



EFFECT OF TRAY DESIGN ON THE PERFORMANCE OF A VIBRATING PLATE EXTRACTION COLUMN

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Nomakhosi Dorothy Sincuba

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Supervisors: Dr. S. Rathilal

: Prof. M. Carsky



DECLARATION

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ABSTRACT

There are various types of separation processes used in industries, such as, distillation, liquid-liquid extraction and gas absorption. Liquid-liquid extraction (LLE) is a separation process that involves mass transfer from a liquid mixture to an immiscible extraction solvent. This process is generally used where the mixture cannot be separated by fractional distillation due to close relative volatilities of the mixture components or for heat sensitive components.

LLE is rated as the second most important separation process after distillation and it is widely used in chemical engineering industries. Different types of columns are available for LLE. Reciprocating and vibrating plate extraction columns are mechanically agitated extraction columns. In this research a vibrating plate extraction column was utilised. It uses perforated plates with down-comers. The perforations provide passage for the dispersed phase, while down-comers provide passage for the continuous phase.

The effectiveness of a vibrating plate extractor was previously investigated but limited research was conducted on the effect of the different tray designs and agitation levels (as the product of amplitude and frequency of vibration). These variables affect the hydrodynamics and mass transfer in the column. The determination of the optimum process parameters is important in achieving the highest separation efficiency of the vibrating extraction column.

The focus of this research is to test the effect of different tray designs in order to enhance the separation process efficiency. All tests were conducted using the toluene-acetone-water system as a standard test system proposed by the European Federation for Chemical Engineering, (1985). The experiments were conducted to test the effect of mass transfer on the hydrodynamics of the system while changing the agitation levels, downcomers and hole diameters. A comparative investigation was conducted on straight segment and circular downcomers, then hole diameters analysis was performed on the effective downcomer

Comparison of the effect of the tray design types on the amount of acetone extracted was utilised to select the effective tray. Tray with the circular downcomer and 3.0 mm hole diameter had high

dispersed phase holdup values and with the highest percentage amount of acetone extracted (95.05%) was obtained compared to other tray designs investigated.

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NOMECLATURE

Symbol	Definition	Units
a	amplitude(half stroke)	m
A	Stroke	m
A_c	Cross-sectional area of column	m ²
C_N	Orifice coefficient = 0.67 for circular holes	
C_o	Orifice discharge coefficient through perforations	
c_i	Concentration of the i^{th} phase (mass or volume basis)	
c_i^*	Concentration at equilibrium (mass or volume basis)	
d	Dispersed phase drop diameter	m
d_c	Characteristic dimension	m
d_e^0	Value of d_e at the linear velocity of 3 cm/s in the perforation	m
d_e	Equivalent drop diameter	m
d_0/hd	Perforation diameter	m
d_{32}	Sauter mean drop diameter	m
D	Diffusivity	m ² /s
D_c	Column diameter	m
dsd	Drop size distribution	
E_f	Tray efficiency	
E	Extraction factor mU_x/U_y	
E_o	Overall tray efficiency	
E_c	Axial dispersion coefficient of continuous phase	m ² /s
E_B	Extraction factor of component B	
E_M	Murphree efficiency in terms of the i^{th} phase	
E_n	Entrainment of dispersed phase, ppm (by volume)	

E_v	Volumetric efficiency	l/hr
	Solute-free extract flow	kg/h
f	Frequency	Hz
F_i/Q_i	Volumetric flow rate of the i^{th} phase	m^3/s
	Solute-free feed flow	kg/h
g	Acceleration due to gravity force	m/s^2
g_c	Gravitational conversion factor	$\text{kg}.\text{m}/\text{s}^2.\text{N}$
h	Centre to centre plate spacing	mm
h_t	Liquid head of static holdup	m
H	Height of the active part of the extractor	m
HETS	Height equivalent to a theoretical stage	m
H_{ox}	True height of transfer unit	m
H_{oxp}	Plug flow height of transfer unit	m
J	Interfacial flux	$\text{kmol}/\text{m}^2.\text{s}$
k_E	Extract mass transfer coefficient	m/s
k_R	Raffinate mass transfer coefficient	m/s
K_i	Dimensionless constant	
K_A	Distribution coefficient of component A	
K_C	Distribution coefficient of component C	
K_{equ}	Equilibrium ratio	
$K_E, K_R,$	Overall mass transfer coefficient	m/s
k_{ox}, k_{oy}		
k_m	Marangoni mass transfer coefficient	m/s
L	Ratio U_d/U_c	
N	Number of plates	
N_{ox}	True overall number of transfer units based on the x phase	
N_{oxm}	Measured overall number of transfer units based on the x phase	
N_{oxp}	Apparent or piston number of transfer units assuming plug flow	
N_{ox}^{-1}	Number of transfer units per stage	
N_R	Solute flux	m/s
P_i	Turbulent Peclet number of the i^{th} phase = $U_i d_c / e_i$	

Pe^*	Average droplet Peclet number = $u_t d_{32}/D_{AB}$	
Δp	Pressure difference across a plate	kPa
P_m	Power input	W
q	Backmixing between adjacent stages	
q_c	Effective coefficient of backmixing in the continuous phase	
R	As defined in the equation	
	Solute-free raffinate flow	kg/h
U_c	Superficial velocity of continuous phase	m/s
U_d	Superficial velocity of dispersed phase	m/s
U_T	Total throughput = $U_c + U_d$,	m/s
U_o	Hole or perforation velocity	m/s
u_k	Characteristic slip velocity	m/s
u_N	Velocity at the nozzle or orifice	m/s
u_p	Velocity of plate	m/s
u_s	Slip velocity	m/s
v	Phase volume in stage	m ³
W_1, W_2	Adjustable velocity parameters defined by equation	
X, Y	As defined in equation	
x	Mol fraction in raffinate phase	
x^*	Raffinate mol fraction in equilibrium with extract phase	
y	Mol fraction in extract phase	
y^*	Extract mol fraction in equilibrium with raffinate phase	

LIST OF SYMBOLS

Symbol	Definition	Units
γ	Interfacial tension	N/m
γ_{pef}	Interfacial tension of pure substance	N/m
γ_{eq}	Interfacial tension at equilibrium	N/m
$\Delta\gamma$	Difference in surface tension	N/m
γ_{Be}	Activity coefficient of component B in extract phase	
γ_{Br}	Activity coefficient of component B in raffinate phase	
γ_{AB}	Activity coefficient of component A in B	
μ_{d}	Dispersed phase velocity	Pa.s
μ_{c}	Continuous phase velocity	Pa.s
μ_{w}	Velocity of water = 10^{-3}	Pa.s
ϕ	Dispersed phase holdup	
ϕ_{I}	Defined by equation	
β	Separation factor	
ρ_{d}	Dispersed phase density	kg/m ³
ρ_{c}	Continuous phase density	kg/m ³
$\Delta\rho$	Density difference, ($\rho_{\text{c}} - \rho_{\text{d}}$)	kg/m ³

CHAPTER 1: INTRODUCTION

Liquid-liquid extraction (LLE) is a separation process that involves the mass transfer from a liquid mixture to an immiscible liquid solvent. It is also known as solvent extraction. It is ranked as the second most vital separation process that is extensively applied by industries (Mohanty and Volgelgohl, 1997).

Solvent extraction is normally used in cases where the distillation process is impractical because of the close relative volatility of two components and when there is a possibility of azeotrope's formation. It is used by many industries for various applications such as the production of active agents by the pharmaceutical industry, the production of monomers and aromates by the petroleum industry, and for cleaning of waste water to separate dissolved compounds (Usman *et al.*, 2008 and Tsouris *et al.*, 1994).

LLE may utilise gravitational or centrifugal forces for separation purposes. Columns used can be static or agitated in-order to improve interfacial area and mass transfer (Seader and Henley, 2006; Rama Rao *et al.*, 1991). The vibrating plate extractor is one type of mechanically agitated columns and it is also a modification of the reciprocating plate column.

The report entails the research done using the vibrating plate extractor. The literature review divulges that most research work has been previously done on mechanically agitated columns. However, it is limited when it comes to reciprocating plate column and vibrating plate extractors, hence much research is still being done for these columns, and hence limited papers were written in the 2000's.

The main advantage of the vibrating plate extractor compared to the reciprocating plate column is that it operates at relatively low amplitudes and frequencies which indicate that it has a lower mechanical stress and energy consumption (Lo and Prochazka, 1983).

The vibrating plate extractor has many variables that can be adjusted to achieve the desired results such as the tray downcomers, tray hole diameters and the amplitude and frequency of vibrations. These variables affect the mass transfer and hydrodynamics in the vibrating plate extraction column. Therefore the determination of the optimum process variables is significant in attaining the highest efficiency for the column.

The main aim of this research was to investigate the effect of different plate designs in order to enhance the effectiveness of mass transfer in the vibrating plate extractors. The evaluation is based on the investigation of the effect of tray hole diameter, downcomer types and agitation level on mass transfer efficiency. The mass transfer efficiency was determined using the percentage of acetone extracted.

This research project focuses on the literature review concerning the research topic, the performed experimental work and the results acquired from experimental work as well as the discussion of the results.

Dissertation outline

Chapter 1

The introduction outlines the purpose of this study. It provides the background information of liquid-liquid extraction using the vibrating plate extractor.

Chapter 2

This chapter reviews literature which deals with the separation process methods. The main focus is liquid-liquid extraction using vibrating plate extractor and using different tray designs in-order to improve the performance of vibrating plate extraction column.

Chapter 3

Chapter 3 presents the experimental and analysis procedures, the analytical methods used for the evaluation of drop size distribution, holdup, Sauter mean diameters and mass transfer. It also presents the description of apparatus and the chemicals used.

Chapter 4

This chapter contains the results and discussion obtained from:

The study of the effect of hydrodynamics conditions on the mass transfer.

The study of the effect of tray downcomers and different hole diameters on the mass transfer.

Chapter 5 and Chapter 6

These chapters present the conclusions and recommendations related to the effect of tray design in the liquid-liquid extraction using vibrating plate extractor and optimum parameters that need to be used in-order to achieve higher separation efficiency.

CHAPTER 2: LITERATURE REVIEW

2.1. Introduction

LLE is a separation process that involves mass transfer from a liquid mixture to an immiscible extraction solvent. The ability of the LLE process to separate chemicals according to their type has made it popular compared to other separation processes, such as distillation, absorption and drying.

LLE is used by various industries like chemical, nuclear, petroleum, and biotechnology and waste management related industries (Pietzsch and Blass, 1987). It is the second most important and mainly applied separation process after distillation.

For the extraction method to be categorised as simple, it involves: 1) solute, which is substance that needs to be extracted, 2) carrier, and 3) solvent and the substance that is used to extract solute from the carrier. Figure 2.1 illustrates the counter current extraction process and table 2.1 provides the terminology.

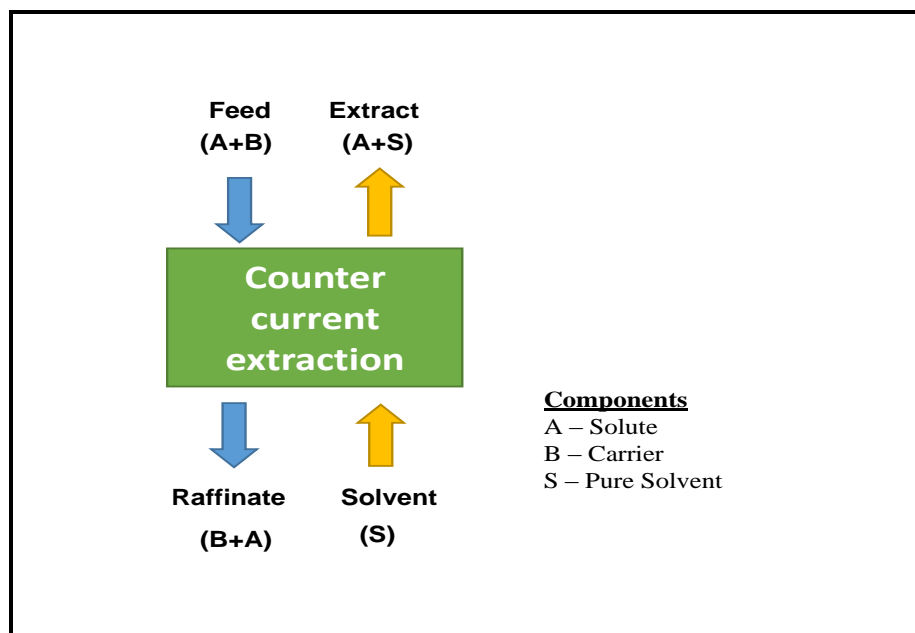


Figure 2.1: Counter current extraction (Seader and Henley, 2006)

Table 2.1: Terminology that may be used to describe the extraction system (Seader and Henley, 2006)

Terminology	Definition
Feed	The substance that contains a carrier and the solute that needs to be extracted.
Carrier	The non-solute portion of the mixture (portion that remains after extraction takes places).
Solute	The substance that needs to be extracted.
Solvent	The fluid that is used to influence the extraction.
Extract phase/stream	The output stream that contain high amount of solutes.
Light phase/stream	The input stream that has lower density and flows up the column and then accumulates at the top.
Heavy phase/stream	The input stream that has higher density which flows down the column and then accumulates at the bottom.
Dispersed phase/stream	The input stream that is distributed in a drop form through the distributor pores.
Continuous phase/stream	The input stream that flows in bulk and does not form drops.
Raffinate phase/stream	The output stream that contains small or zero amounts of solute.

For a highly efficient contact between the solvent and the feed, a large interfacial area is required. An increase in vibrating levels can increase the interfacial area and also enhance the mass transfer (Seader and Henley, 2006). The phase with the largest volume is commonly selected as a continuous phase. However, each of the phases can be dispersed by setting the interface level in either the top or the bottom section (Keller and Humphrey, 1997).

Extraction processes may be arranged into three main multistage arrangements, such as, co-current, crosscurrent and counter-current. However, counter-current is the most common one (Robbins, 1996). In a counter-current flow, the feed is introduced at the bottom and the solvent is introduced from the top of the column or vice versa. Figure 2.2 illustrates the counter-current flow system with an interface located at the top of the column.

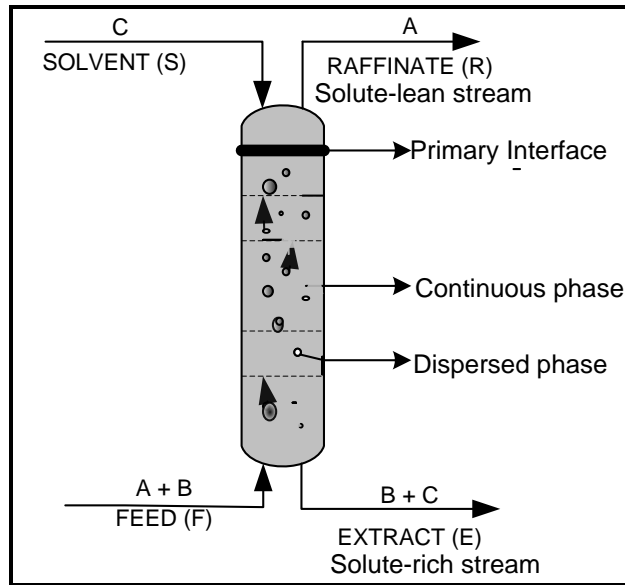


Figure 2.2: Typical counter-current flow system (adapted from Seader and Henley, 2006)

Normally the heavy liquid (higher density) is the continuous phase and introduced from the top of the column and the light liquid (lower density) is the dispersed phase and introduced at the bottom of the column. In most cases the liquid that preferably wets the column internals is the continuous phase so that the dispersion drops can move through the continuous phase freely (Laddha and Degaleesan, 1983).

The principal interface depends on which phase is dispersed. If the dispersed phase is the light phase, the principal interface will be located at the top of the column. If the dispersed phase is the heavy phase it will be located at the bottom of the column. Normally the phase that is fed at the lower rate is the dispersed phase (Keller and Humphrey, 1997).

2.1.1. Advantages of liquid-liquid extraction

In comparison to distillation, liquid-liquid extraction can be used in the separation of azeotropes as well as for components that have close boiling points. It is usually operated at a lower cost, and can be operated at low to moderate temperature for recovery of heat sensitive products in the petroleum, food and pharmaceutical industries (Keller and Humphrey, 1997).

In selecting a separation process the relative cost is significant. Even though the extraction process may involve other separation units beside the extractor, the relative cost may be lower. In case of more dilute solutions, where water needs to be vaporised in the distillation for separation, extraction is more economical due to the facts that the heat of vaporisation of most organic solvents is substantially lesser than that of water (Treybal, 1980).

