Measurement of Wetting Efficiency in Packed Tower Using Stimulus Response Technique.

By

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Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

Supervisor: Prof. Duvvuri Subbarao

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CERTIFICATION OF APPROVAL

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Approved by:

_____

Prof. Duvvuri Subbarao

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
AUGUST 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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Muhammad Izzat Bin Azmi

ABSTRACT

Packed towers are used in many unit operations such as absorption, extraction, distillation, humidification and hydrotreating reactors. Liquid flows down the packed bed by gravity over the packing elements to create gas-liquid / liquid-solid contact area for mass transfer across the phases. Ratio of gas-liquid area to surface area of packing element is known as effective interfacial area; ratio of liquid solid contact area to surface area of packing elements is known as wetting efficiency. Efficiency of a packed tower depends on the wetting efficiency of packing elements. Many researchers investigated wetting efficiency in packed towers. Yet there is still no reliable scientific basis to accurately estimate wetting efficiency. In this investigation, available literature information on wetting efficiency in a packed tower is reviewed and experimentally measured using stimulus response technique of pulse input at three different flow rates using RTD Studies in Packed Bed equipment. The result obtained is compared with other literature data. Through experimental data simple model for estimating wetting efficiency is developed. However further refinement of equation is needed for better accuracy. The data are analysed through a rivulet flow model.

ACKNOWLEDGEMENTS

First and foremost, thanks to God for His blessing in completing this two semesters project. The author would like to outstretch gratitude to Professor Duvvuri Subbarao, Chemical Engineering Department, Universiti Teknologi PETRONAS. It is a privilege to be under his supervision. Even with his tight schedules as lecturer and high commitment to Universiti Teknlogi PETRONAS, there was no moment where he fails to provide support and guidance. His advices and moral support gave a sense of strength and confidence in conducting the final year project.

Many thank to Final Year Project Coordinators, for their unlimited contributions success in providing the students with guidelines and seminars to enlighten hopes of confidence. Many thanks also to all lab executives and technicians as their willingness in providing helps and support during conducting the project.

Last but not least, thanks to all the Universiti Teknologi PETRONAS involved lecturers and students who have been contributing great efforts and ideas making this final year project a success.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Packed tower is fixed-bed of particles in tubular vessel where liquid fall downward by gravity over the fixed bed and in contact with gas transverse counter-current or co-currently over the same bed. These devices are extensively utilized in lots of industries such as fine chemistry, water treatment, and electrochemistry and especially in oil refinery and petrochemical [1]. Packed towers are being utilised in lots of unit operations for examples catalytic gas-liquid reactions, absorption, distillation and water cooling [2]. Below figure shows a typical packed tower absorber:

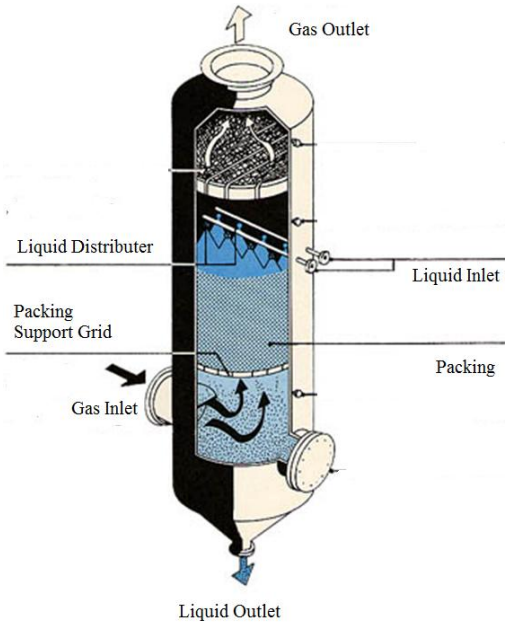


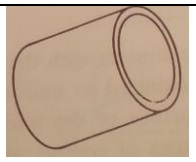
FIGURE 1.1. Typical Packed Tower Absorber.
(Carbo-Tech Environmental Group Inc. (2013).)

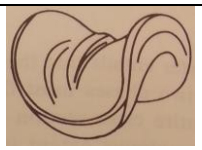
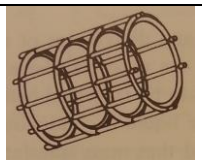
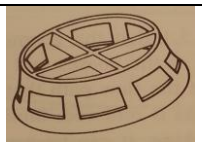
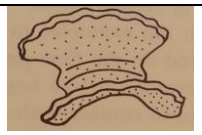
Typical components of a packed tower consist of liquid and gas inlet and outlet, liquid distributors, packing particles and packing support grids. In liquid inlet and outlet, liquid is commonly introduced from the top and allow trickling down by gravity through packing particles and going out at the bottom of the vessel. Whereas for gas inlet can either enter from top or bottom, this is to allow for co-current or counter-current flow of gas with liquid. Liquid distributors on the other hand are used to attain uniformly distribution of liquid over the entire cross-sectional area of packing. Packing support grid is used to hold the packing together inside the vessel.

1.1.1 Packing Elements.

Packing particle are categorised into two main types either random packing or structure packing. In random packing, vessel is filled by random dumped of bed particles which typically used in a small diameter vessel. Whereas for structured packing, it is much more advance as it provides larger effective void space compared to random packing. This will then provide an advantage of lower pressure drops inside the vessel [3]. Below table 1.1 shows several types of random and structured packing which commonly used:

TABLE 1.1**.** Basic structure for random and structured packing.[3]

Packing Particle	Name
	Sphere
	Raschig Ring
	Pall Ring

	Saddle
	Lattice Work
	Shaped Ring
	Treated Surface
	Structured Packing

1.1.2 Working Principle.

In packed tower, flowing gas needs to be brought into intimate contact with liquid flowing on the packing particles in the form of rivulets or films [2]. Through this intimate contact, mass transfer is expected between phases.

Rate of Mass transfer, N is defined as:

$$N = k\ A\ (C_{Liquid} - C_{Gas}) \qquad (1.1)$$

Where: k is mass transfer coefficient.

A is area.

C is concentration.

Mass transfer rate increase with increase in the area of contact between the two phases. Therefore the liquid should wet the fixed bed particles as completely as possible to maximize the mass transfer contact area between phases.

1.1.3 Wetting Efficiency.

The performance of a packed tower depends on the surfaces of packed particles wetted by the liquid phase. Incomplete wetting of packed particles can influence the performance and efficiency of packed tower [5]. The wettings of packed particles are measured base on wetting efficiency. Wetting efficiency is defined as the ratio of gas-liquid contact area to the particle surface area. It has been experimentally observed that wetting efficiency can be less than one depending on the liquid flow rate, type of liquid distributor, particle shape and size and material of construction [2].

1.2 Problem Statement.

The packed tower absorption columns are widely used in petroleum refining, petrochemical, fine chemistry biochemical and other processes [6]. It is necessary to achieve higher wetting efficiency as it affects the performance and efficiency of packed tower. In past decades lots of attempts were made to measure wetting efficiency [1]. Still, there is no clear agreement on scientific basis for the analysis of wetting efficiency.

Therefore, investigation on measuring wetting efficiency needs to be done for better estimation wetting efficiency and to be applied to increase the performance and efficiency of packed tower.

1.3 Objectives.

Objectives of this research are:

1). to investigate response of packed tower by stimulus respond technique using pulse input

2). to compared experimental result with literature data.

3). to develop a simple technique to estimate wetting efficiency in packed towers by pulse response technique

1.4 Scope of Study.

The scope of study of this research involved:

I. Measuring wetting efficiency using Resident Time Distribution (RTD) by stimulus respond technique of pulse input at three different liquid flow rates of 0.5 l/min, 1.2 l/min and 1.9 l/min using RTD studies of Packed Bed Equipment.
II. Comparing the results with the literature data.
III. Developing simple model in estimating the wetting efficiency from RTD by pulse response technique through mass transfer and

CHAPTER 2

LITERATURE REVIEW

2.1 Wetting Efficiency.

Researches on wetting efficiency were done for the past few decades as many attempts were made in developing model for measurement of wetting efficiency inside a packed tower. Among pioneers in this research would be Colombo et al 1976, Mills and Dudukovic 1981, and El-Hisnawi 1982. Even though lots of models/correlations had been developed, unfortunately these proposed correlations suffer discrete results over the same operating conditions range and it is very difficult to choose the more accurate one. Moreover these correlation express wetting efficiency mainly as a function of gas/liquid flow hydrodynamic but none of them include the effects of solid intrinsic wettability [7]. For instance, it is conceived that correlation developed by El-Hisnawi et al. would over predict the value of wetting efficiency at a not too high liquid flow rate, because it would give values exceeding 1.0, whereas based on the Mills and Dudukovic expression is conservative because the value of wetting efficiency would not be 1.0 unless the liquid flow rate is infinitely large [5]. Therefore up until now researches are still being conducted in determining the models/correlations to measure wetting efficiency.

For a qualitative understanding of the wetting phenomenon, direct observation using technique such as dye adsorption [8] and computer assisted tomography are used and did provide an understanding regarding wetting efficiency [9]. For quantitative measurement of wetting efficiency tracer respond technique [10] is being used and found to be reliable.

It has been observed that wetting efficiency dependent few important factors. (eg liquid and gas flow rate, type of liquid distributor,liquid/solid interaction (wettability), operating pressure, particle shape and size). For packed tower absorbers Wang et al (2005) [6] presented a review on the available correlation to estimate the mass transfer coefficient and estimate interfacial area in packed bed. To understand on what affect wetting efficiency of a packed tower in developing a model for wetting efficiency, Baussaron et al, (2007) [1] has performed a parameters study to determine the averaged wetting efficiency. Below are the results of the study:

TABLE 2.1**:** Qualitative effect of studied parameters on average wetting efficiency.

Qualitative effect of studied parameters on averaged wetting efficiency

Studied parameters	Effect on wetting efficiency
Better liquid flow distribution	↗↗
Increase of liquid superficial velocity: V_{SL} ↗	↗↗↗
Increase of gas superficial velocity: V_{SG} ↗	↗
Pressure or gas density increase: P or ρ_G ↗	–
Particle diameter decrease: d_p ↘	↗
Particle shape	–
Bed prewetting	↗↗↗
Liquid/solid wettability ↗	$V_{SL} > 2 \cdot 10^{-3}$ m/s – ; $V_{SL} < 2 \cdot 10^{-3}$ m/s ↗↗

2.2 Hydrodynamic Model for Measurement of Wetting Efficiency.

Subbarao et al (2013) [2] developed a rivulet flow model for the measurement of wetting efficiency in a packed bed. The model suggested that the width of rivulet on a plane surface increases as for increases the liquid flow rate, from this the increase on width of rivulet eventually will spreads all over the bed particle surface.

Through this idea, a simple hydrodynamic experiment was conducted on an incline glass surface. The experiments were conducted to measure the width of rivulet flowing down an incline plane as the function of flow rate. Below figures shows the experimental setup:

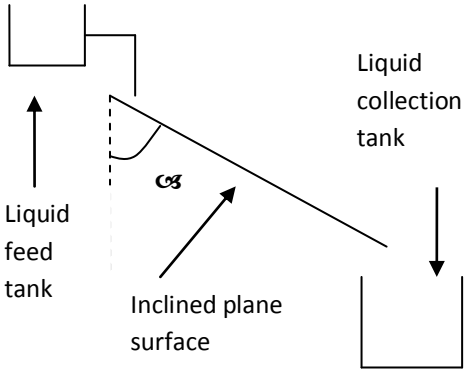


FIGURE 2.1: Hydrodynamic Experimental Setup.

