation
   a. Describe the tools used.
   b. Fill the reactor with water and Na_2SO_3….. N.
   c. Turn on the compressor.
   d. Adding dye to the downcomer reactor.
   e. Measuring the time it takes for a liquid with a dye indicator certain way to reach the trajedctory that has been used.
   f. Calculate the magnitude of the circulation speed.

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