LLE is cheaper and can be used instead of using the chemical methods because chemical methods use reagents that result in expensive disposal for chemical by-products (Seader and Henley, 2006; Treybal, 1980). It has rapid and very selective separations that are usually highly efficient.

2.1.2. Disadvantages of liquid-liquid extraction

Normally, the extraction system consists of one distillation to recover the solvents. However, if the solvent is soluble in the carrier, multiple extractions might be required to recover the solvent from the raffinate, thereby increasing time, consumption of materials, and generation of waste (Humphrey and Keller, 1997). Other disadvantages mentioned by the authors are as follows:

- Can be time consuming, especially if attainment of equilibrium is slow;
- Can be affected by small impurities in the solvent(s);
- Cumbersome for a large number of samples or for large samples;
- Formation of emulsions can interfere with the phase-separation process;
- Counter-current process can be complicated and can require complicated equipment;
- Alteration of the chemical form can occur, going from one phase to the other, thereby altering the distribution coefficient and effectiveness of the extraction.

2.2. Thermodynamic and mass transfer principles

2.2.1. Thermodynamic principles

The measure of how well the solute is extracted by extraction solvent is referred to as the distribution ratio or selectivity. Distribution ratio of the species is the concentration of a solute in the organic phase divided by solute concentration in the aqueous phase. Distribution ratio can be a function of temperature or solute concentration in the system depending on the system (Seader and Henley, 2006).

For simple ternary systems the equilibrium distribution of solute and carrier between the phases is critical to the process. A distribution coefficient or equilibrium ratio is defined as:

$$K_C = \frac{X_{C,E}}{X_{C,R}} \quad (2.1)$$

Where K_C is a distribution coefficient for component C, $X_{C,E}$ is the mole fraction of component C in the extract phase, and $X_{C,R}$ is the mole fraction of component C in the raffinate phase

And

$$K_A = \frac{X_{A,E}}{X_{A,R}} \quad (2.2)$$

Where K_A is the distribution coefficient for component A, $X_{A,E}$ is the mole fraction of component A in the extract phase, and $X_{A,R}$ is the mole fraction of component A in the raffinate phase. The separation factor selectivity, β , is defined as the ratio of the K values:

$$\beta_{CA} = \frac{K_C}{K_A} \quad (2.3)$$

For liquid-liquid extraction with ternary mixtures, phase equilibrium is suitably represented on an equilateral or right angled triangle diagram. There are three types of ternary systems. Type 1 ternary system is simplest and most common systems. It consists of only one miscible pair (solute - solvent) and is shown in Figure 2.3. It has a plait point and a tie line. The plait point is the points where the raffinate phase and extract phase boundary curves intersect and above it there is no more separation. Tie lines join equilibrium concentration of the different phases. It converges to the plait point and the two phases become one phase. The two phase region exists below the solubility lines while the single phase region exists above, (Fair and Humphrey, 1983).

Type 2 ternary system is less common. It consists of two immiscible pairs. It only has tie lines and no plait point. A type 2 ternary system is shown in Figure 2.4.

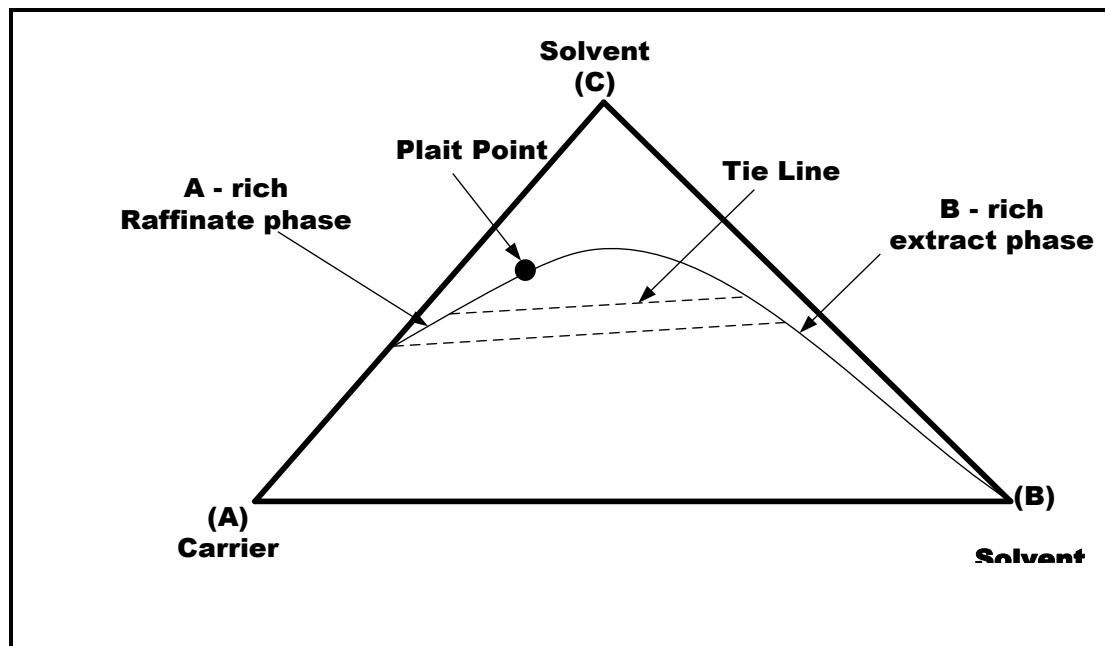


Figure 2.3: Type 1 ternary system (Fair and Humphrey, 1983)

The toluene-acetone-water system is one example of a type 1 ternary system. Toluene-acetone-water system is the standard system recommended by the European Federation of Chemical Engineering as a test system for liquid-liquid extraction (EFCE, 1985; Steiner et al., 1990).

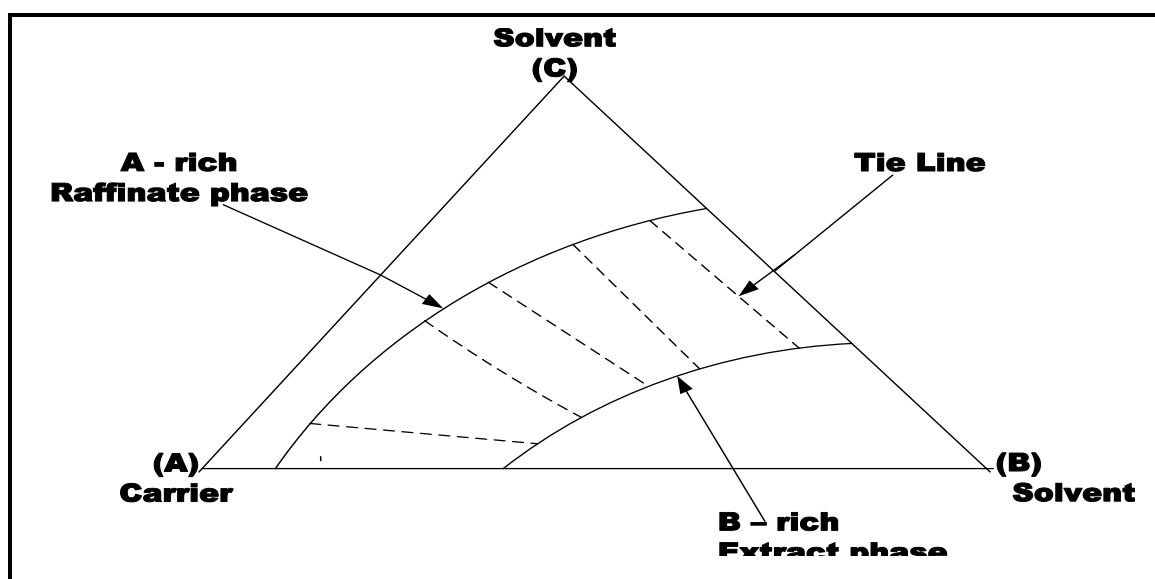


Figure 2.4: Type 2 Ternary system (Fair and Humphrey, 1983)

A type 3 ternary system consists of three immiscible pairs, (Fair and Humphrey, 1983).

2.2.2. Mass transfer principles

2.2.2.1. Two film theory

During the liquid-liquid extraction process it is imperative to consider the mass transfer resistance for both the continuous and the dispersed phase. Therefore two films are used to describe the mass transfer method in the extraction process where the solute is transferred between the raffinate and extract phases. This mass transfer method proposes that each thin film layer presents a resistance to mass transfer and that the two phases are in equilibrium, as a result there is no resistance to mass transfer from the interface itself. The concentrations in the bulk liquids are assumed to be uniform because of eddy and molecular diffusion. Figure 2.5 shows the concentration gradient in inter-phase mass transfer (Schweitzer, 1997; Humphrey and Keller, 2006; Seader and Schweitzer, 1997).

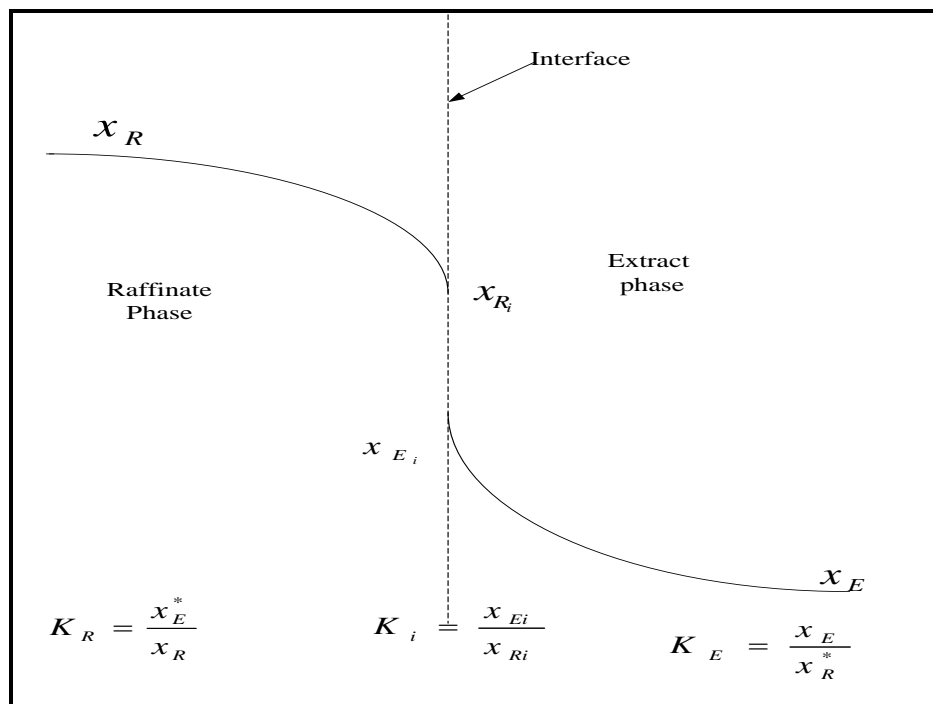


Figure 2.5: Concentrations profiles in interface mass transfer
(Keller and Humphrey, 1997)

As indicated before, the two resistance theory assumed that the resistance to mass transfer is negligible at the interface. However, Lisa *et al.* (2003), observed the existence of an interfacial resistance when they studied the extraction using toluene-acetone-water system.

2.2.2.2. Rate of mass transfer

The rate of mass transfer of the solute from the bulk solution of A to the bulk solution of B is illustrated in Equations 2.4 and 2.5 where k that is the film mass transfer coefficient for a particular phase (Schweitzer, 1997).

$$N_R = k_R(x_R - x_{Ri}) = k_E(x_{Ei} - x_E) \quad (2.4)$$

$$N_R = K_R(x_R - x_R^*) = K_E(x_E^* - x_E) \quad (2.5)$$

Where k_R and k_E are individual phase transfer coefficients, and K_E and K_R are overall transfer coefficients that incorporate the resistance of both phases. On the basis of the extract phase, Equation 2.4 and 2.5 can be combined as follows:

$$\frac{1}{K_E} = \frac{1}{k_E} + \frac{m_E}{k_E} \quad (2.6)$$

Where m_E is the slope of the equilibrium curve plotted with the extract mole fraction on the ordinate scale (Keller and Humphrey, 1997). If the extract is the dispersed phase, Equation (2.6) becomes:

$$\frac{1}{K_d} = \frac{1}{k_d} + \frac{m}{k_c} \quad (2.7)$$

Mass (or mole) ratios, X (raffinate phase) or Y (extract phase) are defined as the ratio of mass (or moles) of the solute to the mass (or moles) of the other components in a particular stream or phase. They are related to mass (mole) fractions as follows (Seader and Henley, 2006)

2.2.2.3. Factors affecting rate of mass transfer

According to Lo and Baird (1994) and Schweitzer (1997), these are the main issues that can influence the mass transfer rate in extraction:

i. Mass transfer coefficients

- The diffusivity and results in interfacial turbulence is governed by the phase composition. The diffusion rates are affected by the temperature;
- The direction of mass transfer is dependent on the dispersed phase;
- The film thickness and the interfacial turbulence can be affected by the type and degree of energy input.

ii. Concentration driving force

- Distribution Coefficient;
- Temperature controls the distribution coefficient; and
- The solute bulk concentration of both phases.

iii. Interfacial area

- Temperature controls the interfacial tension;
- Type and degree of the amount of energy input in the system cause a more close dispersion of the two phases;
- Physical properties of the system includes interfacial tension; and
- Solvent/Feed ratio.

2.3. Solvent selection

In extraction processes, selection of the solvent is an imperative decision. The following criteria must be considered for solvent selection (Keller & Humphrey, 1997):

Distribution coefficient – A high value of the distribution coefficient indicates a high solvent affinity for the solute, which permits lower solvent/feed ratios.

Separation factor – A high value of the separation factor (β_{CA}) is desired in order to reduce the number of equilibrium stages required. The separation factor can be expressed as the ratio of activity coefficients for solute/solvent and carrier/solvent pairs at infinite dilution.

$$\beta_{CA} = \gamma_{AB}^o / \gamma_{CB}^o \quad (2.8)$$

Density and viscosity – A large difference in density between the extract and raffinate phases permit high capacities in extraction devices using gravity for phase separation. High viscosities lead to difficulties in pumping, dispersion, and in rates of mass transfer.

Recoverability of solvent - A clean and efficient separation of the solute and solvent is desirable for solvent recovery.

Solubility of solvent - Mutual solubility's of the carrier and solvent must be low. If there is a significant solubility of the solvent in the carrier, an additional distillation column would be needed to recover the solvent.

Interfacial tension – A low interfacial tension helps the dispersion of the phase and improves contacting efficiency and is thus called “easy” systems. However, they are slow to coalesce, and they require longer contacting times for phase separation. Understanding of coalescing mechanisms is the key challenge in extraction research.

Availability and cost –The solvent must be available at a reasonably low cost.

Toxicity and flammability – Toxicity and flammability of the solvent are vital occupational health considerations.

Stability – Considerations of the stability of solvent are crucial, especially if the solvent is susceptible to decomposition or polymerisation, or it tends to react with any of the components in the feed.

Corrosivity – Corrosiveness of the solvent can damage the construction material. Corrosion rates need to be considered if corrosion is a problem.

Compatibility – Some solvents can contaminate food and pharmaceutical products, and therefore may not be right in such applications.

2.4. Extraction equipment

2.4.1. Column selection

Generally selection of an appropriate extractor is based on a large number of factors. Table 2.2 lists the advantages and disadvantages of the various types of extractors. Figure 2.6 illustrates a selection scheme for commercial extractors according to the stages and maximum throughput required.