The result of this hydrodynamic test on width of a rivulet as a function of liquid flow rate along with literature data Ataki and Bart (2002) [11] and Luo et al (2009) [12], are correlated with through a friction factor and Reynolds Number. The result gives:

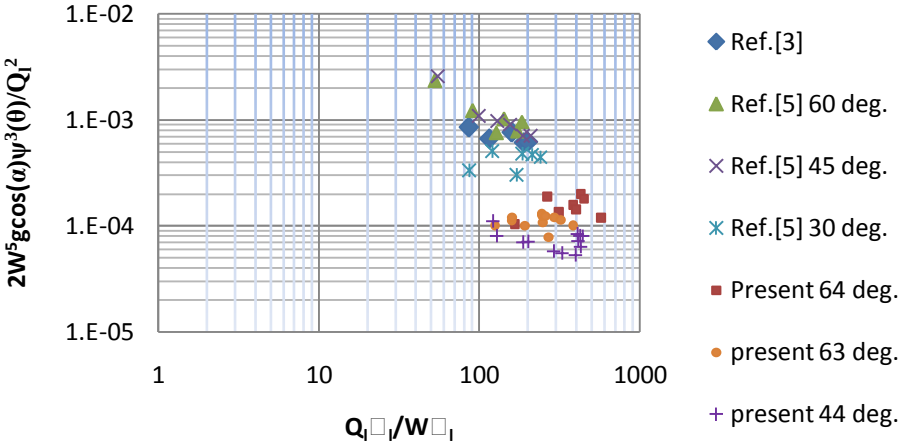


FIGURE 2.2**:** Friction Factor for Rivulet Flow on Inclined surfaces as a function of Reynolds Number

From the figure obtained, friction factor is said to be independent of Reynolds number greater than 3 to 4 hundreds. The model of rivulet flow model was then extended to the surface of packing element as means to develop a model based equation for wetting efficiency in packed tower. Liquid flowing on a single spherical surface is considered. The area wetted by liquid flow is proportional to WD_p.

Therefore wetting efficiency is taken as the ratio of wetted are to particle surface will obtained the following equation:

$$\eta \propto \frac{WD_p}{D_p^2} \propto \frac{W}{D_p} \qquad (2.1)$$

In a complete and uniform liquid distribution, liquid flow in a form of rivulet across packing element Q_1 is expected to be proportional to total liquid flow rate over the number of wetted packing element in the cross sectional plane, which gives:

$$Q_l \propto \frac{Q}{\eta\left[A(1-\varepsilon)/D_p^2\right]} \propto \frac{u_{ol} D_p^2}{\eta(1-\varepsilon)} \qquad (2.2)$$

Wetting efficiency then can be taken as:

For laminar flow

$$\eta \propto \left[\frac{u_{ol}^2}{gD_p}\frac{1}{(1-\varepsilon)^2}\frac{\mu_l(1-\varepsilon)}{D_p\, u_{ol}\rho_l}\right]^{1/5} \qquad (2.3)$$

For turbulent flow

$$\eta \propto \left[\frac{u_{ol}^2}{gD_p}\frac{1}{(1-\varepsilon)^2}\right]^{1/7} \qquad (2.4)$$

These two correlations for laminar and turbulent flow were then validated using literature data by the research done of Julcour-Lebigue et al. (2009) [13]. In which the measurement of wetting efficiency on different effect of liquid viscosity and bed packing size was done.

When their data were compared with laminar flow using equation (2.3) the following results were obtained:

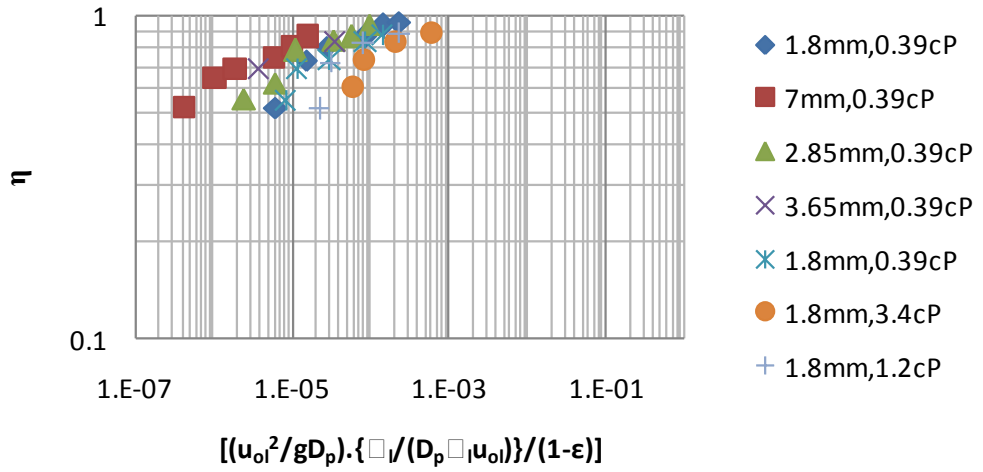


FIGURE 2.3: Evaluation of data of Julcor-Lebique [10] with laminar equation.

For turbulent flow their data were compared with equation (4):

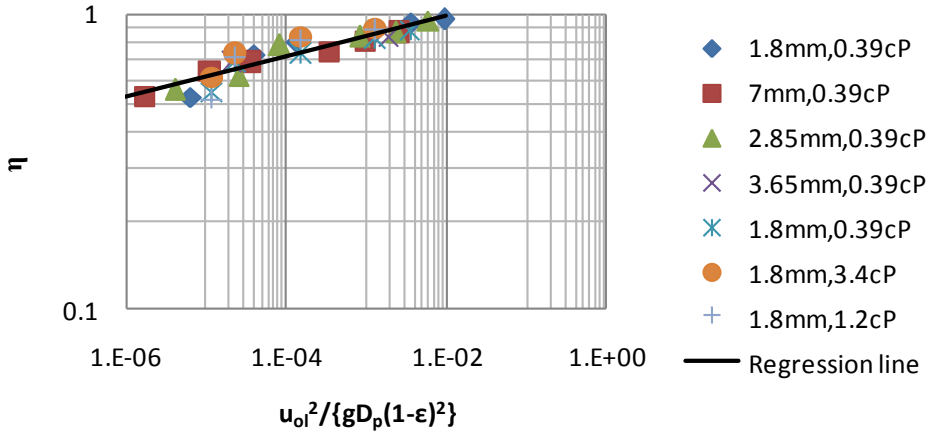


FIGURE 2.4**:** Evaluation of data of Julcor-Lebique [10] with turbulent flow equation

For a laminar flow, there is a lot of scatter in the result obtained. However when it is compared with turbulent flow the data is less scatter which produced a better result. This indicated that the rivulet is flowing in a turbulent regime.

From this it is concluded that wetting efficiency is well correlated with $\left[\frac{u_{ol}^2}{gD_p}\frac{1}{(1-\varepsilon)^2}\right]$

Packing elements undergo complete wetting for $\left[\frac{u_{ol}^2}{gD_p}\frac{1}{(1-\varepsilon)^2}\right]$ greater than or equal to 0.01.

From the developed correlation of $\left[\frac{u_{ol}^2}{gD_p}\frac{1}{(1-\varepsilon)^2}\right]$, it is further refined with inclusion of surface tension and inertial flow control. The equation is validated with the literature data. Below are the result obtained:

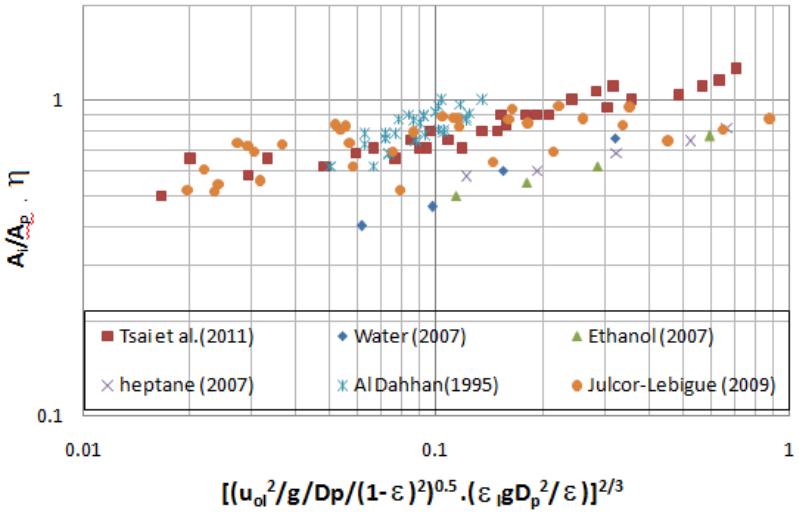


FIGURE 2.5**:** Wetting Efficiency proposed by Subbarao et al inclusion of surface tension and inertial flow control

However yhe result obtained is scattered and better refinement is needed.

2.3 Tracer Method for Measurement of Wetting Efficiency.

Lots of measurements were used in previous years to determine the wetting efficiency, fraction of the external catalyst surface wetted by the flowing liquid, used chemical reaction, dissolution technique, more recently pressure drop, but most preferable and popularly used is dynamic tracer methods [10]. The advantage of dynamic tracer technique is it allows determining wetting efficiency with the actual bed under operation.

Julcour-Lebigue et al 2007 [10], investigates the tracer technique for the measurement of catalyst wetting efficiency in trickle bed reactor. This work was done based on the model proposed by Remachandran et al, 1986. It extended to account for the effects of axial dispersion, liquid-solid mass transfer, pattern of the wetted zone of pellet and distribution of the partial wetting along the reactor. This investigation studies the influence of wetting efficiency on dynamic response, influence of tracer adsorption, wetting heterogeneity and location of wetting zone.

In tracer technique, liquid is introduced at the top of packed tower under operation at the required solvent liquid flow rate. Liquid tracer is then to be introduced to produce step or pulse change. The impulse produce by step or pulse change will be measured at the outlet of the packed tower. The impulse is produced by the packed bed under operation, were analyzed based on time distribution of tracer concentration. From RTD variance, particle effective diffusibilities for reactor operating under full condition and partial wetting regime can be calculated.

Based on previous model developed by Mills and Dudukovic, 1981, wetting efficiency f is deduced from:

$$f= (D_{eapp,\ TB}/D_{epp,\ LF})^{1/2}$$

Where: f, wetting efficiency

$D_{epp,\ LF}$,"true" effective diffusivities.

$D_{epp,\ LF}$, "apparent" effective diffusivities

For using tracer method deriving the exact relation required an appropriate modelling of the tracer diffusion under non-symmetrical condition due to non-uniform mass transfer flux on the outer surface of the catalyst.

Julcour-Lebigue et al developed model for wetting efficiency based on the few assumptions:

- Complete pore filling (i.e., internal wetting) due to capillary forces.
- Spherical catalyst pellets.
- Steady flow (no pulsation).
- The outer surface of the pellet is wetted zone around the north pole and a dry zone underneath:
- Tracer is transferred to the catalyst pores through the wetted zone only.
- Same effective internal diffusivity in radial and angular directions.
- Negligible tracer vapourization.
- Instantaneous and linear adsorption equilibrium.
- Liquid plug flow with axial dispersion.

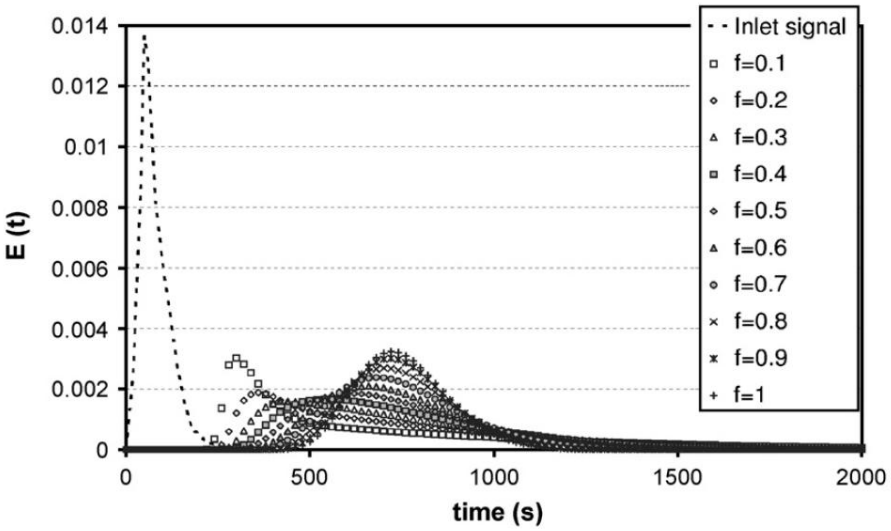


FIGURE 2.6**:** Effect of f on the dynamic respond. [10]

Based on this reseach, it gives the dynamic respond at different measurement of wetting effeicency. Through this, rough estimation for wetting efficiency can determine by analyzing dynamic respond of the system. In this investigation it is also concluded that wetting efficiency can evaluate accurately from RTD data.