Table 2.2: Advantages and disadvantages of different extraction equipment (Seader and Henley, 2006)

Class of equipment	Advantages	Disadvantages
Mixer-Settlers	<ul style="list-style-type: none"> Good contacting Handles wide flow ratios Low headroom High efficiency Many Stages available Reliable scale-up 	<ul style="list-style-type: none"> Large hold-up High power cost High investment Large flow space Inter-stage pumping may be required
Continuous, counter-flow contactor (no mechanical drive)	<ul style="list-style-type: none"> Low initial cost Low operating cost Simplest construction 	<ul style="list-style-type: none"> Limited with small density difference Cannot handle high flow ratios
Continuous, counter-flow contactor (mechanically agitated)	<ul style="list-style-type: none"> Good dispersion Reasonable dispersion Many stages possible Relatively easy to scale-up 	<ul style="list-style-type: none"> Limited throughput with small density difference Cannot handle emulsifying systems Cannot handle high flow ratios
Centrifugal extractors	<ul style="list-style-type: none"> Handles low-density difference between phases Low hold-up volume Short hold-up volume Small inventory of solvent 	<ul style="list-style-type: none"> High initial cost High operating cost High maintenance cost

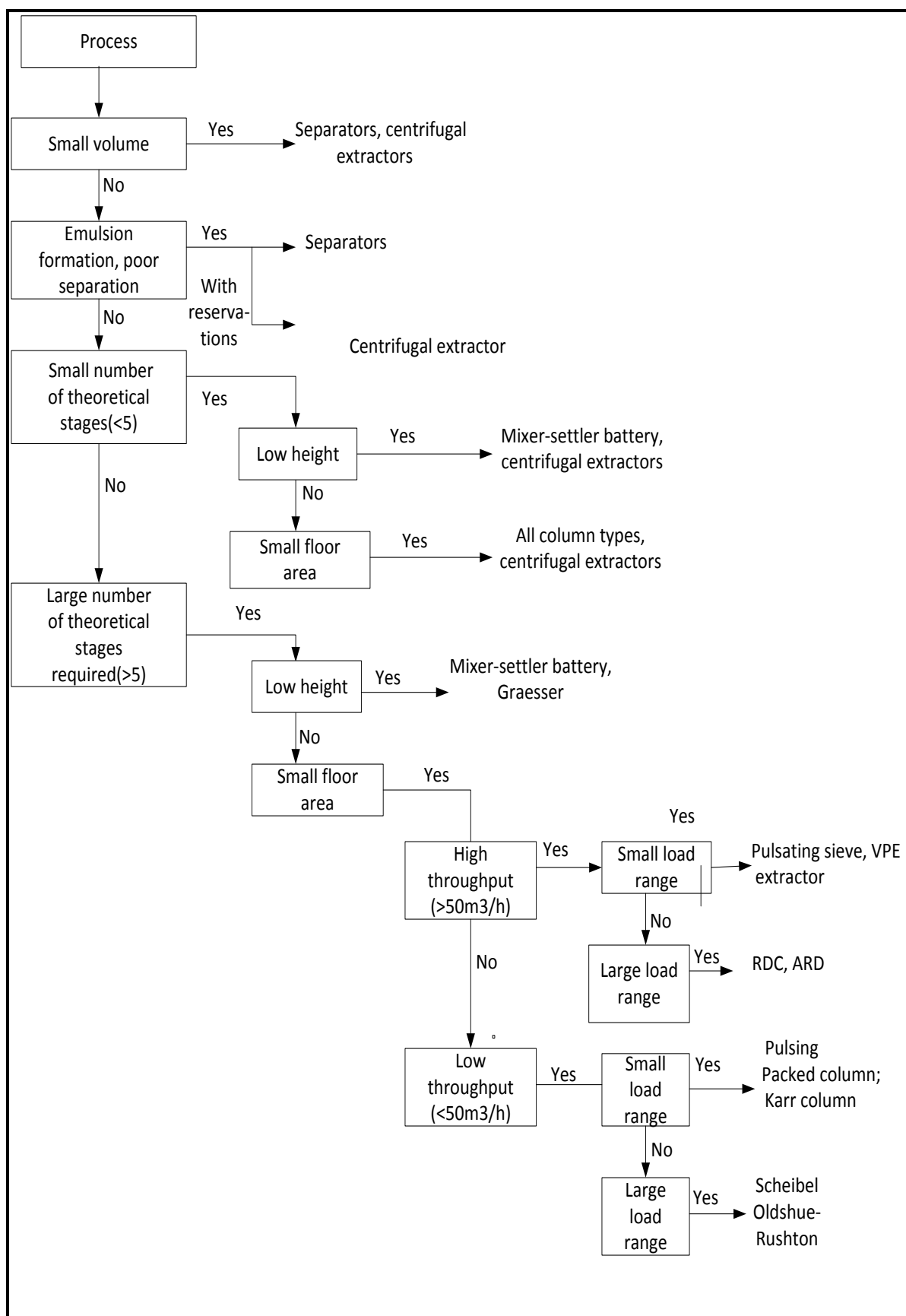


Figure 2.6: Schematic for selecting extractor (Seader and Henley, 2006)

2.4.2. Types of extraction columns

There are many devices that may be used for extraction purposes such as gravity or centrifugal force columns and columns that may be static or agitated in order to improve the interfacial area.

2.4.2.1. Non- mechanically agitated columns

Figure 2.7 illustrates some static column extractors that are discussed below

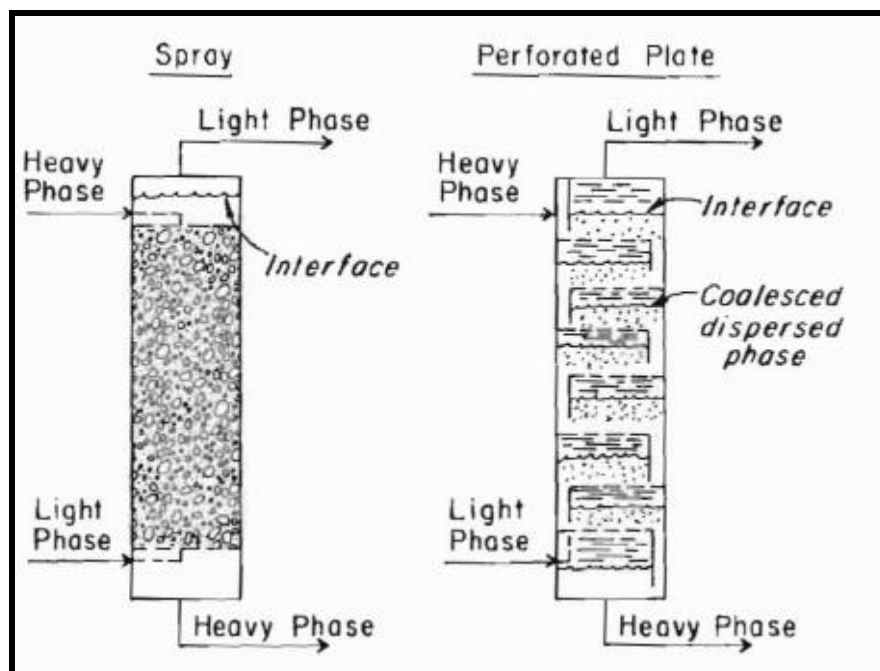


Figure 2.7: Static column extractors (Fair and Humphrey, 1983)

Spray columns

These columns are the oldest and the simplest extraction columns that are used in the industry for simple operation like washing. They have very low cost and efficiencies because of high back-mixing in the continuous phase which limits these columns to an operation of one or two stages (Fair and Humphrey, 1983; Lo, 1997; Mohanty, 2000).

Perforated plate column

These columns have a semi-stagewise operation and are reasonably flexible and efficient. Normally, if the light phase is dispersed, the fluid flows through the perforations of each plate and is dispersed through the continuous phase. The continuous phase flows through the down comer of each plate to the plate beneath. However, if the continuous phase is dispersed, the column is reversed and up-comers are used for the continuous phase (Fair and Humphrey, 1983; Keller and Humphrey, 1997).

Packed columns

A packed extractor is more efficient than a spray extractor. The packing material reduces back mixing in the continuous phase. This results in better mass transfer. It is essential that the packing material be wetted by the continuous phase in-order to prevent coalescence of the dispersed phase. Normally packed columns are preferable over spray columns due to the reduced flow capacity (Keller and Humphrey, 1997; Mohanty, 2000).

Pulsed column

The efficiency of sieve plate and packed columns is increased by the application of an oscillating pulse to the contents of the column which increases both interfacial area and turbulence. A well distributed turbulence promotes mass transfer efficiency and dispersion at low HETS or HTU values. A uniform distribution of drops is attained producing high efficiencies with low axial mixing (Lo, 1996).

Pulsed perforated columns found considerable appreciation in the nuclear industry in 1950. However, their popularity declined due to mechanical problems and the difficulty of propagating a pulse through a large column. Based on pulse intensity equivalence, the reciprocation plate column has been found to have similar operational characteristics with less maintenance costs (Fair and Humphrey, 1983; Keller and Humphrey, 1997).

2.4.2.2. Mechanically aided extractors

Figure 2.8 illustrates some mechanical agitated columns that are discussed below:

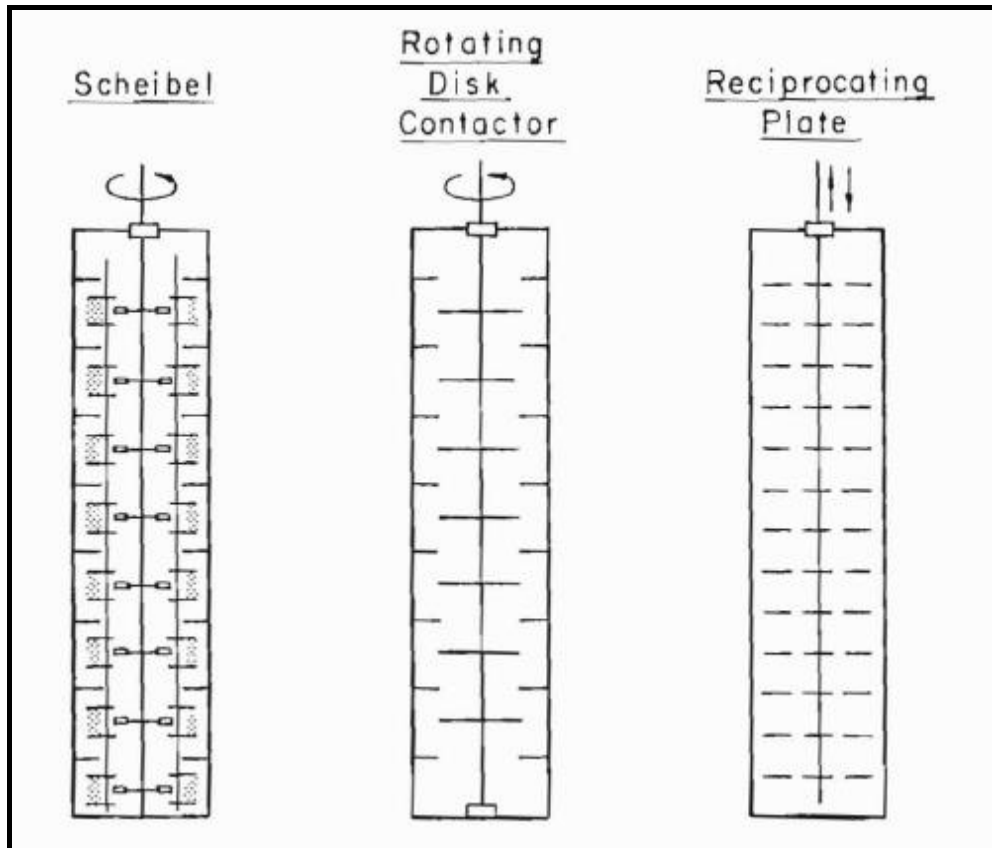


Figure 2.8: Agitated column extractors (Fair and Humphrey, 1983)

Scheibel column

Scheibel columns are under the name Yolk-Scheibel columns. These columns are designed to imitate a series of mixer settlers extraction units. Scheibel stages are limited to two stages, one for agitation and one for coalescence. It is moderately expensive with high contacting efficiency (Keller and Humphrey, 1997; Mohanty, 2000).

Rotating-disk column

Rotating-disk column (RDC) was introduced by Shell and it has been used by petroleum industries for purification of hydrocarbons. It uses a rotor that is located on a central shaft to create dispersion and for movement of the phases; hence the stator provides the counter-current motion (Fair and Humphrey, 1983; Keller and Humphrey, 1997).

Oldshue-rushton extractor

This column was developed in the 1950s and has been broadly used in petrochemical, metallurgical, pharmaceutical and fertilizer industries (Mohanty, 2000). It consists of a number of compartments separated by horizontal stator-ring baffles, each fitted with vertical baffles and a turbine-type impeller mounted on the central shaft (Oldshue and Rushton, 1951).

Kuhni extractor

The Kuhni extractor is similar to the Scheibel column, but it does not have the coalescing section. It consists of a baffled turbine impeller that promotes radial discharge within a compartment, while horizontal baffles separate the compartments (Seader and Henley, 2006). This type of column has been used in the separation of aromatic and aliphatic hydrocarbons (Mohanty, 2000).

Centrifugal extractor

The centrifugal extractor uses centrifugal forces to increase the rates of counter current flow and separation of the phases instead of using the gravity force like other extraction columns. It can handle a system characterised by emulsion. Advantages of this column include short contacting time for unstable materials and the ability to handle fluids that have small density differences. The disadvantages include complexity and high capital and operating costs (Keller and Humphrey, 1997).

Reciprocating plate column

The reciprocating plate extraction column is also known as a Karr column. Karr column uses the same hole area for the two phases and it has no downcomers. This type of column features open type perforated plates with about 60% open area (Karr, 1956). It operates only in the emulsion flow regime and has been used industrially with diameters up to and exceeding 1.0 m (Baird and Shen, 1984; Karr and Ramanujam, 1986). Reciprocating plate (Karr) columns have been found to have similar operating characteristics with pulsed columns, but due to less maintenance cost, it has gained extensive acceptance in the industry (Keller and Humphrey, 1997).

This type of extractor is used in the pharmaceutical, petrochemical, metallurgical and chemical industries (Mohanty, 2000). The advantages of the Karr column compared to other agitated columns, such as, Kuhni or Scheibel columns include higher extraction efficiency with less energy requirement and a higher throughput (Prabhakar *et al.*, 1988).

In the reciprocating plate column, phase dispersion can also be achieved by reciprocating or vibrating of the plates in the column. Reciprocating plate extractors were first proposed in 1952 and its development started in the 1960s (Lo *et al.*, 1992).

Normally, counter-current liquid extraction is performed in columns in which counter-current motion is effected only by gravity due to difference in density of the two phases. Therefore, the rate of mass transfer in these columns is limited because gravitational forces are usually inadequate for proper phase dispersion and the creation of turbulence. This results in the adoption of moving inbuilt components in order to supply additional energy to increase interfacial area and reduce mass transfer resistance. In practise this can be achieved either by introducing rotating impellers or disks or by some kind of vibration or pulsation (Vijayan and Baird, 1991).

Most of the agitated extractor columns have a tendency of reducing capacity while increasing longitudinal mixing due to the non-uniform distribution of additional energy along the column cross sectional area, leading to a uniform sized dispersion. In order to reduce longitudinal mixing in these columns, baffles have been introduced. Therefore, the baffles also reduce capacity. These conditions have led to the development of reciprocating plate columns (Prochazka *et al.*, 1971).

Vibrating plate extractor (VPE)

A modified Karr column is the vibrating plate extractor which uses perforated plates of smaller hole size and a smaller percentage hole area than the Karr column (Prochazka *et al.*, (1971). The small holes provide passage for the dispersed phase, while downcomers on each plate provide passage for the continuous phase. The down-comers permit a much higher throughput than would be possible using perforations alone (Lo *et al.*, 1992).

The vibrating plate extractor has a capability to operate in both mixer settler and emulsion regimes (Lo and Prochazka, 1983; Shen *et al.*, 1985). Due to the complexity of mass transfer and hydrodynamics in these columns, designing of vibrating plate column is difficult since there are no existing models for designing these columns. Pilot scale test is the only way of ensuring that all unknown variables or parameters are considered before large columns are designed (Lo and Prochazka, 1983).

In order to determine optimum operating agitation levels, experiments are conducted at different agitation levels and the HETSs (height equivalent to theoretical stages) are plotted against agitation levels. In order to find maximum throughput, maximum volumetric efficiency is plotted against throughput. For a large column a scale-up procedure can be used.

Generally, the following empirical equations can be used for scale-up of Karr columns (Lo and Prochazka, 1983).

$$\frac{(HETS)_2}{(HETS)_1} = \left(\frac{D_2}{D_1} \right)^{0.38} \quad (2.9)$$

D is the diameter of the column.

And the corresponding reciprocating speed needed for the larger diameter column is given by:

$$\frac{(SPM)_2}{(SPM)_1} = \left(\frac{D_1}{D_2} \right)^{0.14} \quad (2.10)$$

Where SPM is the speed of the motor in revolution per minutes or per second and amplitude is assumed to be constant (Karr, 1980).

Axial dispersion effects need to be taken into account during scale-up, since it may cause a problem if not considered properly. Axial dispersion increases with diameter because of the circulation effects (Hafez *et al.*, 1979).

Smith *et al.* (2008), subsequently investigated the performance of Karr reciprocating extraction columns. They found that there was no significant change in either the dispersed phase hold-up or mass transfer coefficient with column diameter changing. The empirical equations for scale up that were used by Karr (Lo and Prochakza 1983; Karr 1980) were too conservative. But they indicated that the factors, such as droplet and plate coalescence, contamination of liquid systems, aging of column internals, and variation in physical properties was significantly influential to the column's performance and need to be carefully considered when designing a Karr column.

2.4.3. Advantages of agitated columns

Agitation is effective because it creates turbulence which breaks up the dispersed phase droplets, thus, increasing the interfacial area or improving mass transfer area (Baird *et al.*, 1991).

2.4.4. Disadvantages of agitated columns

Agitation may create very fine droplets at the lower range of the size distribution that can result in entrainment losses. Baird *et al.*, (1991) indicated that large energy inputs can increase the axial mixing hence the overall efficiency of the column reduces.

2.4.5. Comparison of commercial extractors' performance

An exploratory comparative analysis of ten different counter current extraction columns for a toluene-acetone-water system was conducted by Stichlmair (1980). Figure 2.9 illustrates the efficiency versus capacity for several extractors.

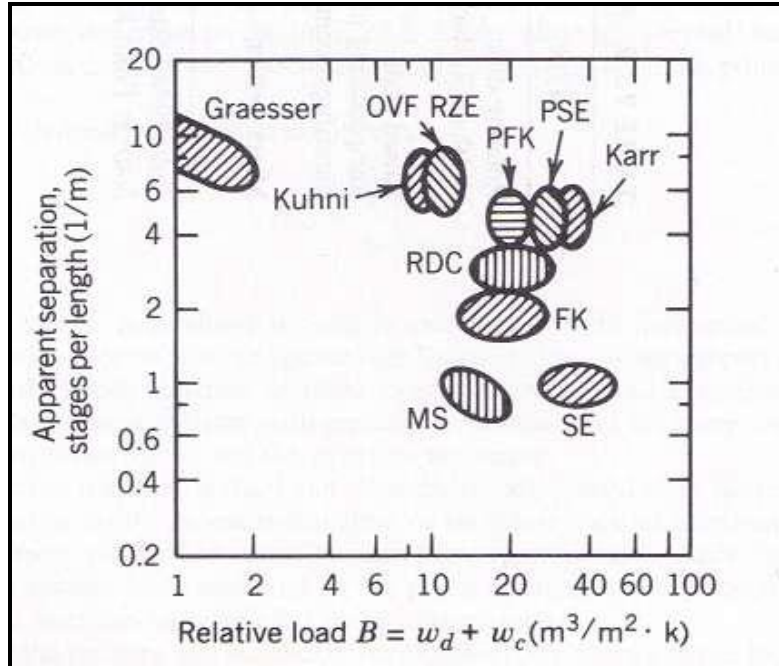


Figure 2.9: Efficiency versus total flow (capacity) for several extractors (Humphrey and Keller, 1997)
PSE (Pulsed sieve tray extraction), RDC (Rotating disc column),
QVF (Agitated cell extractor) and MS (Mixer settler)

Results for the mixer-settler in Figure 2.9 indicate that the system was operating at high efficiency. The static perforated plate column and the Karr extraction column was found to give the highest capacities for this ternary liquid-liquid system, however results also denoted that the Karr extraction column displayed significantly lower HETP ((height equivalent to a theoretical plate) values. The performance of the other types of mechanically agitated extraction columns were close to that of the Karr column, however with slightly smaller capacities

HETP values of several types of mechanically aided extraction columns are shown in Table 2.3 (Humphrey and Keller, 1997; Karr and Ramanujam, 1987 and Holmes *et al.*, 1987).

Table 2.3: Efficiencies of mechanically aided extractors (Humphrey and Keller, 1997; Karr and Ramanujam, 1987 and Holmes *et al.*, 1987).

Type of Extractor	Chemical System	HETP (ft.)
Reciprocating Plate	Toluene-Acetone-Water	0.5 – 1
Reciprocating Plate	Methyl isobutyl ketone-Phenol-Water	2 – 6
Rotating-Disc Contactor	Toluene-Acetone-Water	0.5 – 2
York-Scheibel	Toluene-Acetone-Water	0.3 – 0.8
Centrifugal	Toluene-Acetone-Water	0.6

Humphrey and Keller (1997), also illustrate a graph similar to Figure 2.9 shown in Figure 2.10 that relates the efficiency to the total flow for numerous types of liquid-liquid extraction columns. Humphrey and Keller (1997) indicated that when six or more stages are required, mechanically aided extractors should be considered.

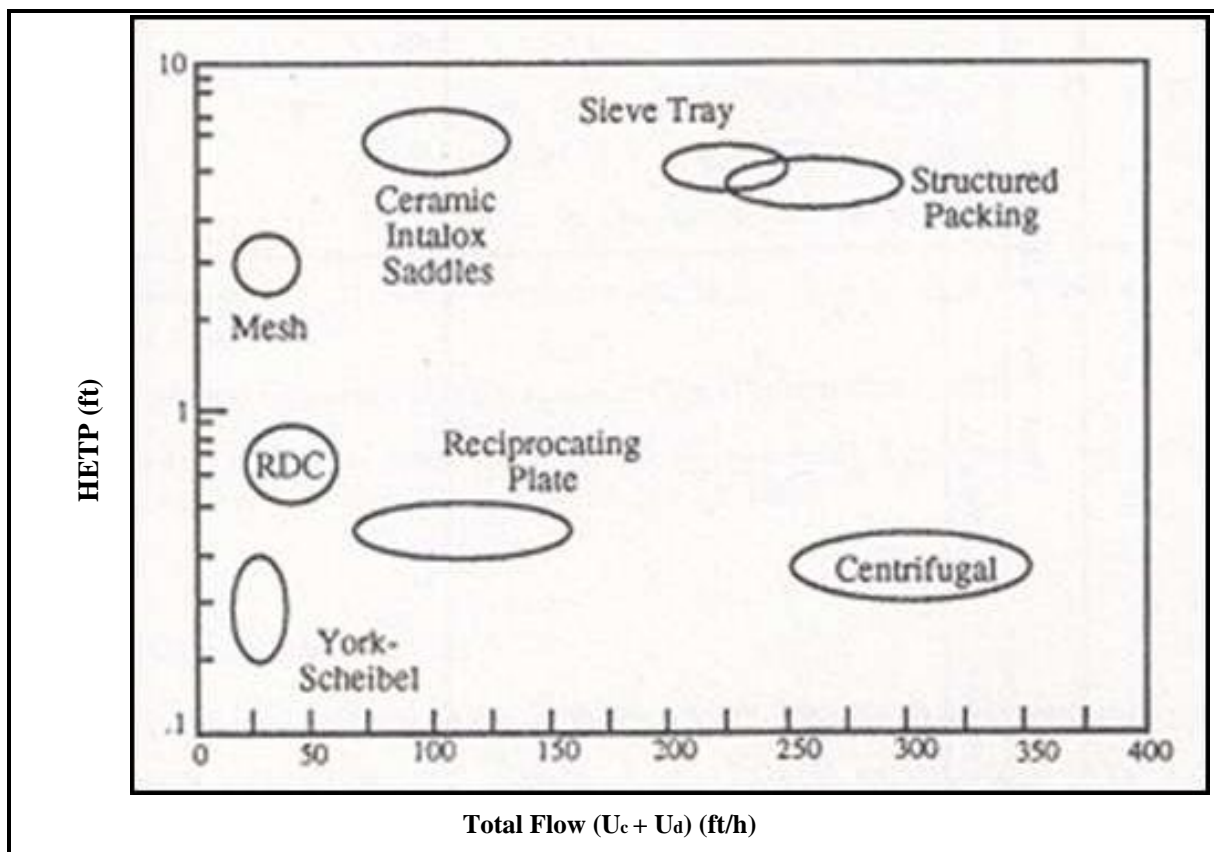


Figure 2.10: Efficiency versus total flow (capacity) for several extractors (Humphrey and Keller, 1997)

2.5. Reciprocating plate extraction column plates

Reciprocating plate extraction columns were developed in 1935 (Van Dijck, 1935). Reciprocating Plate Extraction Columns (RPCs) are used in three different types. Karr Column was developed in the Union of Soviet Socialist Republics. All of them operate on the same principles, however they are different in terms of their plate design characteristics.

Figure 2.11 illustrates other different tray design types for reciprocating plate extraction column that are used in the industry. Mounted on an upright shaft of reciprocating plate extraction columns is a stack of perforated plates, which are driven by a vibrating motor above the vibrating plate extraction column (Lo *et al.*, 1992).

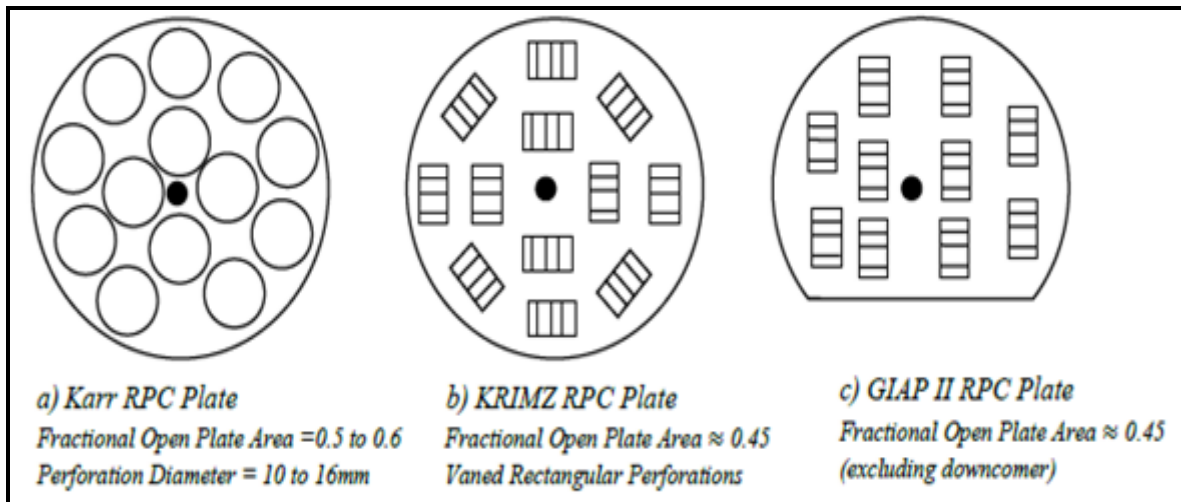


Figure 2.11: Types of RPC plates used in industry (Lo *et al.*, 1992)

Figure 2.11 indicates the other types of reciprocating plate column plates used in industry besides the Prochazka plate shown in Figure 2.12. A downcomer is represented by a tube or an opened section of a plate, (Lo *et al.*, 1992). The vibrating plate extractor which is also known as the Prochazka plate functions in the mixer-settler hydrodynamic regime or, if the VPE is operated at greater agitation rates, operates in the emulsion hydrodynamic regime, (Lo *et al.*, 1992).

When the VPE plates are compared to other types of reciprocating plate extraction columns they can operate at higher frequencies and lower amplitudes.

Prochazka *et al.* (1971) research reports that as a result of drops formed from the small perforations a considerably uniform dispersion is formed. The RPC with perforated plates and downcomers (VPE) could use a segmental downcomer for large columns instead of the tubular downcomer. The drop size is determined by the amplitude, frequency of vibration and the perforation diameter. Axial dispersion is reduced for the VPE column since the drops are evenly dispersed through the column (Prochazka *et al.*, 1971).

Lo and Prochazka (1983) developed the vibrating plate extraction column (VPE) by modifying the RPC. VPE features a smaller hole diameter which facilitates the movement of the dispersed phase while downcomer(s) facilitate the movement of the continuous phase as shown in Figure 2.12. Vibrating plate extraction columns have a small fractional open plate area compared to RPC plates. It has a fractional open plate area of approximately 0.04 to 0.3 mm² excluding the downcomer(s) and smaller hole diameter of approximately 2 to 5 mm (Lo *et al.*, 1992). Reducing sieve hole diameter typically enhances the tray capacity by 3 to 5 percent Sieve holes smaller than 5mm are used in clean, noncorrosive services (Green and Perry, 1997).

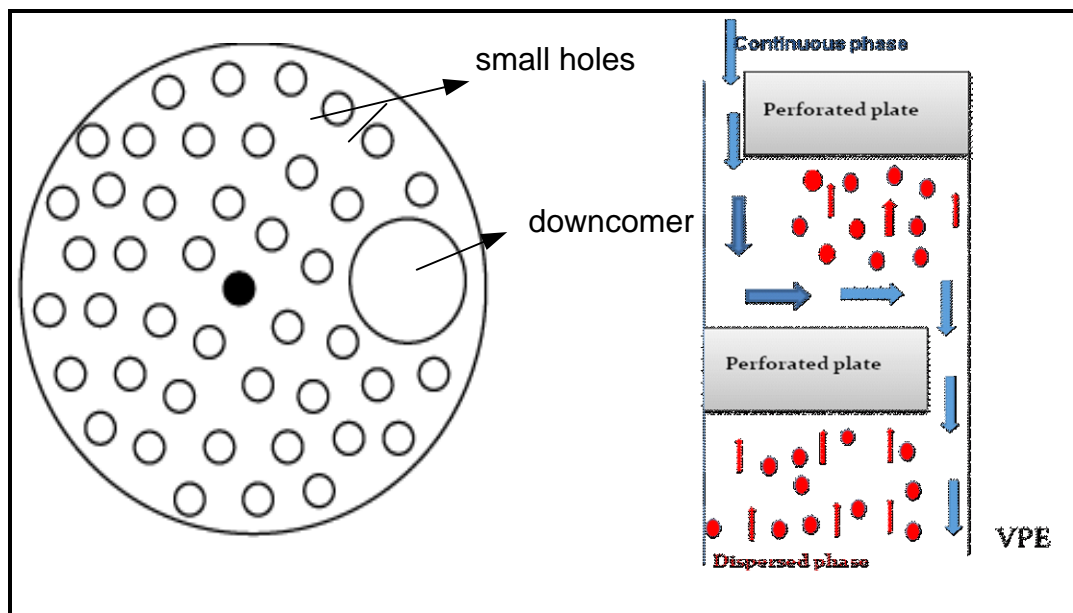


Figure 2.12: Prochazka RPC plate (Lo *et al.*, 1992)

Types of downcomers

Downcomers are used to guide liquid flow from an upper tray to a lower tray. The most common type of the downcomer is the segment type that can be straight, sloped or angled. The vertical straight segment downcomers are widely used as it provides good consumption of the column area for down flow and has a cost and operational simplicity advantage. Sloped or angled downcomers are commonly used if LLE is complicated (for example due to foaming). Sloped downcomers also provide a slightly larger active area for liquid-liquid contact; nevertheless, they are more expensive (Green and Perry, 1997).

It is imperative to design a downcomer which is sufficiently large in order to allow liquid to flow smoothly without choking. Enough time must also be given in the downcomer to allow liquid separation from the down flowing liquid, so that the liquid is relatively free of other liquid when it enters a tray below. Inadequate or not enough downcomer area will lead to downcomer choking, whereby liquid backs up the downcomer into the tray above and eventually floods the column (Green and Perry, 1997).

The straight, segmental vertical down comer is the most common downcomer geometry. Circular downcomers (down pipes) are cheaper, but poorly utilize tower area and are only suitable for very low liquid loads. Sloped downcomers improve tower area utilization for down flow (Green and Perry, 1997)

Downcomers provide sufficient area and volume for liquid-liquid disengagement at the top, gradually narrowing as the liquid disengages, minimizing the loss of bubbling area at the bottom of the downcomers. Sloped downcomers are vital when large downcomers are required at high liquid loads, high pressures, and foaming systems. Typical ratios of downcomer top to bottom areas are 1.5 to 2 (Green and Perry, 1997).

2.5.1. Advantages of vibrating plate extraction column

According to Prochazka *et al.* (1971) and Lo *et al.* (1992), the following are the advantages of vibrating plate extraction columns.

- Ability to operate at high flow rates, therefore, achieving a high efficiency;
- Easy modification to differences in conditions;
- Ability to adapt to an extensive array of system properties;
- Easy and simple construction and maintenance of the column; and
- Simple scale-up.

2.5.2. Disadvantages of vibrating plate extraction column

According to Rama Rao *et al.* (1991) and Takacs *et al.* (1993), the following are the disadvantages of vibrating plate extraction columns.

- For larger energy aids, axial mixing is increased, thus, reducing overall effectiveness of the column;
- Sensitive to impurities - not suitable for liquid mixtures containing solid materials; and
- Fine droplets result in entrainment problems.

2.6. Hydrodynamic conditions

2.6.1. Hydrodynamic regimes

Vibrating plate extractors can operate in three flow regimes depending on the mixing energy inputs viz. mixer settler, dispersion and emulsion regimes (Grinbaum, 2006; Nemecek and Prochazka, 1974).

The mixer settler regime

In the mixer settler regime a layer of concentrated dispersion forms on the plate; whereby the thickness of the layer changes periodically and the hold-up of the dispersed phase in the remaining volume is insignificant. The drop size is not uniform. No back flow of the dispersion through the plate occurs.