2.4 Hydrodynamic Model and Tracer Method.

The hydrodynamic model proposed by Subbarao et al (2013) [2], promised a good result in quantification for the measurement of wetting efficiency. The equation proposed is rather simple and can be easily apply in the industry. Even though the hydrodynamic model had been validated by using research data from other literatures, but yet it had never been apply to a packed tower under operation and the result obtained is scattered. Therefore further refinement is required.

The work of Julcour-Lebigue et al (2007) [10], using tracer technique for the measurement of catalyst wetting efficiency in trickle bed reactor can prove to be an excellent bench mark for the measurement of wetting efficiency. It is also proven in this research that it is one of best method for the estimation of wetting efficiency. Therefore this investigation used the same technique to measure wetting efficiency and develop simple model to estimate wetting efficiency. Later chapter further discuss on the methodology used in this investigation.

CHAPTER 3

METHODOLOGY

This chapter will discuss on the methodology used for this investigation. In this work stimulus response technique of pulse input is used for the measurement of wetting efficiency, by residence time distribution (RTD) studies. The result obtained will be compared with the work of Julcour-Lebigue et al [10] as for the reference. The technique used is the same as the method used by Julcour-Lebigue et al [10]. Fortunately, the equipment needed is already available in UTP laboratory.

3.1 Investigation Approach.

The figure below shows the general experimental approach that was implemented in this investigation:

3.1.1 Defining Investigation Parameters.

Research was started by defining parameters to be investigated. As wetting efficiency is proportional to liquid flow rate, three different flow rates were used in the studies of wetting efficiency. The flow rates used is at 500 ml/min for minimum liquid flow rates, 1200 ml/min for medium liquid flow rate and 1900 ml/min for maximum liquid flow rate.

For the selection of the type of solvent and tracer to be used would be deionised water and 0.2M of NaCl respectively. This is because they are safe and can be easily acquired.

3.1.2 Experiments.

Once the parameters were clearly defined, experiments were conducted. As discussed in earlier chapter tracer technique of pulse input was chosen to measure the wetting efficiency. In tracer technique, residence time distributions for all liquid flow rates were measured. The selected equipment for the measurement residence time distribution is RTD studies in Packed Bed equipment which already available in UTP.

3.1.3 Result Analysis.

Once result is obtained, data will be analyzed to the measurement wetting efficiency based on residence time distribution studies. This available processed data were then compared with the work of Julcour-Lebigue et al [10] as for the experimental result reference point and validation on the measurement of wetting efficiency.

In addition simple model for estimation of wetting efficiency is developed based on the mass transfer principles.

3.2 Raw Materials.

For preliminary experimentation:

I. 0.2 M of salt solutions (NaCl)
II. De-ionized water.

3.3 Equipment Setup.

FIGURE 3.1. RTD Studies of Packed Bed Equipment.

Figure 3.1 shows the equipment setup of RTD studies of Packed Bed equipment which available in UTP laboratory This equipment has a bed length of 150 cm with 8.2 cm internal diameter. The bed particles are made from of 8 x 8 mm plastic raschig ring with bed void fraction of 0.76.

Figure 3.2 shows the schematic diagram of RTD studies of Packed Bed equipment.

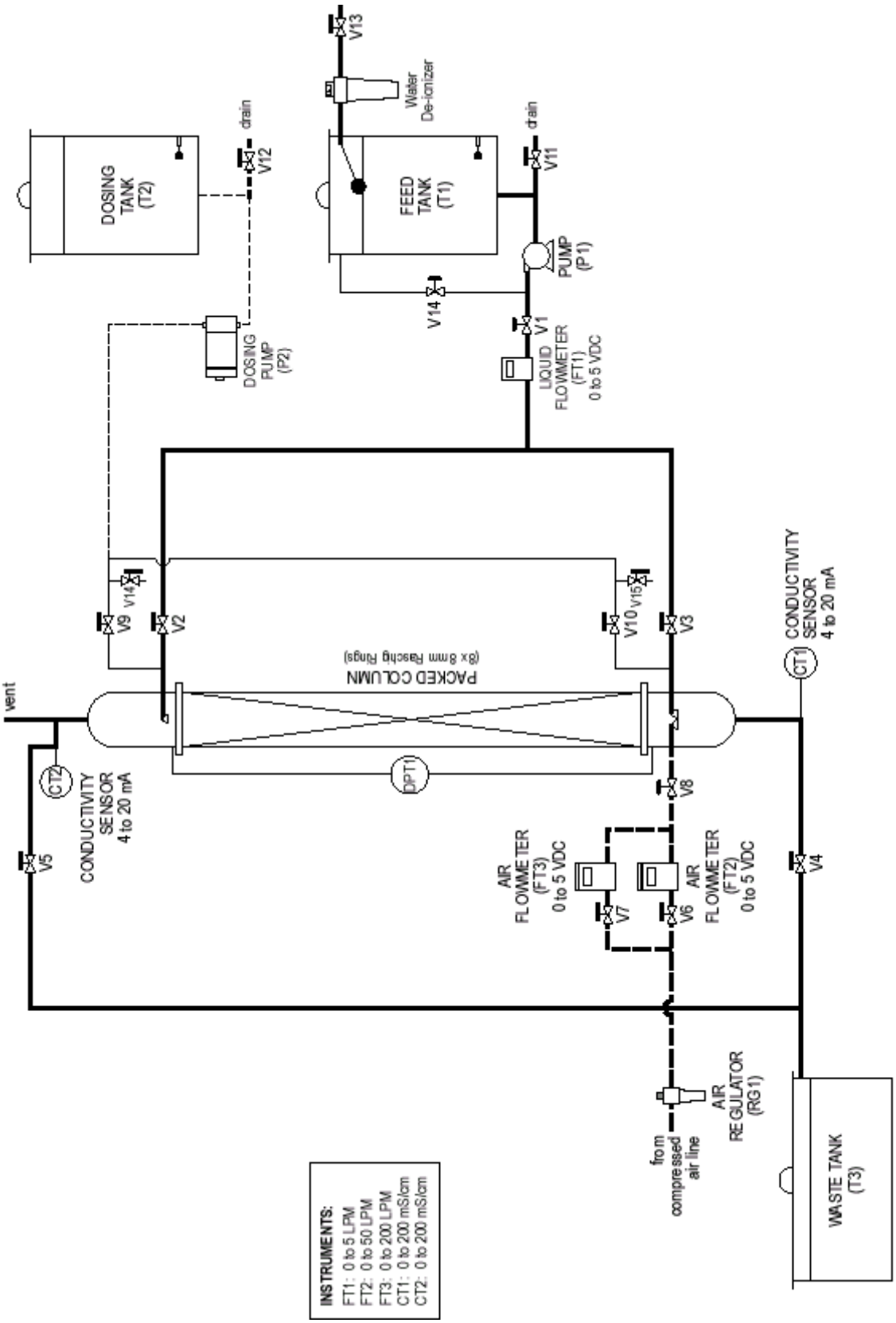


FIGURE 3.2: Schematic RTD Studies of Packed Bed Equipment.

3.4 Theory of the Experiment.

Stimulus response technique is used to experimentally measure Residence Time Distribution (RTD) by injection of an inert chemical, called a *tracer*, into the inlet stream of process equipment and observe its concentration in the outlet stream with time. The two most widely used methods of injection are pulse input and step input. For the purpose of this investigation, pulse input was chosen in measuring wetting efficiency as it is able to give information on how long the individual molecules stay in the packed tower or distribution of residence time.

The tracer concentration is then measured in the effluent stream as a function of time. Besides being a nonreactive species that is easily detectable, the tracer should have physical properties similar to those of the reacting mixture and be completely soluble in the solvent. **Tracer should not adsorb on the surface of packing elements in the reactor. The latter requirements are needed so that the tracer's behaviour will honestly reflect that of the material flowing through reactor.**

In a pulse input, an amount of tracer suddenly injected in one shot into the feed stream entering the reactor in as short a time as possible. The outlet concentration is then measured as a function of time. The distribution of times for stream of liquid exit the vessel is called as the exit age distribution, E(t). Typical exit age distribution curve also referred to as the E-curve in RTD analysis, is used to measure wetting efficiency. Figure 3.3 shows a typical pulse response for any plug flow vessel.

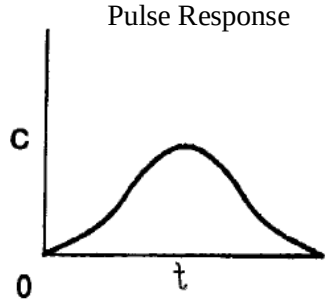


FIGURE 3.3: E-curve.

Once E-curve is produced base on experimental data of pulse input, it will be then be compared with the E-curve obtained by the work of Julcour-Lebigue et al 2007 [10] in determining wetting efficiency.

3.5 E-curve

As discussed in previous section, E-curve is used in analysing the RTD of liquid flow. E curve were plot as E(t) versus t/τ. E(t) can be calculated as:

$$E(t) = \frac{Ct}{area} \tag{3.1}$$

Where area of the curve is approximately to:

$$Area = \sum C\Delta t \tag{3.2}$$

Mean residence time, τ can be calculated by using the following correlation:

$$\tau = \frac{L}{U_{ol}} \tag{3.3}$$

Where: L is bed length

U_{ol} is superficial velocity

3.6 Experimental Procedure.

Experiment was conducted based on three different flow rates at 500 ml/min, 1200 ml/min and 1900 ml/min. For each flow rate the experiment was repeated for three times. Experiments were conducted based on the standard operating procedure (SOP) which available in the appendix.

In the beginning of each experiment, equipment was to be ON first before the computer. Once this was done, preliminary checking were done on the connection of cable, making sure all drain valves were close and making sure the de-ionized water is full. Before the experiment start de-ionized water was flush to make sure there is no trace of salt in the system. Once this was done, experimental setting on computer was then selected base on experiment B: The effect of Pulse Input. The packed bed is then been prewet with de-ionized water. The experiment started as soon as the tracer pump was ON.

The tracer was injected for the first three minutes of the experiment. The equipment measures the outlet concentration of tracer in interval of 1 minute for the maximum time of 2 hours. After each experiment any liquid from the packed bed was drained off, all liquid was disposed off from tank and packed bed was flush with de-ionized water.

E-curve was then constructed based on the result obtained from the experiment. This C-curve was then compared with the work of Julcour-Lebigue et al 2007 [10] to determine wetting efficiency of the packed bed. Based on the flow rate of liquid wetting efficiency was also quantify by using the hydrodynamic correlation proposed by Subbarao et al 2013. The two the wetting efficiency from the work of Julcour-Lebigue et al 2007 and Subbarao et al 2013 was then be compared and discussed.

3.7 Gantt Charts and Key Milestones

This investigation was done based on the schedule and key milestone set at the beginning of this investigation. The schedule was made based on two difference semesters which is Final Year Project I and Final Year Project II. Table 3.1 and 3.2 show the Gantt charts and key milestones for this investigation for Final Year Project I and Final Year Project II respectively.