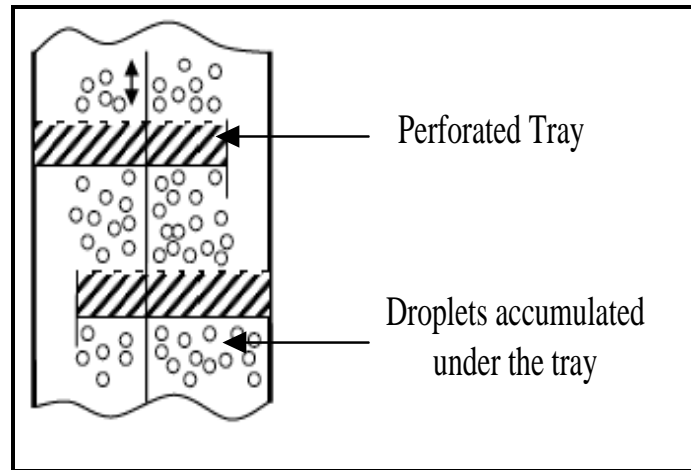


Figure 2.13: mixer-settle regime (Lo *et al.*, 1992)

The dispersion regime

It is where the dense layer of the drops on the plate increases over the height of the stage. The drops are relatively large and uniform and move mostly in the vertical direction. No back flow of the dispersion through the plate occurs.

The emulsion regime

It is where the dispersed phase is uniformly distributed over the height of the stage. The drops are relatively small and move randomly giving rise to their back flow through the plate. At high pulsation velocities, flooding occurs due to excessive intensity of vibrations, the dispersed phase hold-up increases extremely, causing the droplets to coalesce faster. The dispersion thus grows strongly non-homogenous and motion of the large drops resembles the mixer settler regime while the motion of the small droplets retains the character of the emulsion regime. Most of the industries do not want to operate at this regime (Grinbaum, 2006).

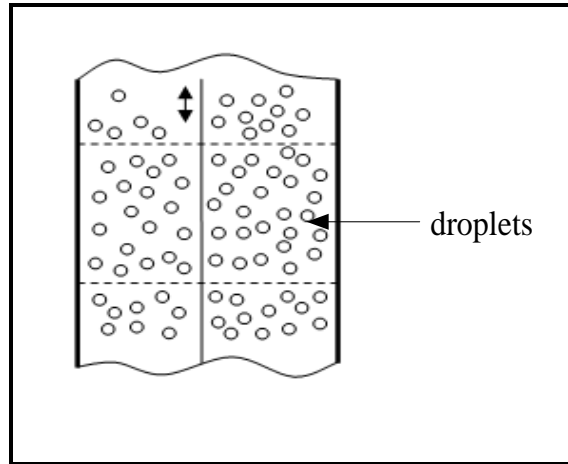


Figure 2.14: Dispersion regime (Lo *et al.*, 1992)

2.6.2. Hold-up and flooding

In most cases the phase that is selected as the continuous phase is the phase that preferentially wets the column and internals in-order for the droplets to move freely within the continuous phase. The total dispersed phase in the active part of the column at a steady operation is called the effective volume (holdup) of the column (Laddha and Degaleesan, 1983).

For a counter-current column it is essential to make sure that the rate of drop arrival at the principal interface does not exceed the rate of coalescence to prevent the build-up of drops at the interface which will result in the flooding of the column.

In a sieve plate column, stationary hold up, including the coalesced layer formed under each plate is the result of three major effects attributable to continuous flow rate, orifice resistance, and interfacial tension effects.

2.6.3. Dispersed phase hold-up

When a solvent is introduced into a vibrating plate extractor it causes droplets to undergo repeated coalescence and breakage, resulting in an equilibrium drop size distribution. The resulting fractional volumetric holdup, x_d , is defined as the volume fraction of the active section of the column that is occupied by the dispersed phase:

$$x_d = \frac{V_d}{V_c} \quad (2.11)$$

Where, V_d is the volume of the dispersed phase and V_c is the total volume of the two phases in the effective length of the column. The dispersed phase holdup is a key variable in the design of solvent extraction columns as it is related to the interfacial area for mass transfer, a given by

$$a = \frac{dx_d}{d_{32}} \quad (2.12)$$

2.6.4. Drop size distribution.

A critical parameter required for the design and efficient operation of extraction columns is the Sauter mean drop diameter. The Sauter mean drop diameter is established on the mean drop size of a population of droplets in the extraction column (Boyadzhiev and Spassov, 1982). The Sauter mean diameter plays a fundamental role in the hydrodynamics of the column since it affects the dispersed phase hold-up, the residence time of the dispersed phase and the throughputs (Kumar & Hartland, 1996). According to Boyadzhiev and Spassov (1982), the Sauter mean diameter may be defined by the following equation:

$$d_{32} = \frac{\sum n_i d_i^3}{\sum n_i d_i^2} \quad (2.13)$$

Where n_i is the number of drops of diameter d_i (mm) Baird *et al.* (1991), in their investigation found that frequency of agitation levels had a more significant effect on the droplet size than the amplitude. They also suggested that the data for a given flow rate and system could be correlated quite well with the product af^2 . They then proposed the empirical correlations for Sauter mean drop diameter as follows:

$$d_{32} = 0.001 \exp(-X) \quad (2.14)$$

Where

$$X = 8.98 \times 10^{-5} af^2 d_o^{0.5} U_o^{-1.2} U_d^{-1.5} \Delta\rho^{-1} \sigma^{-0.4} \quad (2.15)$$

Then the above correlation becomes

$$d_{32} = 0.001 \exp(-8.98 \times 10^{-5} af^2 d_o^{0.5} U_o^{-1.2} U_d^{-1.5} \Delta\rho^{-1} \sigma^{-0.4}) \quad (2.16)$$

2.7. Axial mixing in extraction columns

2.7.1. Introduction to axial mixing

Ideal or plug flow has been assumed in the prediction of the height of an extraction column for a specified separation. In fact, a part of each main phase flow is mixed back. Thus, these back mixing currents, so-called axial mixing currents, and flow counter currently to the main phase flows. A non-uniform movement of the continuous phase through the extraction column that leads to a drop in the driving force for mass transfer is axial dispersion (Stella and Pratt, 2006).

Due to the fairly low superficial velocities of the flowing streams, reciprocating plate columns (RPC) are typically prone to axial mixing effects (Baird, 1974; Stella and Pratt, 2006). The scale-up of extraction columns is affected by axial dispersion. For example, the axial dispersion effects may increase with the column diameter causing a poorer large scale performance (Rosen and Krylov, 1974).

Stella and Pratt (2006) stated the following to be contributing factors to the continuous phase axial dispersion:

- Entrainment of the continuous phase in the wake of droplets;
- Different sizes of droplets due to their size distribution;
- Energy dissipation of droplets causing a circulatory movement of the continuous phase;
- Turbulence, mainly due to agitation;
- Circulation or flow non uniformity mainly due to the flow of dispersed droplets; and
- Axial transport in the wake of droplets.

2.7.2. Effect of axial mixing on extraction efficiency

The interactions between these effects are quite complex. For example, increasing mechanical agitation can reduce circulation and therefore in some cases reduce the efficiency. Extractor designs are established on the basis of plug flow patterns of each phase. In practice, this assumption is incorrect, as a result of deviations caused by axial mixing. Axial mixing thus results in a decrease in the concentration driving force, which in turn causes an increase

in the HTU or HETS values. Figure 2.15 depicts the effect of axial mixing on the concentration profiles in a counter-current extraction column (Wasowski and Blab, 1987).

Axial mixing negatively affects the mass transfer in column extractors. This is due to the fact that axial mixing significantly reduces the driving forces for mass transfer by reducing the concentration difference between the phases. This is particularly observed at both entries of a column where concentration jumps are produced due to axial mixing, see figure (2.15) below.

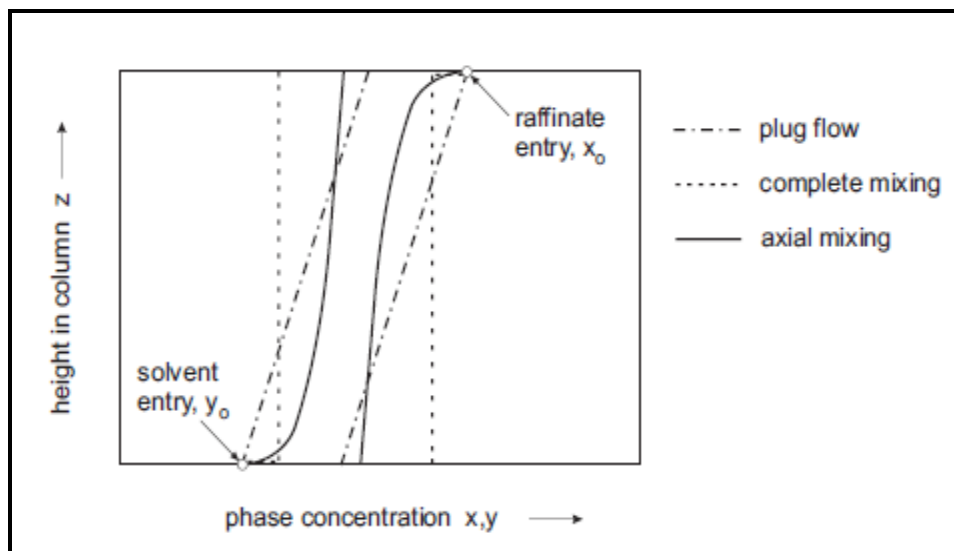


Figure 2.15: Influence of axial mixing on the concentration profile (Wasowski and Blab, 1987)

The reduction of the concentration difference by axial mixing can be understood by analysing, for instance, the solute concentration of the raffinate phase. The solute concentration of the raffinate phase is reduced on its way through the column (see figure 2.15). If a fraction of the raffinate phase which has already passed through a section of an extractor is mixed back into this section, it will be mixed with the new incoming raffinate.

Since the back mixed raffinate has a lower solute concentration than the incoming raffinate, the concentration of the mixture is reduced. Subsequently, the concentration difference between the main phases is reduced (Daniel, 2006). Similarly, the solute concentration of the solvent phase is increased by axial mixing. This again results in a reduction of the driving concentration difference (see Figure 2.15).

2.7.3. Axial mixing correlations

The effects of axial mixing on the reciprocating plate and pulsed perforated-plate columns for single phase flow were first explored by Novotny *et al.* (1970) and thereafter Nemecek and Prochazka (1974). The model developed by these researchers was centered on the backflow model. The backflow model assumes that axial dispersion is caused by both backflow of the liquid through the perforated plate and by axial mixing between neighbouring plates in the extraction column.

Table 2.4 contains different correlations developed for axial mixing in reciprocating plate columns (Novotny *et al.*, 1970; Nemecek and Prochazka, 1974; Kostanyan *et al.*, 1980)

Table 2. 4: Axial dispersion correlations

Karr (RPC) Column (Single phase system)	
$q_c = \left\{ \left(1 + \frac{1}{q} \right) \exp \left[6.6(h - 4.5) \frac{\varphi^{3/2} U_c}{d_o a f} \right] \right\}^{-1}$ $q = \frac{\alpha}{\pi} - \frac{1}{2} + \frac{2af}{U_c}$ $\alpha = \arcsin \left(\frac{U_c}{2\pi a f} \right)$	Novotny et al., (1970)
Vibrating Plate Column (Two phase system)	
$q_c = \frac{U_c + U_d}{U_c} \left(\frac{\phi 1}{\pi} - \frac{1}{2} + \frac{2af}{U_c + U_d} \cos(\phi 1) \right) + \frac{U_d}{U_c} (1 + q_d)$ $\phi 1 = \arcsin \left(\frac{U_c + U_d}{2af} \right)$	Nemecek & Prochazka (1974)
Vibrating Plate Column (Two phase system)	
$q_c = \frac{h}{100} \left\{ \frac{1}{0.5U_c + af} + 8 \left(\frac{\delta}{d_o} \right)^{1/3} \left(\frac{\left(\frac{h}{1000} \right) + D_c}{D_c} \right)^{4/3} \frac{\varphi^{2/3}}{af} \right\}^{-1}$	Kostanyan et al., (1980)

2.8. Back mixing in the continuous phase

For designing liquid extraction columns, such as the reciprocating plate column, it is important to be able to predict the column height required for a specified separation. Ideal or plug flow has been used in the past and has proven not to be successful in predicting performance. So a number of empirical relationships have been used to scale up pilot tests.

The major deviation from ideal or plug flow is continuous phase axial dispersion, which is a non-uniform flow of the continuous phase through the column that leads to the reduction of the effective driving force for mass transfer.

Factors that contribute to the non-uniform flow of the continuous phase include:

- Entrainment of the continuous phase in the column;
- Wake of the dispersed-phase droplets;
- Circulatory flow of the continuous phase caused by energy dissipation of droplets; and
- Molecular and turbulent eddy diffusion as well as channelling and stagnant flow effects.

Dongoankar *et al.* (1999) found that there is no significant effect of backmixing in the dispersed phase. Some other studies conducted by Miyauchi and Oya (1965) and Kumar and Hartland (1989 & 1999) have focused on the prediction of the axial mixing coefficients in the continuous phase, given that it is normally acceptable that back-mixing effects from the dispersed phase are not insignificant.

Novotny *et al.* (1970) and Nemecek and Prochazka (1974) were first to investigate longitudinal mixing in the reciprocating plate and pulsed, perforated plate columns for single phase flow. They developed a model based on the backflow model and assumed axial dispersion resulted from both backflow of liquid through the plate holes and axial mixing between neighbouring plates. Nemecek and Prochazka (1974) later presented the role of axial dispersion in a vibrating sieve plate column for two-phase flow.

Hafez *et al.* (1979) presented results on axial mixing for two-phase operation in a 150 mm diameter reciprocating plate column. Two highlighted differences were underlined that are different to previous work conducted by Kim and Baird (1976). At low levels of agitation, the axial-dispersion coefficient increased for the larger column, whereas no effect was present in the single-phase smaller column. The axial-dispersion coefficient was influenced by phase velocity in the larger column but not in the single phase 50 mm column.

2.9. Number and heights of transfer units

The mass transfer effectiveness for liquid-liquid extraction processes is represented as the height of a transfer unit (H_{ox}). It is correlated considering the axial mixing and depends on the flow rates, the mass transfer direction and the agitation level (Shen *et al.*, 1985).

According to Shen *et al.* (1985), the mass transfer is more effective which entails that a lower height of transfer unit was attained for mass transfer from the continuous phase to the dispersed phase.

Chilton and Colburn, (1935) developed the correlation for the true number of transfer units represented by Equation 2.17, where a is the interface area per unit volume of the column (m^2/m^3)

$$N_{ox} = \frac{K_c a H}{U_c} \quad (2.17)$$

In support of the piston model, the measured number of transfer units is depicted by Equation 2.18 (Usman *et al.*, 2006) where inlet and outlet molar concentrations in the continuous phase, (mol/L) is represented by x_i and x_o respectively, while x_e is the equilibrium molar concentration in the aqueous phase and x , the molar concentration in the continuous at any point in the liquid-liquid extraction column. The measured number of transfer units can be launched by graphical or numerical integration using the known concentration profile along the column (Usman *et al.*, 2006).

$$N_{oxm} = \int_{x_i}^{x_o} \frac{1}{x_e - x} dx \quad (2.18)$$

In integrating Equation 2.18 and considering the operating and equilibrium curves as straight lines, the understandable number of transfer units can be established from Equation 2.19 (Usman *et al.*, 2006). The outlet and inlet molar concentrations in the dispersed phase is represented by y_o and y_i respectively, whereas m shows the slope of the equilibrium curve.

$$N_{oxp} = \frac{1}{[(y_1 - y_0)/m(x_0 - x_1)] - 1} \ln \left(\frac{y_1 - mx_0}{y_0 - mx_1} \right) \quad (2.19)$$

Shen *et al.*, (1985), initiates the idea of true heights of a transfer unit, H_{ox} is depicted by Equation 2.20, where H is the height of the active part of the extraction column.

$$H_{ox} = \frac{H}{N_{ox}} \quad (2.20)$$

From the investigation conducted by Baird and Shen (1984), it was shown that the true height of a transfer unit is significantly larger for mass transfer from $d \rightarrow c$ direction than for mass transfer from $c \rightarrow d$ direction. This is due to the fact that there is a large drop size and lower holdup which resulted in a greatly decreased mass transfer performance. Consequently, this shows that the mass transfer is incapable per unit height of dispersion when the mass transfer is from the $d \rightarrow c$ direction (Shen *et al.*, 1985, Baird and Shen, 1984).