3.7.1 Gantt Chart and Key Milestones for Final Year Project I

TABLE 3.1: Gantt chart and key milestone for FYP I

No	Descriptions	Week														
		1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Title	■	■													
2	Preliminary Research Work and Literature Review		■	■	■	■	■									
3	Submission of Extended Proposal Defence							●								
4	Preparation for Proposal Defence							■		■						
5	Proposal Defence Oral Presentation									■	■					
6	Detailed Literature Review											■	■	■		
7	Preparation of Interim Report											■	■	■		
8	Submission of Interim Draft Report														●	
9	Submission of Interim Final Report															●

3.7.2 Gantt Chart and Key Milestones for Final Year Project II

TABLE 3.2: Gantt and key milestone for FYP II

No	Description	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Project Work Continue	■	■	■											
2	Submission of Progress Report				●										
3	Project Work Continue					■	■	■	■						
4	Seminar									■	■				
5	Project Work Continue														
6	Poster presentation											●			
7	Submission of technical paper													●	
8	Submission of Dissertation													●	

CHAPTER 4

RESULT AND DISCUSSION

4.1 Stimulus Respond Technique.

This investigation tries to measure the wetting efficiency inside a vessel as it is important one of the most important parameter which can affect the efficiency of a packed tower. To measure wetting efficiency a complete velocity distribution map inside a vessel need to be known, which is currently impractical. Fortunately, knowing how long an individual molecules stay inside a vessel (distribution of residence time) is enough to estimate of liquid pattern flowing inside a vessel.

For this study wetting efficiency is measured by using stimulus respond technique of pulse input. In conducting this investigation Residence Time Distribution studies in Packed Bed equipment was used. Wetting efficiency is to be measured at three different liquid flow rates in the absence of gaseous flow. The experiments were repeated three times, this is to reduce any random error that occurs during conducting experiments. Below table shows the experimental conditions:

TABLE 4.1: Experimental Conditions used for the calculation of wetting efficiency.

Solvent	Deionized Water
Tracer	0.2 M of NaCl solution
Packed Bed Length (cm)	150
Packed Bed Diameter (cm)	8.2
Equivalent Particle Diameter (cm)	0.3
Liquid Flow Rate (ml/min)	500, 1200, 1900
Bed Void Space, dimensionless	0.76

4.2 Raw Experimental Data.

Below tables show raw experimental result for the three flow rates.

TABLE 4.2: Outlet Concentration at liquid flow rate = 500 ml/min.

1st Trial		2nd Trial		3rd Trial	
Time (min)	Concentration (µS)	Time (min)	Concentration (µS)	Time (min)	Concentration (µS)
0	0	0	0	0	0
1	1986.9	1	2610.1	1	1830.2
2	3324.2	2	3536.4	2	3096.2
3	3347.3	3	3593.8	3	2984.9
4	2363.4	4	1567.6	4	1584.4
5	303.6	5	249.8	5	158.9
6	88.8	6	58.2	6	67.2
7	29.5	7	35.5	7	20.5
8	16.0	8	17.1	8	13.8
9	12.9	9	10.7	9	5.6
10	8.5	10	10.3	10	5.5
11	4.3	11	4.4	11	0.9
12	4.3	12	3.7	12	0
13	4.2	13	1.8	13	0
14	0	14	0	14	0

TABLE 4.3: Outlet concentration at liquid flow rate = 1200 ml/min

1st Trial		2nd Trial		3rd Trial	
Time (min)	Concentration (µS)	Time (min)	Concentration (µS)	Time (min)	Concentration (µS)
1	380	1	377.3	1	376.5
2	1852.2	2	1900.9	2	1919.2
3	2082.9	3	2041.2	3	2046.5
4	2120.7	4	2088.4	4	2065.8
5	611.3	5	557.7	5	575.1
6	394.6	6	394.2	6	391.5
7	385.1	7	384.5	7	381.3
8	382.2	8	382.1	8	377.8
9	380.5	9	377.7	9	377.1
10	381.5	10	378.6	10	377.3
11	381.0	11	377.9	11	377.8
12	380.7	12	377.8	12	377.9
13	380.6	13	377.4	13	376.6
14	380.3	14	377.3	14	376.5
15	380.0	15	377.3	15	376.5
16	380.0	16	377.3	16	376.5

TABLE 4.4: Outlet concentration at liquid flow rate = 1900 ml/min.

1st Trial		2nd Trial		3rd Trial	
Time (min)	Concentration (µS)	Time (min)	Concentration (µS)	Time (min)	Concentration (µS)
0	405.3	0	399.3	0	376.5
1	594.1	1	1210.7	1	1251.7
2	1566.2	2	1562.4	2	1526.9
3	1620.6	3	1572.2	3	1561.3
4	818.5	4	797.4	4	813.3
5	434.0	5	431.3	5	412.5
6	409.3	6	403.0	6	381.1
7	408.0	7	401.8	7	377.9
8	408.5	8	401.0	8	377.9
9	408.0	9	400.1	9	378.0
10	406.8	10	400.8	10	377.4
11	406.3	11	399.9	11	377.2
12	405.3	12	399.3	12	376.5
13	405.3	13	377.3	13	376.2
14	405.3	14	377.3	14	376.4

From raw experimental data obtained, E curves with respect to each flow rates are developed for the measurement of wetting efficiency. Section 4.3 discussed on the processed data obtained from the experiment.

4.3 Processed Experimental Data.

4.3.1 Liquid Flow Rate 500 ml/min

First Experimental Trial at flow rate 500 ml/min.

TABLE 4.5: First trial processed data for E-curve at flow rate 500 ml/min

Time (min)	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	0	0	0	0	0
1	1986.9	1986.9	1986.9	0.172865607	0.063093757
2	3324.2	3324.2	6648.4	0.578428558	0.126187515
3	3347.3	3347.3	10041.9	0.873672122	0.189281272
4	2363.4	2363.4	9453.6	0.82248845	0.252375029
5	303.6	303.6	1518	0.132070055	0.315468787
6	88.8	88.8	532.8	0.046355023	0.378562544
7	29.5	29.5	206.5	0.017966052	0.441656301
8	16	16	128	0.011136342	0.504750059
9	12.9	12.9	116.1	0.01010101	0.567843816
10	8.5	8.5	85	0.007395227	0.630937573
11	4.3	4.3	47.3	0.004115226	0.694031331
12	4.3	4.3	51.6	0.004489338	0.757125088
13	4.2	4.2	54.6	0.004750346	0.820218845
14	0	0	0	0	0.883312603

Total Concentration	Total Ct
11493.9	30870.7

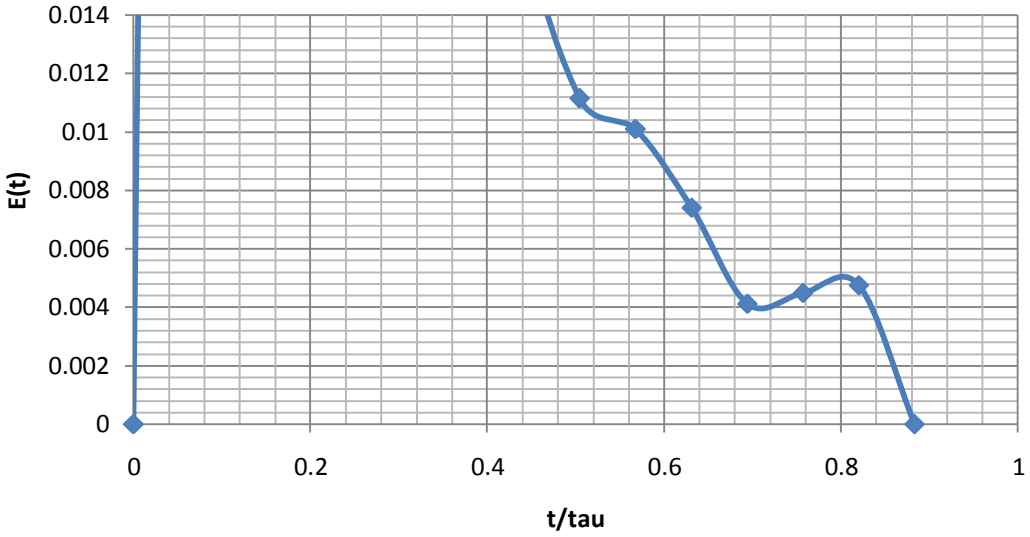


FIGURE 4.1: E-curve of pulse response for 1st Trial at flow rate 500ml/min

Second Experimental Trial at flow rate 500 ml/min

TABLE 4.6: Second trial processed data for E-curve at flow rate 500 ml/min

Time, t	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	0	0	0	0	0
1	2610.1	2610.1	2610.1	0.22310	0.0631
2	3536.4	3536.4	7072.8	0.60454	0.1262
3	3593.8	3593.8	10781.4	0.92153	0.1893
4	1567.6	1567.6	6270.4	0.53596	0.2524
5	249.8	249.8	1249	0.10676	0.3155
6	58.2	58.2	349.2	0.02985	0.3786
7	35.5	35.5	248.5	0.02124	0.4417
8	17.1	17.1	136.8	0.01169	0.5048
9	10.7	10.7	96.3	0.00823	0.5678
10	10.3	10.3	103	0.00880	0.6309
11	4.4	4.4	48.4	0.00414	0.6940
12	3.7	3.7	44.4	0.00380	0.7571
13	1.8	1.8	23.4	0.00200	0.8202
14	0	0	0	0.00000	0.8833
		Total Concentration	Total Ct		
		11699.4	29033.7		

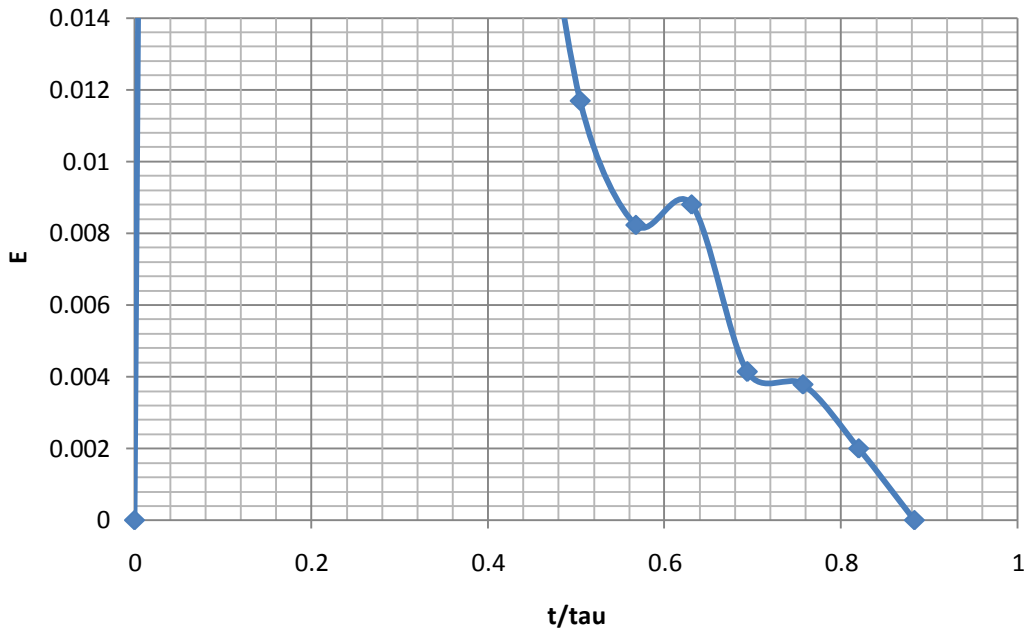


FIGURE 4.2: E-curve of pulse response for 2nd Trial at flow rate 500 ml/min

Third Experimental Trial at flow rate 500 ml/min.

TABLE 4.7: Third trial processed data for E-curve at flow rate 500 ml/min

Time (min)	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	0	0	0	0	0
1	1830.2	1830.2	1830.2	0.18736	0.06309
2	3096.2	3096.2	6192.4	0.63394	0.12619
3	2984.9	2984.9	8954.7	0.91673	0.18928
4	1584.4	1584.4	6337.6	0.64881	0.25238
5	158.9	158.9	794.5	0.08134	0.31547
6	67.2	67.2	403.2	0.04128	0.37856
7	20.5	20.5	143.5	0.01469	0.44166
8	13.8	13.8	110.4	0.01130	0.50475
9	5.6	5.6	50.4	0.00516	0.56784
10	5.5	5.5	55	0.00563	0.63094
11	0.9	0.9	9.9	0.00101	0.69403
12	0	0	0	0.00000	0.75713
		Total Concentration	Total Ct		
		9768.1	24881.8		

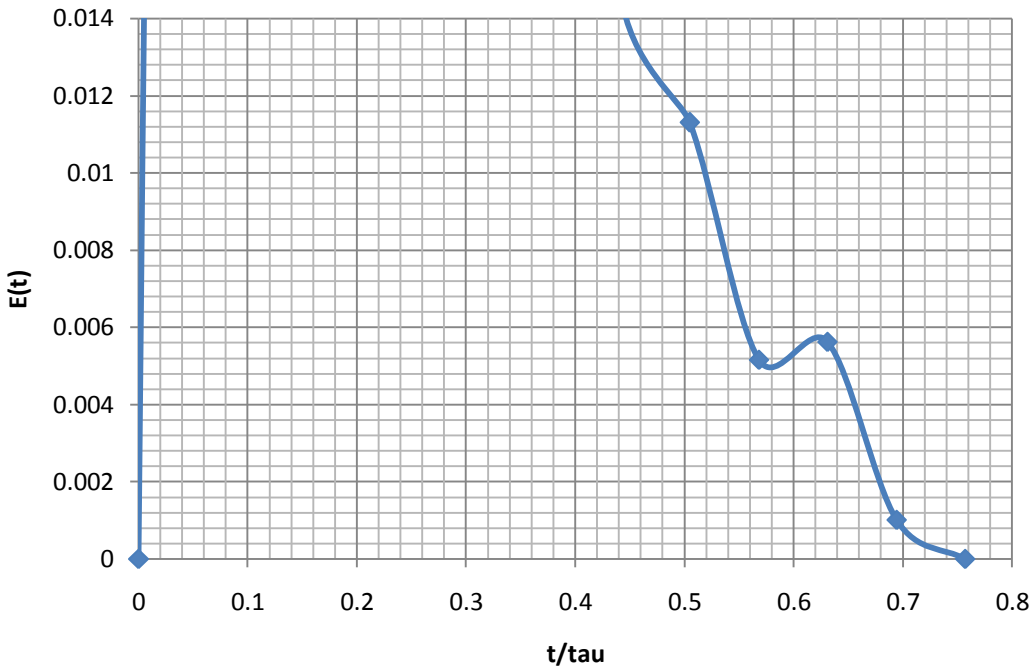


FIGURE 4.3: E-curve of pulse response for 3rd Trial at flow rate 500 ml/min

4.3.2 Liquid Flow Rate 1200 ml/min

First Experimental Trial at flow rate 1200 ml/min.

TABLE 4.8: First trial processed data for E-curve at flow rate 1200 ml/min

Time (min)	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	380	0	0	0	0.1514
1	1852.2	1472.2	2944.4	0.5691201	0.3029
2	2082.9	1702.9	5108.7	0.9874555	0.4543
3	2120.7	1740.7	6962.8	1.3458327	0.6057
4	611.3	231.3	1156.5	0.2235387	0.7571
5	394.6	14.6	87.6	0.0169321	0.9086
6	385.1	5.1	35.7	0.0069004	1.0600
7	382.2	2.2	17.6	0.0034019	1.2114
8	380.5	0.5	4.5	0.0008698	1.3628
9	381.5	1.5	15.0	0.0028993	1.5143
10	381.0	1.0	11.0	0.0021262	1.6657
11	380.7	0.7	8.4	0.0016236	1.8171
12	380.6	0.6	7.8	0.0015077	1.9685
13	380.3	0.3	4.2	0.0008118	2.1200
14	380.0	0	0	0	2.2714
15	380.0	0	0	0	2.4228
		Total Concentration	Total Ct		
		5173.6	16364.2		

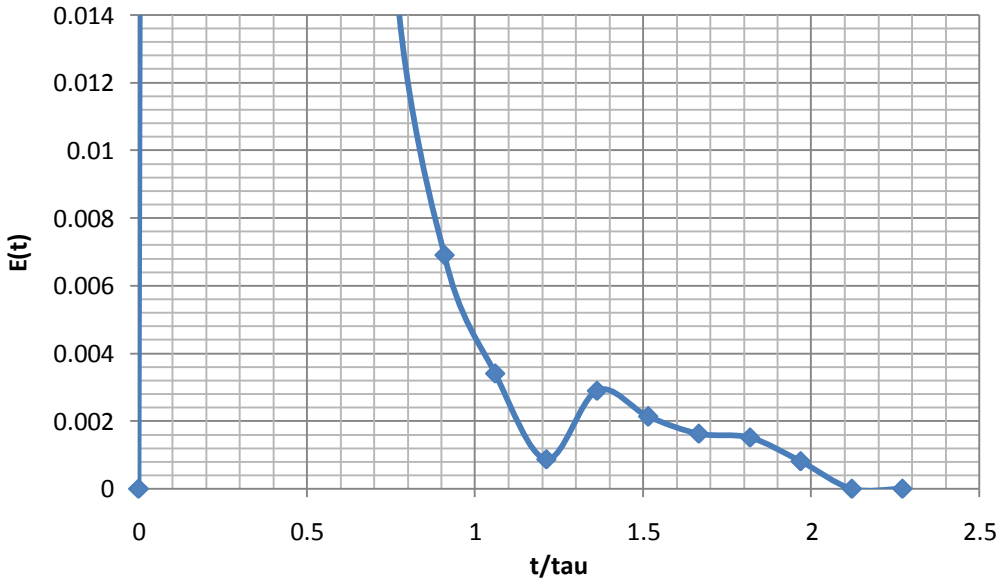


FIGURE 4.4: E-curve of pulse response for 1st Trial at flow rate 1200 ml/min

Second Experimental Trial at flow rate 1200 ml/min.

TABLE 4.9: Second trial processed data for E-curve at flow rate 1200 ml/min

Time (min)	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	377.3	0	0	0	0.1514
1	1900.9	1523.6	3047.2	0.5962276	0.3029
2	2041.2	1663.9	4991.7	0.9766964	0.4543
3	2088.4	1711.1	6844.4	1.3392033	0.6057
4	557.7	180.4	902	0.1764890	0.7571
5	394.2	16.9	101.4	0.0198403	0.9086
6	384.5	7.2	50.4	0.0098615	1.0600
7	382.1	4.8	38.4	0.0075135	1.2114
8	377.7	0.4	3.6	0.0007044	1.3628
9	378.6	1.3	13	0.0025436	1.5143
10	377.9	0.6	6.6	0.0012914	1.6657
11	377.8	0.5	6	0.0011740	1.8171
12	377.4	0.1	1.3	0.0002544	1.9685
13	377.3	0	0	0	2.1200
14	377.3	0	0	0	2.2714
		Total Concentration	Total Ct		
		5110.8	16006		

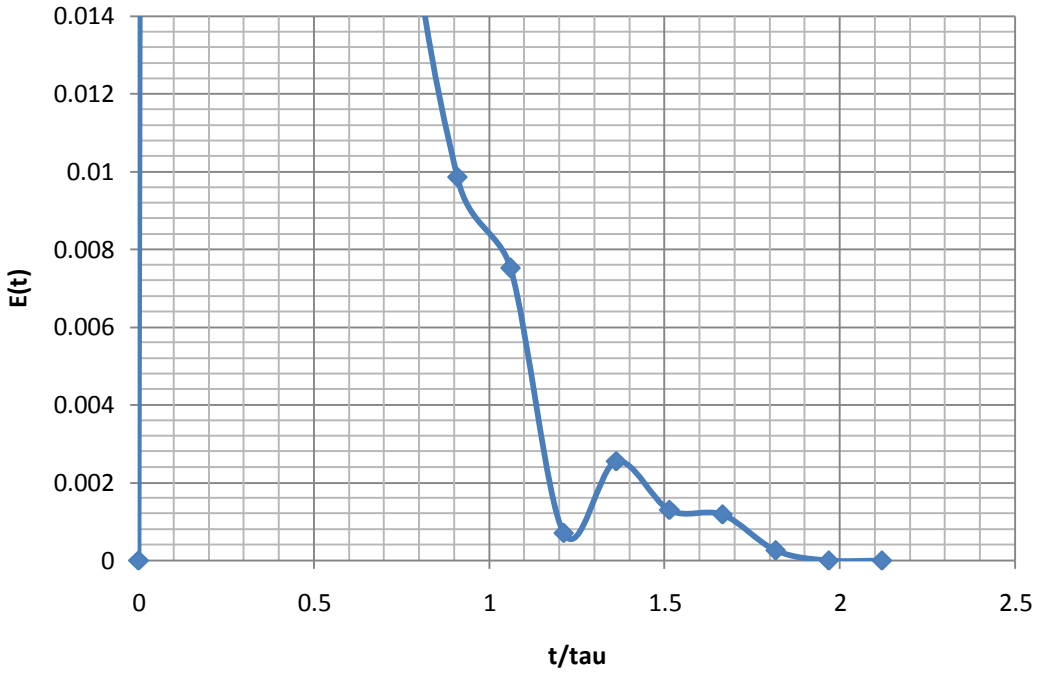


FIGURE 4.5: E-curve of pulse response for 2nd Trial at flow rate 1200 ml/min

Third Experimental Trial at flow rate 1200 ml/min.

TABLE 4.10: Third trial processed data for E-curve at flow rate 1200 ml/min

Time (min)	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	376.5	0	0	0	0
1	1919.2	1542.7	3085.4	0.6019236	0.1514
2	2046.5	1670	5010	0.9773893	0.3029
3	2065.8	1689.3	6757.2	1.3182466	0.4543
4	575.1	198.6	993	0.1937221	0.6057
5	391.5	15	90	0.0175579	0.7571
6	381.3	4.8	33.6	0.0065549	0.9086
7	377.8	1.3	10.4	0.0020289	1.0600
8	377.1	0.6	5.4	0.0010535	1.2114
9	377.3	0.8	8	0.0015607	1.3628
10	377.8	1.3	14.3	0.0027898	1.5143
11	377.9	1.4	16.8	0.0032775	1.6657
12	376.6	0.1	1.3	0.0002536	1.8171
13	376.5	0	0	0	1.9685
14	376.5	0	0	0	2.1200
		Total Concentration	Total Ct		
		5125.9	16025.4		

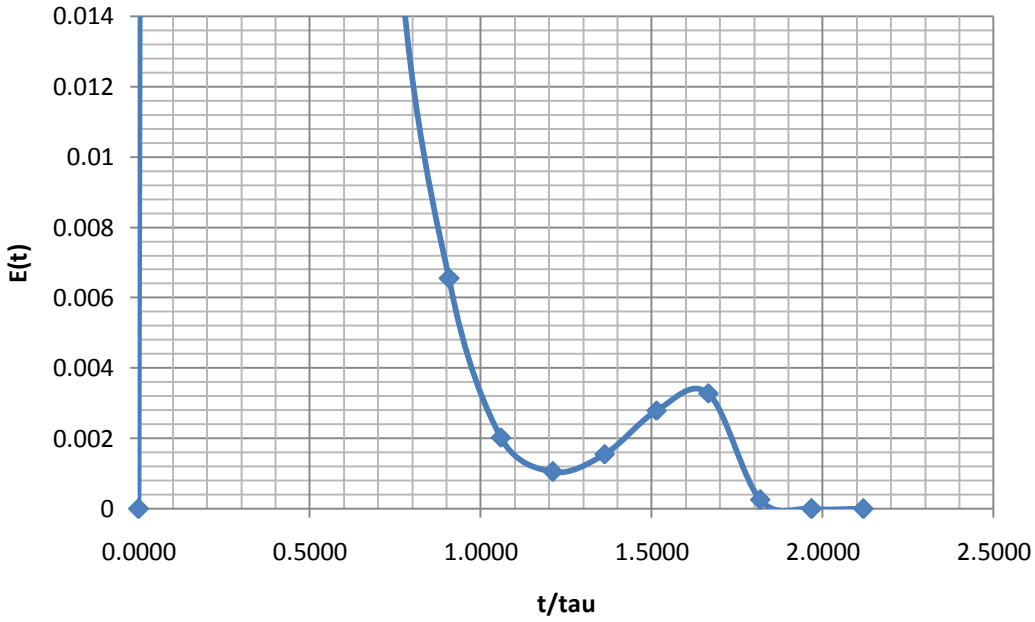


FIGURE 4.6: E-curve of pulse response for 2nd Trial at flow rate 1200 ml/min

4.3.3 Liquid Flow Rate 1900 ml/min

First Experimental Trial at flow rate 1900 ml/min.