Carmurdan's (1986) research work indicates that a minimum HETS can be achieved at agitation levels below the flooding point because of the severe axial mixing effects taking place at high agitation levels. Carmurdan (1986) also shows that it would be very efficient if the holdup was set at a constant value, since in this way the extraction can be operated around the minimum HETS values which in turn results in an increase in the extraction column efficiency.

Rathilal *et al.* (2010) conducted an investigation on vibrating plate extraction columns, which led to the development of a model to estimate the number of transfer units (NTU). It also shows that the agitation level (as the product of the amplitude and frequency of vibration), tray spacing as well as the solvent to feed ratio are some of the dependent variables for the determination of the number of transfer units.

Their study also shows that the NTU remained fairly constant in the mixer settler regime. Thus, as the dispersion flow regime is approached there is an exponential increase in the NTU. The NTU correlation developed by Rathilal *et al.* (2010) is illustrated by

$$N_{oxm} = \left[5.5 + \left(1.8e^{0.258(af)} - 5.5 \right) u \right] \times L \times \left(\frac{100}{h} \right) \quad (2.21)$$

Where L is the solvent to feed ratio (S/F), h represents the tray spacing in mm and u is a unit step function. For the mixer settler regime and dispersion regime u is stipulated as:

$$u = 0 \text{ for } (af) < 3.75 \text{ mm/s} \quad (\text{Mixer settler regime})$$

$$u = 1 \text{ for } (af) \geq 3.75 \text{ mm/s} \quad (\text{Dispersion regime})$$

The NTU model developed by Rathilal *et al.* (2010) can also be used for the prediction of HTU or HETS, by substituting Equation 2.22 in Equation 2.21 resulting in Equation 2.23, where H is the actual height of the extraction column and HTU represents the height of a transfer unit

$$HTU = \frac{H}{N_{oxm}} \quad (2.22)$$

$$\frac{H}{HTU} = \left[5.5 + \left(1.8e^{0.258(af)} - 5.5 \right) u \right] \times L \times \left(\frac{100}{h} \right) \quad (2.23)$$

2. 10 Mass transfer coefficient

The mass transfer coefficient can be calculated using Equation 2.24, where N_{ox} is equivalent to NTU, active height of the column, Sauter mean diameter (m) and the superficial velocity of the dispersed phase (m/s) (Rathilal et al, 2010)

$$K_{oxm} = \frac{N_{ox} U_d d_{32}}{6\phi H} \quad (2.24)$$

Mass transfer coefficient correlation was estimated using the model that was developed by Rathilal *et al* (2010) as shown in Equation 2.25, where the tray spacing is in mm, and the agitation level in mm/s. According to Rathilal et al (2010) the following model only applies to the dispersion flow regime and emulsion flow regime, because the dispersed phase holdup

in the mixer settler flow regime includes the coalesced dispersed phase layer accumulated underneath the perforated plates.

$$K_{ox} = -0.007(af) + 0.06 + \frac{h}{10000} \quad (2.25)$$

2.11. Efficiency for extraction columns

The stage efficiency is used in the extraction in order to relate the performance of a real stage to that of an ideal theoretical stage.

2.11.1. Overall efficiency

According to Pratt (1983), overall efficiency, E_o , of an extraction column is equal to the ideal number of stages divided by the number of real stages needed to obtain the same duty that has a similar concentration change. Equation 2.26 shows the overall efficiency where N represents the number of stages

$$E_o = \frac{N_s(ideal)}{N_s(real)} \quad (2.26)$$

2.11.2. Murphree efficiency

Murphree efficiency is expressed as the actual concentration change of the phase within the stage divide by the concentration change that would have occurred if the equilibrium had been achieved, with the Murphree efficiency being expressed in terms of either the X phase, which is the dispersed phase or Y phase, which is the continuous phase (Pratt, 1983). Equation 2.27 and 2.28 illustrate the Murphree efficiency for both phases utilizing the mass fractions.

$$E_{Mx} = \frac{x_{n-1} - x_n}{x_{n-1} - x_n^*} \quad (2.27)$$

$$E_{My} = \frac{y_n - y_{n+1}}{y_n^* - y_{n+1}} \quad (2.28)$$

where x_{n-1} and y_{n+1} are the concentrations for the streams entering the stage, x_n and y_n are the concentrations for the streams leaving the stage and x_n^* and y_n^* represent the concentrations at equilibrium conditions as shown in Figure 2.16 (Pratt, 1983).

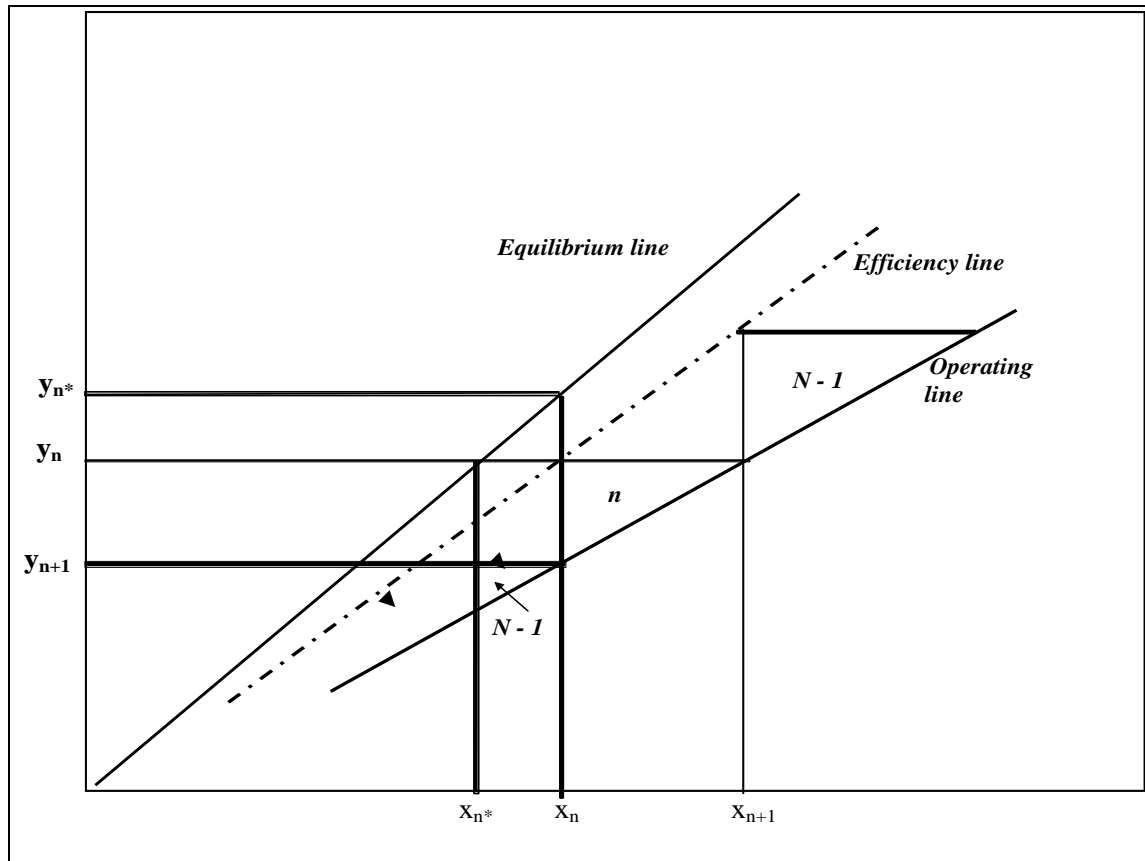


Figure 2.16: x-y Plot for the determination of the Murphree efficiency (Pratt, 1983)

CHAPTER 3: METHODOLOGY

3.1. Choice of system

The study of LLE was done using the toluene-acetone-water system with acetone extracted from the toluene, which is the dispersed phase, by water acting as the solvent and the continuous phase. The toluene-acetone-water system is a standard test system for liquid extraction as recommended by the European Federation for Chemical Engineering (EFCE, 1985).

The chemical system of toluene-acetone-water used has the advantage of having high accuracy and repeatability when analyzed using gas chromatography (Saïen and Daliri (2008). Technical grade of acetone and toluene was used in this research. Tap water was used as the solvent to extract acetone from toluene.

The solvent to feed ratio was maintained at 1:1, since the sieve trays were designed in such a way that the dispersed phase area and the continuous phase area are almost equal.

According to Rathilal *et al.* (2010) an optimum time for a run to reach steady state was 45 min. All experiments were conducted for a period of 45 min (Rathilal *et al.*, 2010).

3.2. Experiment set-up

The experimental part of this study was divided into two portions (hydrodynamics and mass transfer). For mass transfer, 6% acetone in toluene was used as the dispersed phase and tap water was used as the continuous phase due to the density difference between two phases (aqueous and organic phases).

For the hydrodynamics part, only the toluene was used as the dispersed phase and tap water was used as the continuous phase. The interface was located at the top of the column, since the light phase is dispersed and the heavy phase was the continuous phase. Toluene and water are completely immiscible (Keller and Humphrey, 1983; Chowdhury *et al.*, 2008).

3.2.1. Experiment objectives

The main objective was to investigate the effect of tray design in order to enhance the performance of the vibrating plate extractor. The effective tray design was determined by the mass transfer efficiency (percentage of acetone extracted).

Other research objectives were to test the effect of tray downcomer types, frequency and amplitude of plate vibrations, and the tray hole diameters on the number of stages in order to optimise the efficiency of a vibrating plate extraction column. The effects of the agitation level, tray downcomer types, and the plate hole diameters on the mass transfer in the vibrating plate extractor was investigated experimentally and the results were compared with lately published correlations.

This experimental procedure is divided into two sections. The first part entails the investigation of the effect of hydrodynamics on mass transfer efficiency and the second part deals with the effect of tray design on mass transfer efficiency.

Part1: Hydrodynamic Experiments

The hydrodynamic experiment was done using a water-toluene system. The tray downcomer types and agitation level were varied in order to investigate the effect of mass transfer on:

- dispersed phase holdup
- drop size distribution
- Sauter mean diameter

Part2: Mass Transfer Experiments

The Mass transfer experiments were conducted using the acetone-water-toluene test system, with 6 percent of acetone in toluene as the feed. In order to investigate the effect of

- tray downcomer type on the dispersed phase holdup
- tray downcomer type on drop size distribution
- tray downcomer type on Sauter mean diameter
- tray hole diameter on the dispersed phase holdup
- tray hole diameter on drop size distribution
- tray hole diameter on Sauter mean diameter

- agitation level on amount of acetone extracted

3.3. Equipment description

3.3.1. Surge tanks

Surge tanks were introduced by Rathilal *et al.* (2010) in order to control the flow through the rotameters or to eliminate fluctuations in the rotameter readings. Three identical surge tanks were used, two for the dispersed phase and one for the continuous phase or solvent. The surge tanks that were used are illustrated in figure 3.1.

For the feed one surge tank was introduced after the pump before the rotameter to prevent fluctuations of the rotameter caused by the peristaltic motion of the pump and the second one was introduced after the rotameter to prevent the fluctuation caused by pressure variation at the bottom of the column due to the vibration of the plates in the column.

For the continuous phase the surge tank was introduced after the pump before the rotameter to stabilise the flow fluctuations caused by the pump vibration. These surge tanks were designed to hold a maximum liquid of 2 litres. The outside diameter and the height of these tanks were 115 mm and 265 mm, respectively. Surge tanks were pressurised at the top with compressed air.

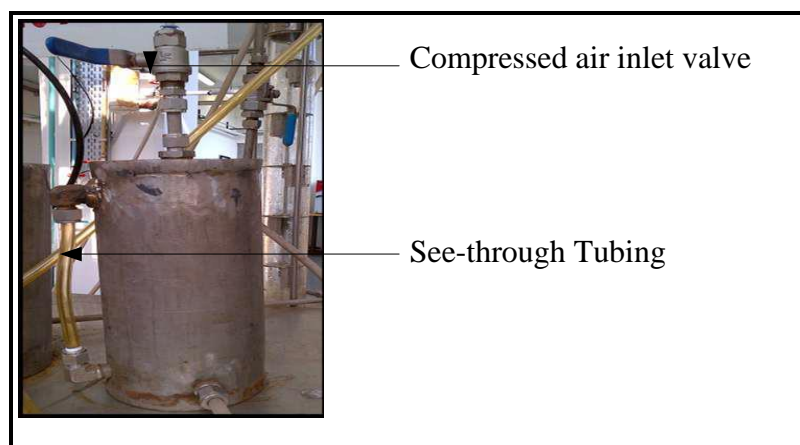


Figure 3.1: Surge tank

3.3.2. Peristaltic pumps

Three peristaltic pumps were used. One was used for pumping the feed, another pump was used for pumping the solvent and the third pump was used for the extract as well as for draining. The extract pump was also used for controlling the principal interface level. The drain/extract pump was connected to the conductivity probe and the level controller. Rotameters were calibrated using a stopwatch and measuring bucket method.

3.3.3. Level controller

The level controller was connected to the conductivity meter. The probe was designed in such a way that it was able to detect the change in conductivity in either of the liquids when it comes in contact with. Since water conductivity is low compared to the conductivity of the hydrocarbon substance (toluene), the change in conductivity was used to set the speed of the drain or extract pump. When the interface level is high the probe senses the conductivity of water and the drain pump operates to drain the water, but with a low interface level the probe senses the conductivity of toluene and the drain pump is shutdown.

3.3.4. Perspex box

The box had dimensions of 130 x 135 x 250 mm and was situated between plates 11 and 12. The Perspex box was open on top to allow water to be filled and consisted of the drainage line at the bottom. Figure 3.2 illustrates the perspex box used.

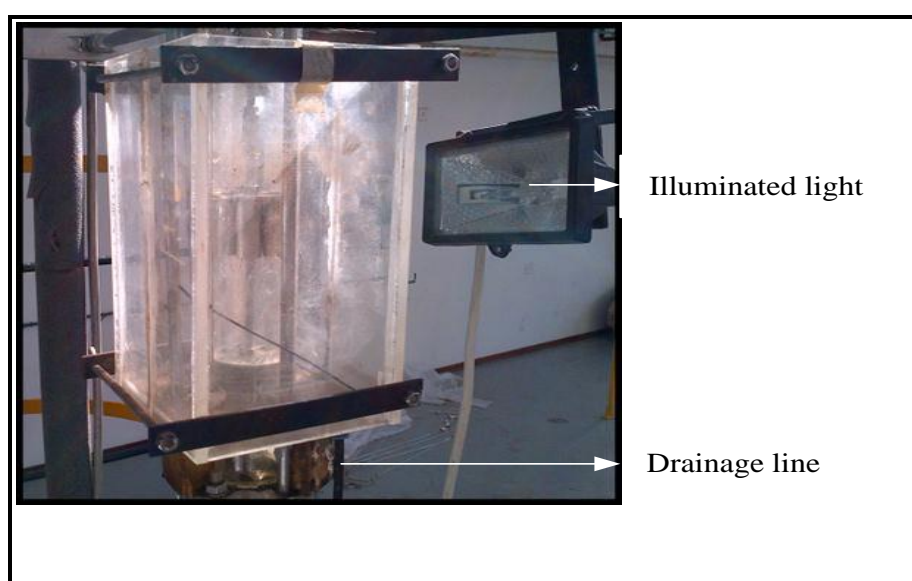


Figure 3.2 Perspex box

3.3.5. Column

The active part of the column consisted of 8 glass tubes that were flanged together giving an effective height of 4.76 m. The glass tube specifications are given in table 3.1.

Table 3.1: Glass tubes specifications

Definition	Values
Outside diameter (mm)	58.7
Inside diameter (mm)	47.7
Column thickness (mm)	5.7
Length (mm)	550
Cross sectional area (m ²)	1.787×10^{-3}

3.3.6. Settling tanks

Settling tanks were used to allow the separation of phases to take place. Two identical settling tanks were used, one was placed at the top and one at the bottom. The top settling tank was used to maintain the interface between the two phases as shown in Figure 3.3 while the bottom settling tank consisted of the distributor for the dispersed phase.

Table 3.2: Settling tanks specifications

Definition	Values
Outside diameter (mm)	160
Inside diameter (mm)	150
Thickness (mm)	5
Length (mm)	250
Cross sectional settling tank area (m ²)	17.671×10^{-3}

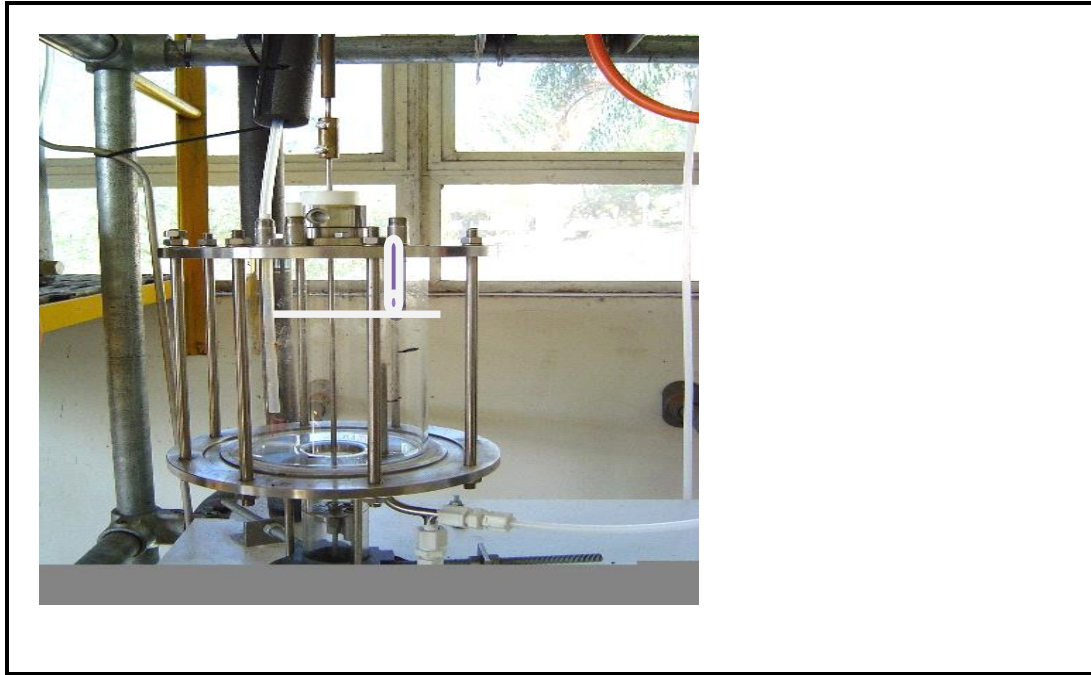


Figure 3.3: Top settling tank with the interface

3.3.7. Vibration motor

The plate stack was mounted eccentrically to a motor at the top of the column. By adjusting the space between the connection point and the centre of the motor using an adjusting yoke, the amplitude of the vibration could be changed. The specifications of the motor were as follows; 220Volts; 50 Hz; 0.75kW; 1430 rmp and 3.37 amps.

3.3.8. Water feed tank

The water tank was selected to hold maximum water for two complete runs and the water for washing the column between runs. Tap water was used for this study.

3.3.9. Feed, extract, and raffinate tanks

Three 25L tanks were used and their capacity was sufficient for the duration of the experiment.

3.3.10. Samplers

The samplers had screws at the ends that controlled the position of Teflon plugs in a glass tube. The adjustment of the screw controlled the flow of the fluid in the sampler. The dispersed phase sampler had a Teflon tip and was faced downwards opposite to the flow direction of the dispersed phase. The Teflon has a preferred wettability for the organic phase and as a result it was possible to remove only the organic phase from that sampler.

The continuous phase sampler had a stainless steel tip and was faced upwards opposite to the flow direction of the continuous phase. The stainless steel has a preferred wettability for the continuous phase and as a result it was possible to remove only the continuous phase from that sampler.

3.3.11. Gas chromatograph

A 0.5 μL sample from the extraction column was injected through the septum of the injector into the packed column in the gas chromatograph. Nitrogen was used as a carrier gas taking the sample through the packed column. Hydrogen gas and air were used to keep the flame inside the gas chromatograph ignited. The specifications of the gas chromatograph used in this research are shown in the Table 3.3.

Table 3.3: Gas chromatograph Specifications

Name:	Shimadzu GC – 2014
Column Name:	Chromosorb WHP SE 30
Column Temperature ($^{\circ}\text{C}$):	90
Column Pressure (kPa):	0.4
Column Flow (ml/min):	25
Column Length (m):	3
Detector Temperature ($^{\circ}\text{C}$):	250
Injector Temperature ($^{\circ}\text{C}$):	200

3.4. Sieve tray / plate design

A sieve tray is a perforated tray with or without the downcomer. The advantage of the perforated tray with downcomers is that, the downcomers allow for a much higher throughput than would be possible using the perforations alone.

Two sets of sieve trays with different downcomer types were used. The down-comer(s) that were used were circular pipes and straight segment as illustrated in figure 3.4 (a) and 3.4 (b). Due to the column diameter size the sloped downcomer design could not work. Sieve trays were designed in such a way that hole diameters were equal for different types of downcomers

All designs ensured that the free area available for the flow of the two liquids were approximately the same. After the more effective down comer was selected, the sieve tray hole diameters were varied in order to check which diameter is more effective in order to improve interfacial area available for mass transfer. The hole diameters that were tested were 1.5 mm, 3 mm and 4.5 mm. as illustrated in figure 3.5 (a), 3.5 (b) and 3.5 (c). Table 3.4 provides the tray specifications.

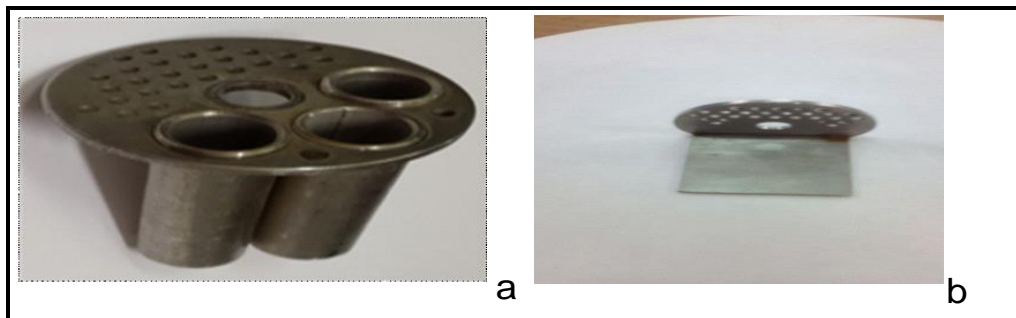


Figure 3.4: Sieve tray (a) with circular downcomers and (b) with straight segment

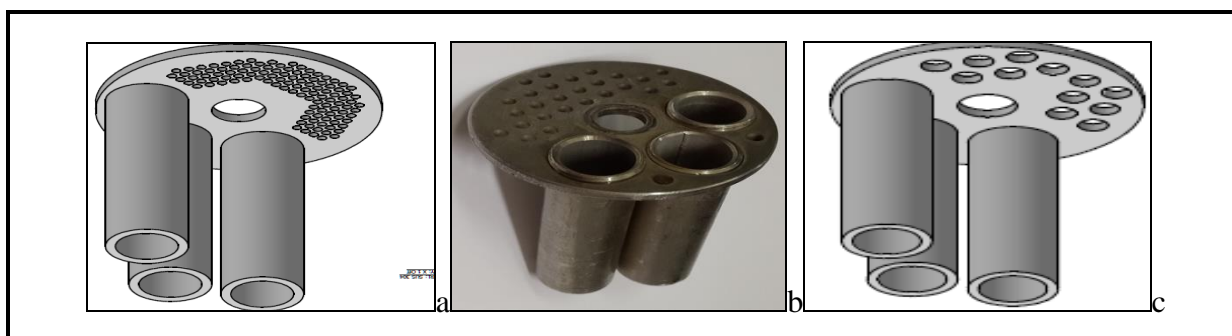


Figure 3.5: Sieve Tray:(a) with hole diameter = 1.5 mm, (b) 3.0 mm, and (c) 4.5 mm.

Table 3.4: Perforated plate specification

Plate/Tray	Tray 1	Tray 2	Tray 3	Tray 4
Perforated Plate Diameter (mm)	47.4	47.4	47.4	47.4
Perforated Plate Thickness (mm)	2	2	2	2
Number of small holes	26	26	98	12
Hole Diameter (mm)	3.0	3.0	1.5	4.5
Type of down-comer(s)	circular	Straight	circular	Circular
Number of Down-comers	3	1	3	3
Down-comer Diameter (mm)	10.3	-	10.9	10.9
Down-comer Length (mm)	43.3	43.3	43.3	43.3

3.5. Experiment equipment

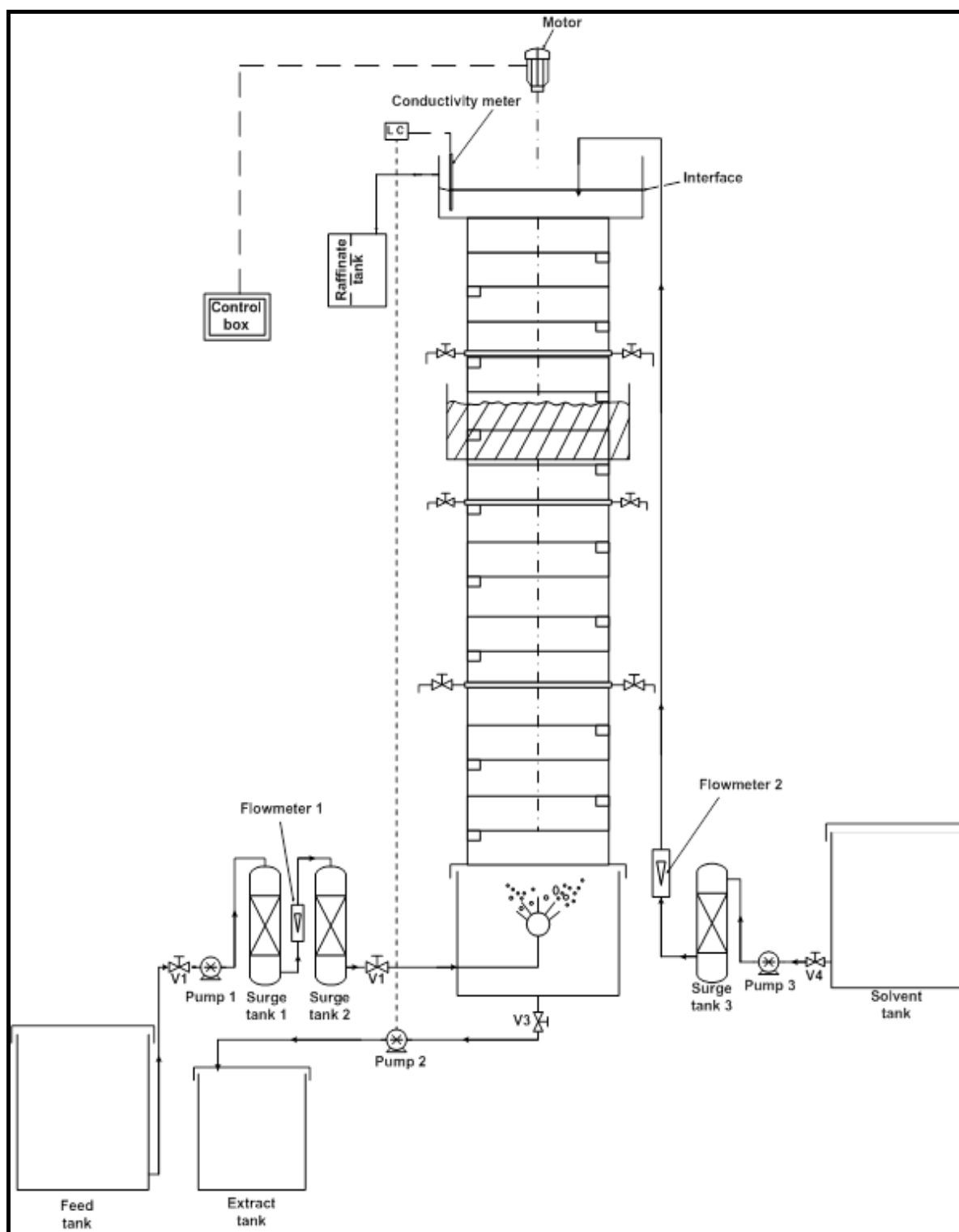


Figure 3.6: Experiment equipment

3.6. Experimental procedure (adapted from Rathilal *et al.*, 2010)

1. The conductivity probe used to maintain the interface level was marked at a fixed measured level below the raffinate overflow line
2. The column was initially filled with water until it reached the conductivity probe
3. The agitation level was set to the required value by adjusting the frequency on the vibration motor controller while the amplitude was kept constant at 2.5 mm
4. Both feed flow rates were set by adjusting the speed of the feed pumps
5. The column was allowed to run for 45 minutes to reach steady state (Rathilal *et al.*, 2010)
6. The lights surrounding the perspex box were switched on and the box was filled with water to reduce droplet size error due to the cylindrical shape of the column
7. Three to five photos were taken per run
8. The interface level in the top settling tank was marked off
9. Raffinate, extract, feed and local point samples were taken and analyzed using the gas chromatograph
10. Both feed pumps were shut down while the column was vibrated at a frequency of 1.5 Hz for a period of 20 minutes to allow all the dispersed phase droplets to coalesce and accumulate in the top settling tank
11. The amount of the toluene accumulated in the top settling tank that was found below the marked interface level was recorded
12. During drainage of the column, the water was collected in a measured bucket in order to get the active volume of the column which was used in the calculation of the dispersed phase holdup. The active part of the column starts from the first plate at the top of the column to the last plate at the bottom of the column

The above procedure was repeated for the mass transfer experiment with a feed of 6 % mass acetone in toluene

Image Pro Plus software

The photos of the droplets were analysed using the Image Pro Plus software package, to determine the drop sizes and number of droplets. Random droplets of various sizes were selected. The software highlights these droplets based on the light and dark areas of the image, the size of the droplets and the number of the same sized droplets contained in the image was established. This information was used to achieve the drop size distribution and then to calculate the Sauter mean diameters.

3.6.1 Drop size distribution or Sauter mean diameter

Firstly, the column was filled with water up to the interface level. Before carrying out the experiments, 6 percent of acetone in toluene was prepared. The vibrating amplitude and frequency were adjusted to the desired values. The continuous-phase and the dispersed-phase flow rates were then set to the required flow rate, and the system was operated to allow steady state to be reached. Once the column reached steady state, three to five photographs of the droplets were taken.

The part of the column where the photos were taken was surrounded by a perspex box filled with water in order to compensate for the curvature of the column. A ruler was placed inside the perspex box for size calibration purposes. The droplet photos were taken in order to determine the drop size distribution as a mean drop diameter. The photos were taken three times per run for repeatability purposes. Image Pro Plus computer software was used to determine the mean drop diameter and drop size distribution.

The Sauter mean diameter was calculated from equation 2.13:

$$d_{32} = \frac{6 \sum_{i=1}^n V_i}{\sum_{i=1}^n S_i} = \frac{\sum_{i=1}^n n_i d_i^3}{\sum_{i=1}^n n_i d_i^2}$$

3.6.2 The effect of agitation levels on mass transfer

Feed of 6 % acetone in toluene was prepared. Samples of each phase were taken at their inlets to the column and used for the determination of the initial solute concentration using the Shimadzu FID gas chromatograph

The column was filled up to the interface with the continuous phase. The amplitude and frequency of vibration were adjusted to the desired values and, the dispersed phase was then introduced. The interface position was then maintained at the selected height by a level controller and the system was allowed to reach steady state.

At the end of each experiment or after the system reached steady state, samples of the organic and aqueous phases were taken at their respective outlets and along the column sampling points. The solute concentration was then determined using gas chromatography.

3.6.3 Dispersed phase hold-up

The continuous-phase and the dispersed-phase flow rates were then set to the required flow rate, and the system was allowed to reach steady state. Then, the inlet and outlet flows were closed simultaneously. The dispersion was then allowed to coalesce and accumulate in the top settling tank below the interface level. The difference between the interface level before and after shutting down both feed flow rates was recorded and multiplied by the cross sectional area of the top settling tank to get the volume of toluene.

3.6.4 Fractional dispersed hold-up calculation

The active volume of the column was determined by filling up the active part of the column with tap water, and then by draining the water into a measured bucket. The fractional dispersed phase hold-up was calculated as the volume of the dispersed phase volume (toluene) divided by the active volume of the column.

CHAPTER 4: RESULTS AND DISCUSSIONS

This chapter is divided into two sections. The first part entails the investigation of the effect of hydrodynamics on mass transfer efficiency and the second part deals with the effect of tray design on mass transfer efficiency.