TABLE 4.11: First trial processed data for E-curve at flow rate 1900ml/min

Time (min)	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	405.3	0	0	0	0
1	594.1	188.8	188.8	0.062475	0.2398
2	1566.2	1160.9	2321.8	0.768299	0.4795
3	1620.6	1215.3	3645.9	1.206453	0.7193
4	818.5	413.2	1652.8	0.546923	0.9590
5	434	28.7	143.5	0.047485	1.1988
6	409.3	4	24	0.007942	1.4385
7	408	2.7	18.9	0.006254	1.6783
8	408.5	3.2	25.6	0.008471	1.9181
9	408	2.7	24.3	0.008041	2.1578
10	406.8	1.5	15	0.004964	2.3976
11	406.3	1	11	0.003640	2.6373
12	405.3	0	0	0	2.8771
		Total Concentration	Ct		
		3022	8071.6		

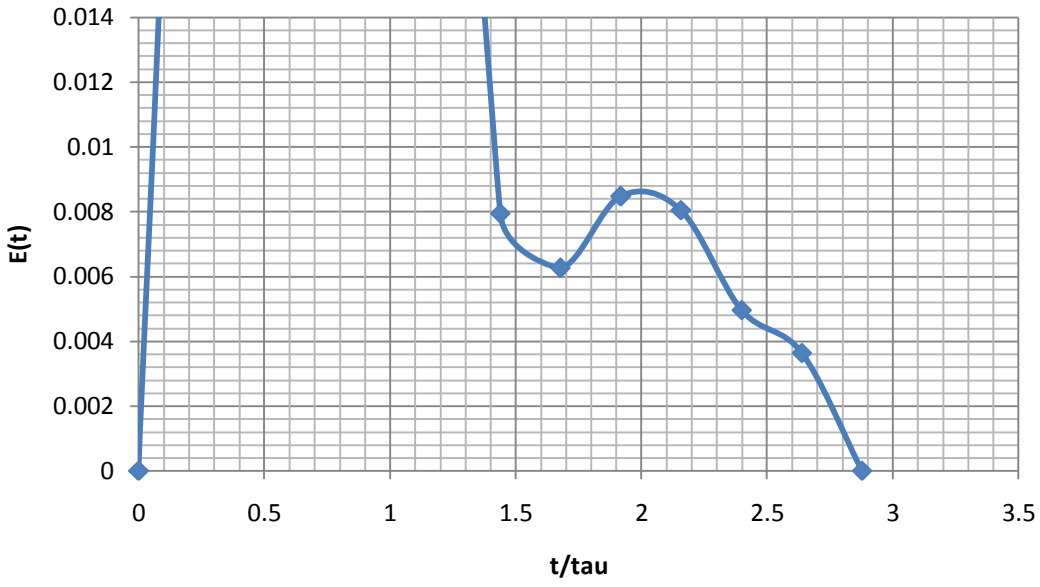


FIGURE 4.7: E-curve of pulse response for 1st Trial at flow rate 1900 ml/min

Second Experimental Trial at flow rate 1900 ml/min.

TABLE 4.12: Second trial processed data for E-curve at flow rate 1900 ml/min

Time (min)	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	399.3	0	0	0	0
1	1210.7	811.4	811.4	0.226124	0.2398
2	1562.4	1163.1	2326.2	0.648274	0.4795
3	1572.2	1172.9	3518.7	0.980604	0.7193
4	797.4	398.1	1592.4	0.443776	0.9590
5	431.3	32	160	0.044589	1.1988
6	403	3.7	22.2	0.006187	1.4385
7	401.8	2.5	17.5	0.004877	1.6783
8	401	1.7	13.6	0.003790	1.9181
9	400.1	0.8	7.2	0.002007	2.1578
10	400.8	1.5	15	0.004180	2.3976
11	399.9	0.6	6.6	0.001839	2.6373
12	399.3	0	0	0	2.8771
		Total Concentration	Total Ct		
		3588.3	8490.8		

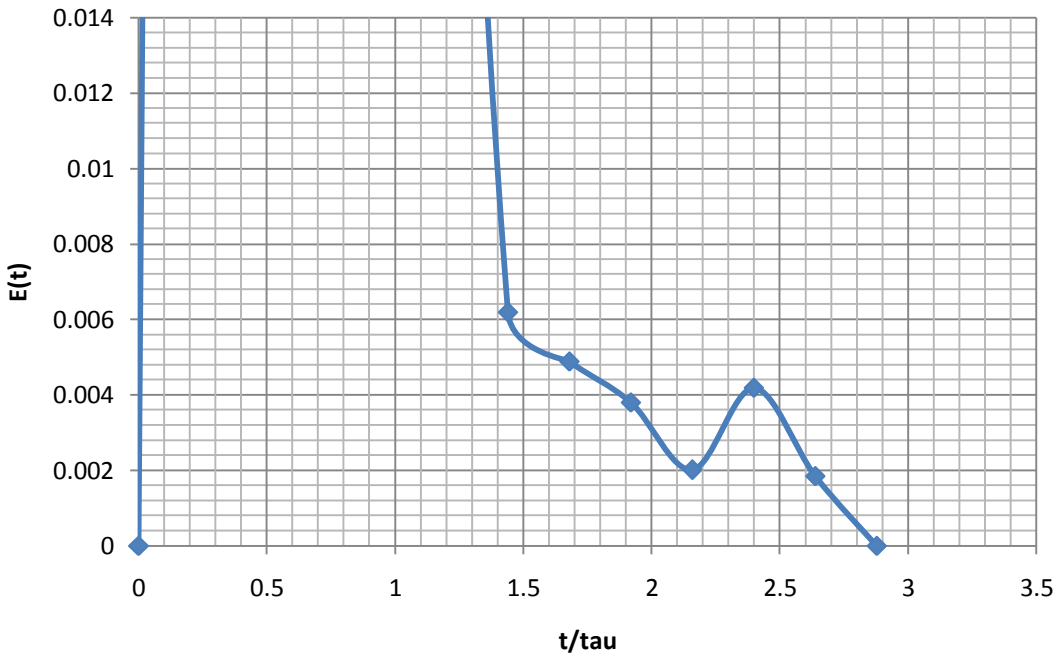


FIGURE 4.8: E-curve of pulse response for 2nd Trial at flow rate 1900 ml/min

Third Experimental Trial at flow rate 1900 ml/min.

TABLE 4.13: Third trial processed data for E-curve at flow rate 1900 ml/min

Time (min)	Concentration (µS)	Concentration Initial (µS)	Ct (µS.min)	E	t/tau
0	376.5	0	0	0	0
1	1251.7	875.2	875.2	0.236944	0.2398
2	1526.9	1150.4	2300.8	0.622898	0.4795
3	1561.3	1184.8	3554.4	0.962287	0.7193
4	813.3	436.8	1747.2	0.473022	0.9590
5	412.5	36	180	0.048732	1.1988
6	381.1	4.6	27.6	0.007472	1.4385
7	377.9	1.4	9.8	0.002653	1.6783
8	377.9	1.4	11.2	0.003032	1.9181
9	378	1.5	13.5	0.003655	2.1578
10	377.4	0.9	9	0.002437	2.3976
11	377.2	0.7	7.7	0.002085	2.6373
12	376.5	0	0	0	2.8771
		Total Concentration	Total Ct		
		3693.7	8736.4		

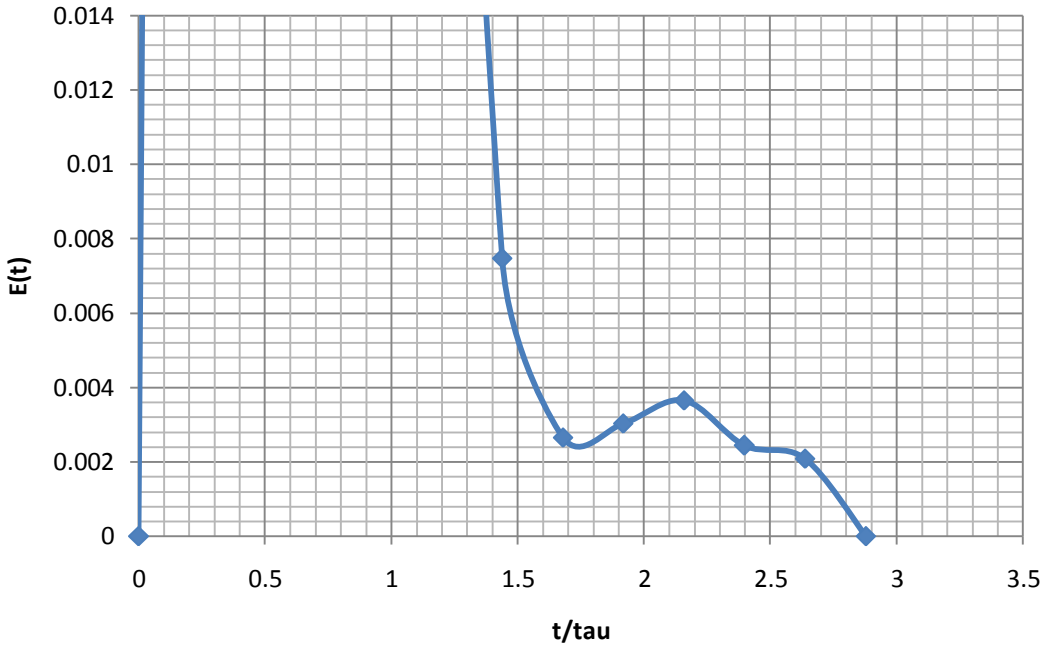


FIGURE 4.9: E-curve of pulse response for 2nd Trial at flow rate 1900 ml/min

4.3.4 Overall E-curve for Different Flow Rate.

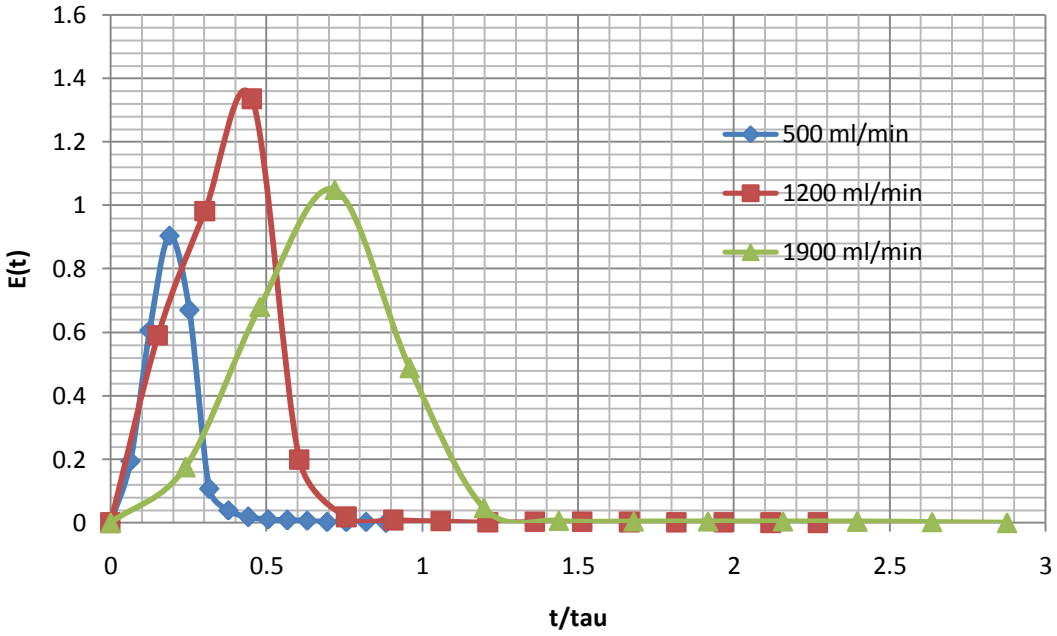


FIGURE 4.10: Typical Pulse Responds.

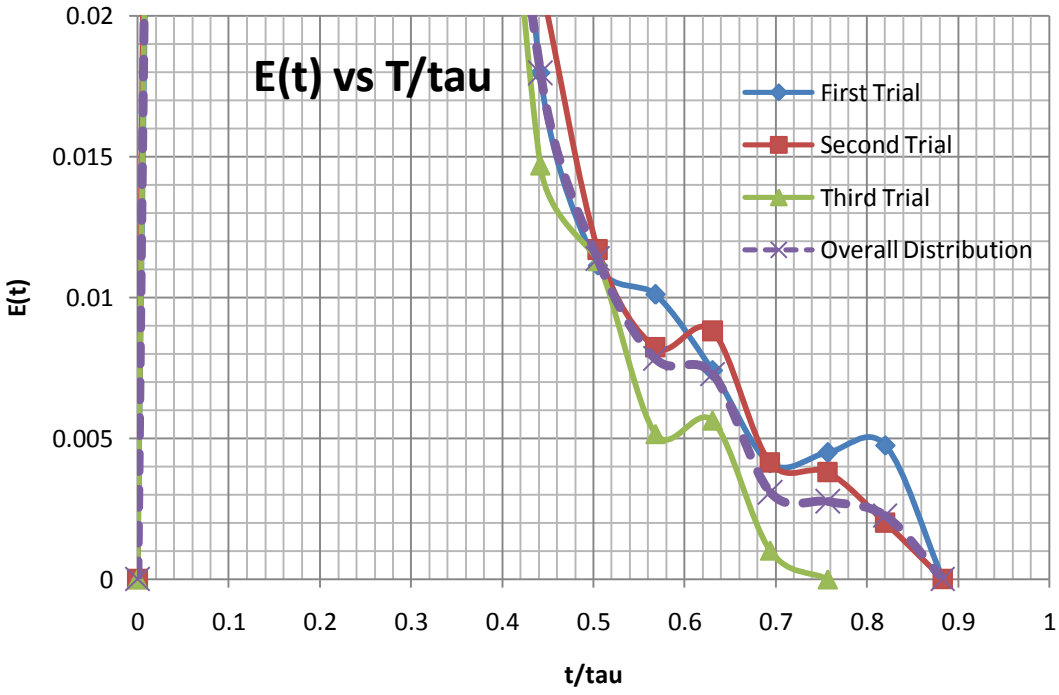


FIGURE 4.11: Overall E-curve distribution at flow rate of 0.5 l/min.

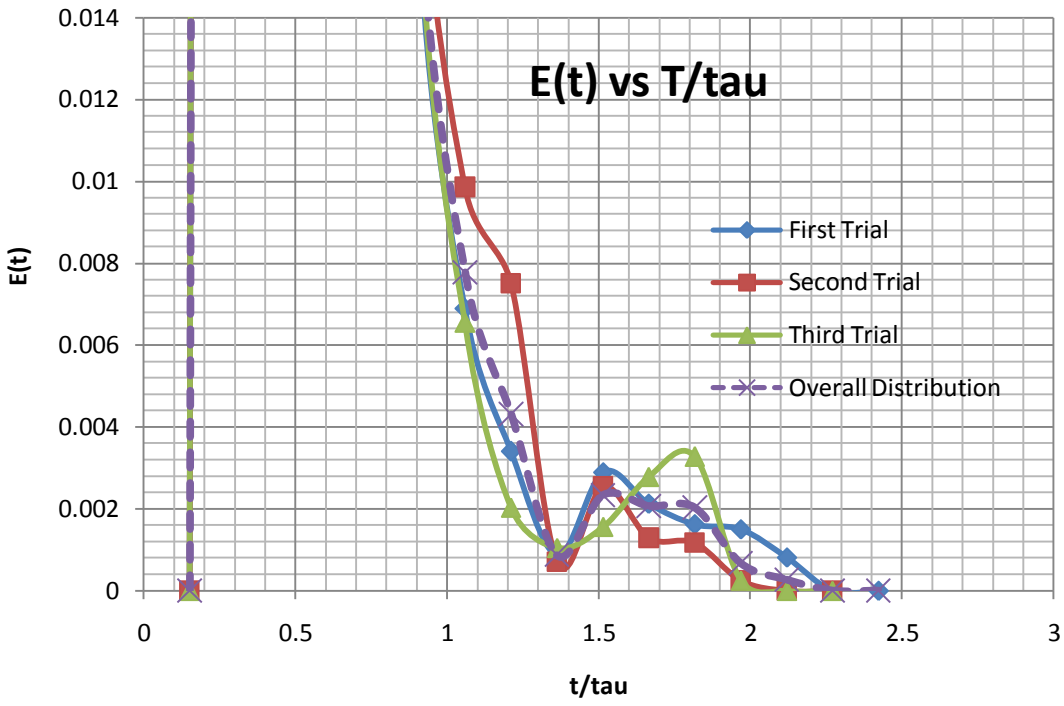


FIGURE 4.12: Overall E-curve Distribution at flow rate of 1.2 l/min

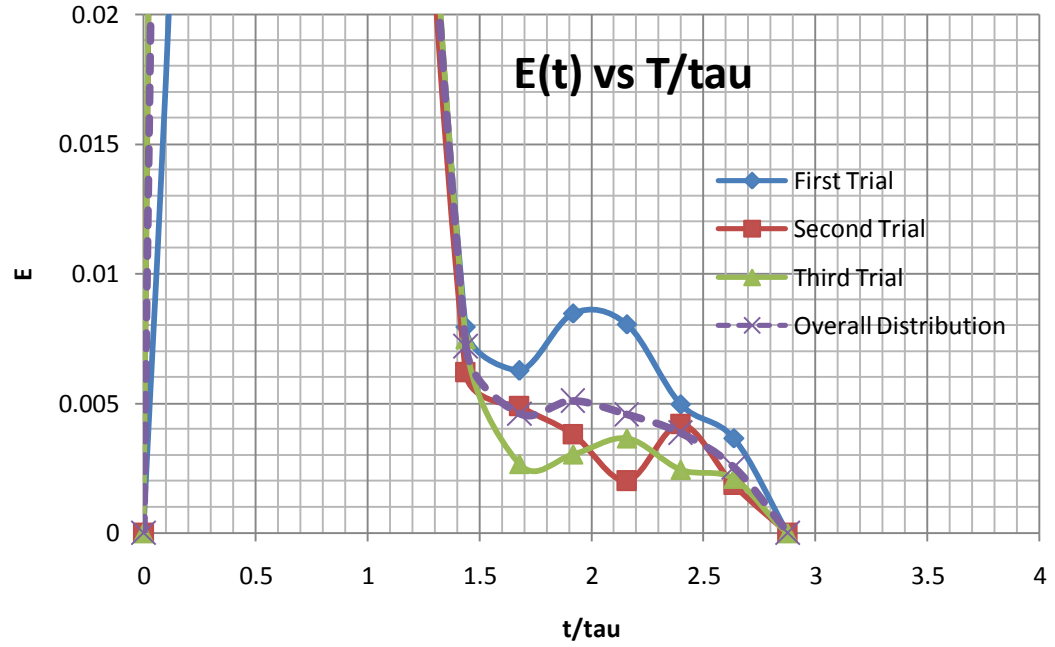


FIGURE 4.13: Overall E-curve distribution at flow rate of 1.9 l/min

All the graphs of different flow rates were combined to find the mean E-curve distribution. Based on the result almost same pulse response signals were obtained for all three different liquid flow rates. In which, when a tracer was introduced there is increased in the signal response. When tracer feed was stop after 3 minutes the signal reduces to its original state (FIGURE 4.10). It gives typical response of pulse input. In which single peaks are produced in response to a pulse change. However if the resolution of the graphs are to be increased from E(t) = 1.2 to E(t) = 0.014 as shown in (FIGURE 4.14). It shows that second peaks are produced. Figure 4.11, 4.12 and 4.13 shows the mean distribution of E-curve at flow rate of 500 ml/min, 1200 ml/min and 1900 ml/min.

This second peaks occurs as the liquid that adhere to the wall of packing starts to come out of the vessel. The occurrence of the second peaks is due to after the tracer was introduced in the packed tower; parts of the tracer got adsorbed on the walls packing elements. These adsorbed tracers, later got desorbed through diffusion and came out at the outlet of packed tower. Thus second peak is produced. For this reason all the graphs in section 4.3 is plotted at the resolution of E(t)= 0.014 to show the second peak which are produced.

The distribution of second peak and the time (t/tau) where the second peak produced reflect the wetting efficiency. At increase in liquid flow rate, the time (t/tau) for the second peak to be produced in tracer respond increases. This increase in time (t/tau) is due to the amount of wetting of packing that was affected by liquid flow rate. Julcour-Lebigue et al 2007 studied the affect of wetting efficiency on dynamic response. This work is used as the bench mark to estimate wetting efficiency. Wetting efficiency will be discussed in much detailed in section 4.4 on the comparison of result based on literature data.

4.4 Comparison of Results with literature data.

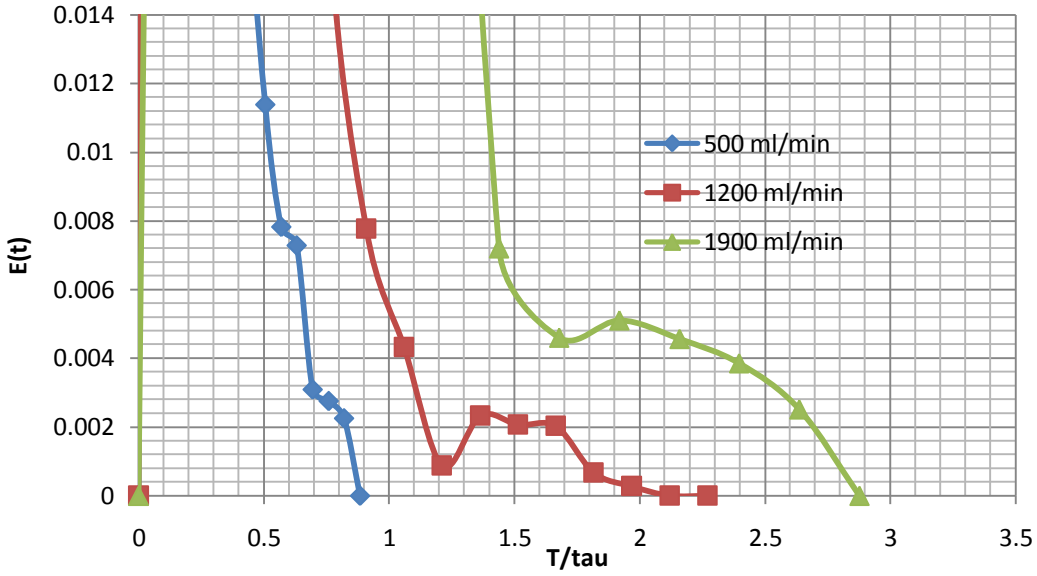


FIGURE 4.14: Overall E-curve Distribution of pulse responds.

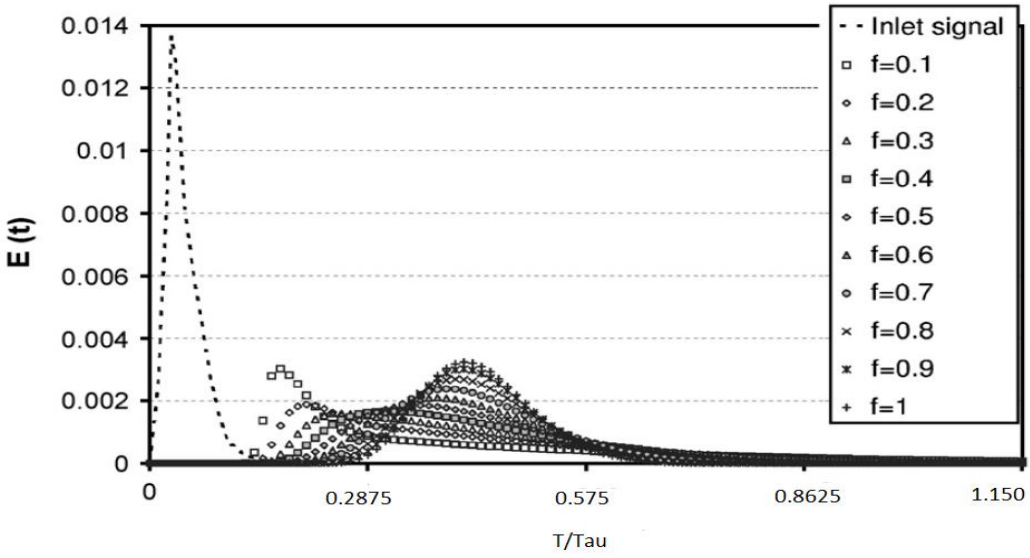


FIGURE 4.15: Effect of wetting efficiency on the dynamic respond. [10]

Julcour-Lebigue et al 2007 studied pulse response at different wetting efficiency (FIGURE 4.15). If experimental data and the work of Julcour-Lebigue to be compared figure 4.14 and 4.15 respectively, at liquid flow rate = 500 ml/min (FIGURE 4.14) it resembles wetting efficiency, f = 0.1 in figure 4.15. However second peak at 500 ml/min (FIGURE 4.14) are not clearly shown. This is because at low wetting efficiency early and rapid second peak is produced and it is believed that equipment used is not sensitive enough to produce a good reading.

As the flow rate increase to 1200 ml/min and 1900 ml/min a wider respond curve are produced and the peaks produced are much later. As compared to figure 4.14 it shows that higher flow rate has a higher wetting efficiency which is expected of this experiment.

Based on the results obtained by Julcour-Lebigue et al 2007 in figure 4.15, graph of t/tau at peak as the function of wetting efficiency was plot (FIGURE 4.16). Through this better comparison of literature data and experimental results can be obtained. Unfortunately the range of t/tau for the experimental results and literature data does not fall into agreement with each other as maximum t/tau for the experimental results is high as 1.9 and literature data falls at only 0.45. This disagreement needs to be further investigated in the future.

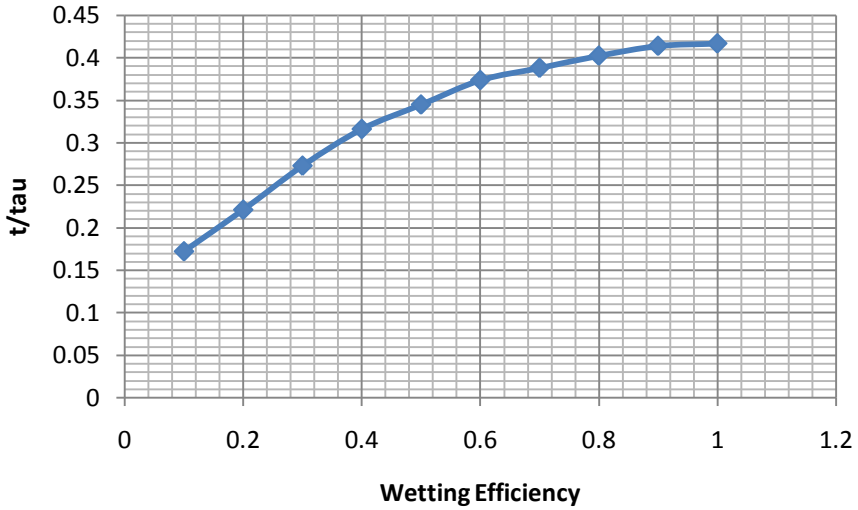


FIGURE 4.16: Effect of wetting efficiency on t/tau at peak.

At high liquid flow rate, there will be more spreading of liquid rivulet on the packing element surfaces. Thus slower liquid velocity on the surfaces of the packing element occurs. This slower liquid velocity causes desorption rate of tracer from the wall of packing element into liquid bulk flow to be lower. Therefore more time is required for adsorb tracer on the wall of the packing element to come out of the packed tower to produce the second peak. That is why at higher liquid flow rate the second peak come out much later compared to lower flow rate.

Whereas lower liquid flow rate, liquid spreading are much lower and have higher liquid velocity on the surface of packing element. Higher liquid velocity means an increase in the rate of diffusion of tracer on the surface of packing element and liquid bulk flow. Therefore earlier peak is produced at lower liquid flow rate.

4.5 Model Development to Estimate Wetting Efficiency.

In this investigation simple model is to be developed for the estimation of wetting efficiency. This model was developed based on mass transfer of tracer on the wall of packing particles into liquid bulk flow.

Physical Description:

Liquid flowing down the packing in the packed tower wet the surface of width,,Rw and length, H as shown the figure 4.17. A pulse of tracer introduced in the input is expected to move along the liquid flow and exit at the bottom of the packed tower. However, part of the tracer can adsorb on the surface and accumulate in the stagnant liquid layer near the surface without exiting with liquid flow at the bottom. Such adsorbed tracer molecules may get desorbed into liquid flow subsequently; this can result in a second weaker pulse of tracer in the exit stream. Thus the second tracer output is due to mass transfer of tracer material adsorbed on the surface.

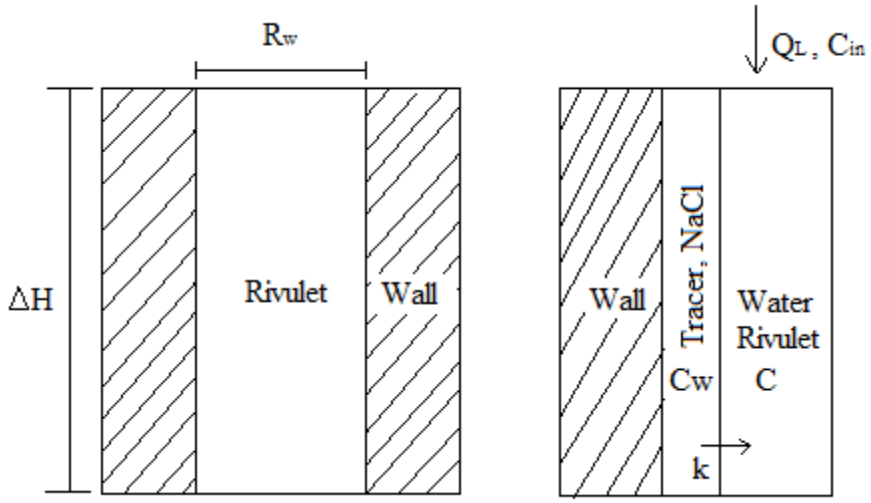


FIGURE 4.17: Front and side view of packing wall.

Mass Balance Equations:

Mass Transfer from adsorbed film to bulk liquid

Depletion of adsorbed tracer at the wall = Mass Transfer from the adsorbed layer to bulk of liquid flow

$$-\frac{d}{dt}\left(R_w \Delta H \Delta Z C_w\right) = k R_w \Delta H\left(Cw - C\right) \quad (4.1)$$

Considering the liquid film as a CSTR, convective flow in the bulk fluid

Mass Transfer from the adsorbed layer to bulk of liquid flow = Convective flow in the Film

$$k R_w H\left(Cw - C1\right) = Ql\left(Cin - C1\right) \quad (4.2)$$

$$C_{in} = 0$$

$$Cw = C1\left(\frac{kRwH - Ql}{kRwH}\right)$$

From equation 4.1 and 4.2,

Depletion of adsorbed tracer at the wall = Mass Transfer from the adsorbed layer to bulk of liquid flow

$$-\frac{d}{dt}\left(R_w H - \frac{Ql}{k}\right)\Delta Z C1 = -Ql\, C1$$

$$C1 = 0 \text{ at } t = 0$$

$$-\left(R_w H - \frac{Ql}{k}\right)\Delta Z\, \Delta C1 = -Ql\, C1 \Delta t$$

$$\left(R_w H \Delta Z - \frac{Ql}{k}\Delta Z\right) = \frac{Ql C1 \Delta t}{\Delta C1}$$

Liquid flow for each stream in a packed bed can be obtained from net liquid flow rate Q as

$$Ql = \frac{QD_p^2}{\eta Ab(1-\varepsilon)}$$

$$\eta = \frac{RwH}{\left(\left(AbH(1-\varepsilon)\right)\Big/\left(D_p^3\right)\right)D_p^2} = \frac{RwDp}{Ab(1-\varepsilon)}$$

$$Ql = \frac{QDp}{Rw}$$

$$\frac{Rw}{Dp}\left(R_w H\Delta Z - \frac{Ql}{k}\Delta Z\right) = \frac{QC1\,\Delta t}{\Delta C1}$$

$$\frac{Rw}{Dp}\left(R_w H\Delta Z - \frac{Ql}{k}\Delta Z\right) = \frac{QC1\,\Delta t}{\Delta C1} \propto f(\text{wetting efficiency})$$

However, liquid flow in the rivulets can approximate to plug flow rather than to a CSTR. These equations need to be further refined for better accuracy

CHAPTER 5

CONCLUSION & RECOMMANDATION

Stimulus response of pulse input of a packed tower was performed by using RTD studies of packed Bed equipment for the measurement of wetting efficiency at three different flow rates. This investigation demonstrates that higher wetting efficiency would give wider E-curve response and second peak to produce much later. Besides that, it is also conclude that liquid flow rate is also proportional to wetting efficiency. The experimental result obtained, almost resembles the work of Julcour-Lebigue et al 2007.

Through this investigation simple model for the measurement of wetting efficiency is developed:

$$\frac{Rw}{Dp}\left(R_w H\Delta Z - \frac{Ql}{k}\Delta Z\right) = \frac{\mathrm{QC1}\Delta \mathrm{t}}{\Delta C1} \propto f\left(wetting\ efficiency\right)$$

However this correlation reflects on the behavior of liquid film as a CSTR rather than plug flow.

For recommendation, further refinement of this equation is needed for better accuracy, to reflect the liquid rivulet to behave as a plug flow rather than CSTR. For more accurate experimental result, more sensitive equipment is needed as the available equipment is did not sufficient sensitivity for this investigation.

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APPENDIX

Standard Operating Procedure (SOP) for *RTD Studies of Packed Bed Equipment.*

1. Make sure ON the equipment 1st before ON the PC.
2. Check the:
   - All Cable
   - All drain valves is close
   - Make sure the de-ionized water is FULL
3. For PC Setting:
   - Click experiment
   - Experiment A: The effect of step Change Input. (Co-current & Counter current)
   - Experiment B: The effect of Pulse Input. (Co-current & Counter current)
4. Perform a quick inspection to make sure that the equipment is properly working condition.
5. Check all valves are initially closed.
6. Open valve V3 to fill feed tank 1 with de-ionized water.
7. Prepare 10L of 0.2M NaCl solution in dosing tank T2. Record the conductivity reading for this solution.
8. Flush the system with DI water until no trace of salt is detected.
9. Change the valve configuration so that the liquid will be introduced at the top.
10. Open the control valve V1 until liquid flow to desire flow rate.
11. Open the dosing for 2 minutes.
12. Record the concentration reading in PC.
13. After each experiment, drain off any liquid from reactor and make sure that the reactor and tubing's are clean and properly flush with de-ionized water.
14. Dispose all liquids immediately after each experiment. Do not leave any solution or waste in tank over a long period of time.
15. Wipe off any spillage from the unit immediately.