4.1 Hydrodynamics Experimental Results

The tray downcomer types and agitation level were varied in order to establish the effect on the dispersed phase, drop size distribution and the Sauter mean diameter. Circular and straight segment downcomers were tested at varied agitated levels from 1.25 mm/s to 7.5 mm/s with the increments of 1.25 mm/s, until flooding occurred in the extraction column. The hydrodynamic experiments were conducted for a tray spacing of 150 mm. The total throughput for the S/F ratio of 1:1 for all the experiments was kept constant at 30 l/h. All experiments were repeated to ensure repeatability of results

4.1.1 Dispersed Phase Holdup Results

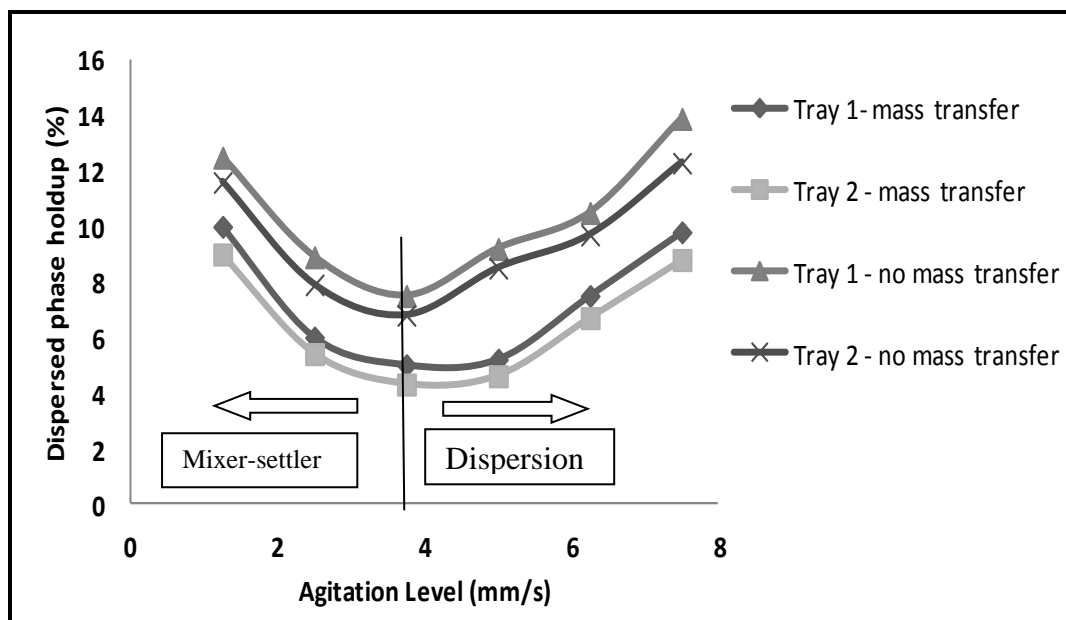


Figure 4.1: Effect of mass transfer on dispersed phase hold up

Figure 4.1 shows the effect of mass transfer on the dispersed phase hold up which shows higher dispersed phase hold up values in the absence of mass transfer and lower dispersed phase hold up values in the occurrence of mass transfer.

The result of a lower dispersed phase holdup for the mass transfer experiments can be attributed to the continuous extraction of the solute in the dispersed phase into the continuous phase during mass transfer, which results in a reduction of the dispersed phase during mass transfer. Since there is less dispersed phase present this contributes to the lower dispersed phase holdup for the mass transfer experiments.

Previous investigations conducted by Rathilal *et al.*, 2013, on vibrating plate extractions show a similar result of a higher dispersed phase holdup for the hydrodynamic experiments, except that a similar minimum holdup value was approached for both the hydrodynamic and mass transfer experiments for his results, which does not seem to be the case in Figure 4.1.

This indicates that the dispersed phase hold up without mass transfer cannot be used in designing the extraction column since it is affected by the mass transfer. A similar conclusion was also made from results obtained by Rathilal *et al.*, 2013.

Figure 4.1 also illustrates two distinct hydrodynamic flow regimes. The first flow regime is the mixer-settler regime where initially a high dispersed phase holdup was observed. This was attributed to the accumulation of the toluene layer under each tray in the column. Subsequently, as the agitation level was increased, this layer decreases, which subsequently corresponds to a decrease in the holdup.

The lowest dispersed phase holdup, which shows the transition from the mixer-settler flow regime to the dispersion regime, was observed at 3.375 mm/s for both trends. Similar trends were observed in literature during dispersion regime (Rathilal *et al.*, 2011, Aravamudan and Baird, 1999). As the agitation level was increased after the minimum holdup was reached, the dispersed phase holdup increased.

An increase in the agitation level caused the plate to vibrate faster which resulted in the formation of smaller droplets. The drag force acting on the droplets increased in relation to the buoyancy therefore the velocity of the drops decreased resulting in the longer residence time which causes a higher holdup (Camurdan, 1986, and Taylor *et al.*, 1982).

Moreover, any further increase in the agitation level in the dispersion regime, an exponential increase in the dispersed phase holdup was expected, which drives the system to the emulsion flow regime. In this regime, intensive agitation levels can result in reduction in the coalescence of droplets (Laddha and Degaleesan, 1983).

The flooding of a column may occur due to the rate of the dispersed phase droplets exceeding the rate of coalescence of the droplets (Laddha and Degaleesan, 1983). Therefore, it was assumed that the emulsion regime was not favoured since the holdup favoured to increase in an unstable manner because of flooding.

According to Aravamudan and Baird (1999), the dispersed phase holdup is a key factor determining the interfacial area for mass transfer as well as the indication of the onset of flooding.

4.1.2 Drop Size Distribution Results

The drop size distribution of the dispersed phase was established for various tray designs and various agitation levels, using Image Pro Plus software. For each hydrodynamic and mass transfer experiment several photographs of the droplets were taken and the Image Pro Plus software was used to analyse the drop size distribution.

Figure 4.2 shows the droplets for different agitation levels investigated for the tray with circular downcomers, 3.0 mm hole diameter.

It can be seen that at low agitation levels, the droplets are large and as the agitation level is increased, smaller droplets are formed increasing the surface area between the two phases.

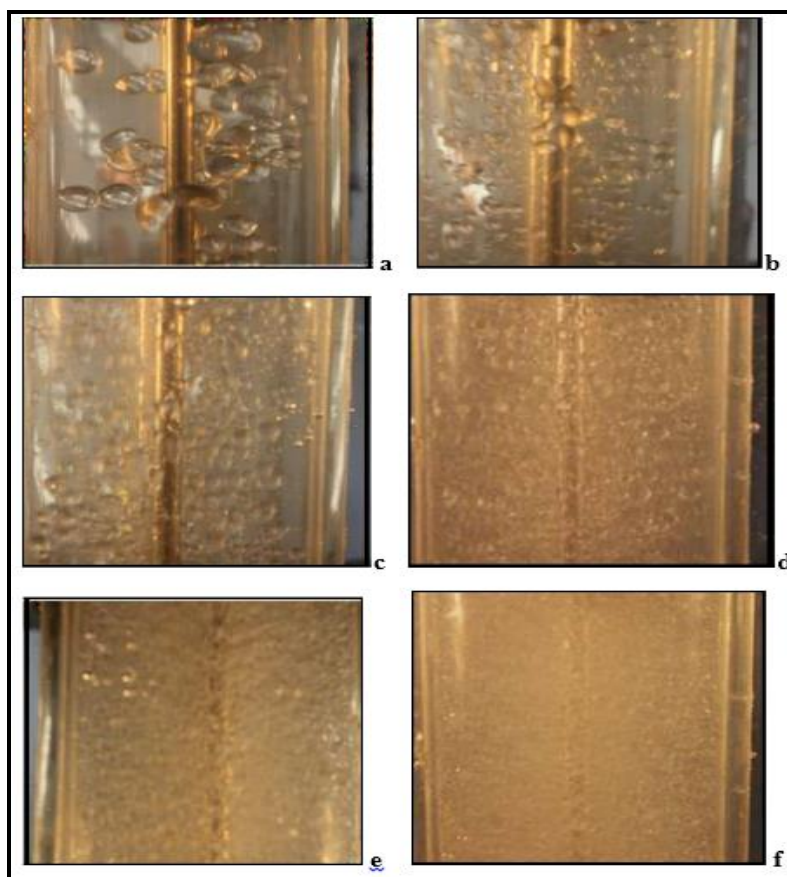


Figure 4.2 Illustration of drop sizes at various agitation levels (a)-(b)-(c)-(d)-(e)-(f) ($af=1.25$ mm/s; (b) $af=2.5$ mm/s; (c) $af=3.75$ mm/s; (d) $af=5.0$ mm/s; (e) $af=6.25$ mm/s; and (f) $af=7.5$ mm/s.)

Figures 4.3, 4.4, 4.5 and 4.6 show the drop size distribution for different agitation levels. The results show an existence of scattered size distribution indicating a wide variety of droplet sizes at lower agitation level and a narrow size distribution for higher agitation levels. This was caused by the production of smaller and more uniform droplets at higher agitation levels as a result of vigorous plate vibration.

Figures 4.3 and 4.5 illustrate the drop size distribution (dsd) where there is mass transfer of acetone from toluene to water, while figures 4.4 and 4.6 illustrate the drop size distribution where there is no mass transfer of acetone. The results show slightly larger droplets size for the case with mass transfer than when there was no mass transfer. This is due to the effect of coalescence during mass transfer. It was observed that there was no significant change in the drop size distribution for both trays with straight and circular downcomers, since the trays had similar hole diameters.

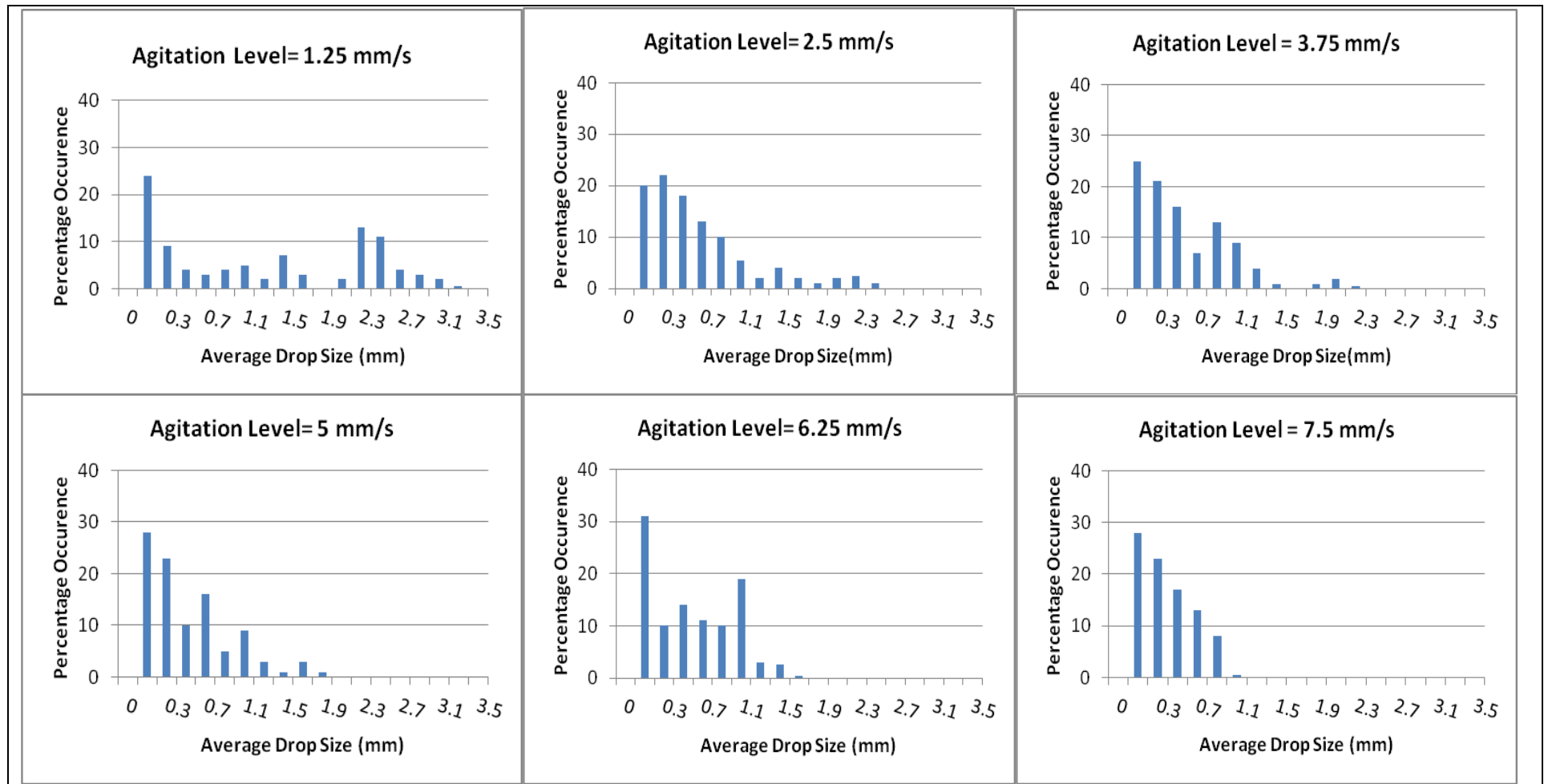


Figure 4.3: dsd at different various agitation levels for mass transfer ($d_o=3.0\text{mm}$), circular downcomers

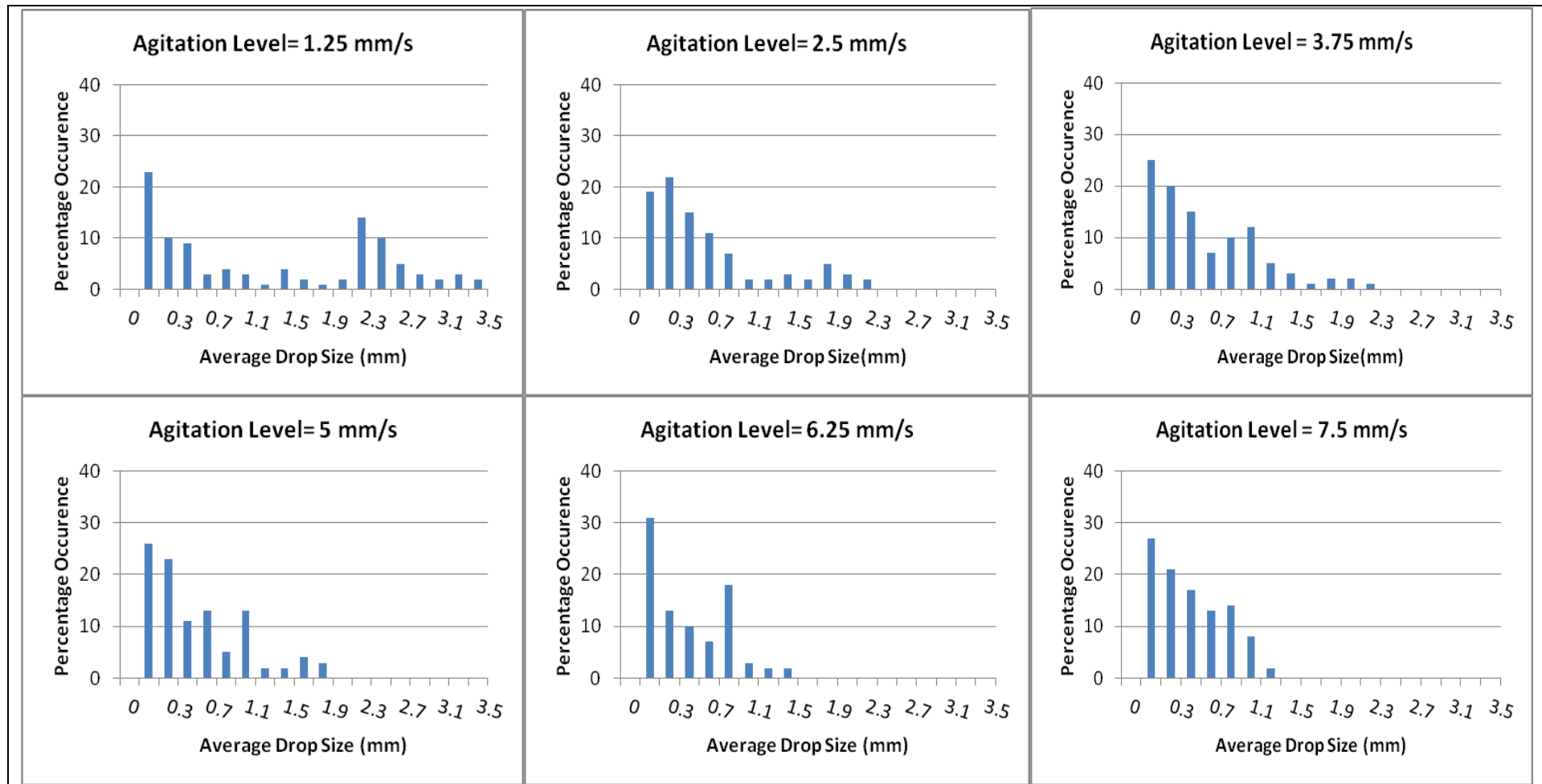


Figure 4.4:dsd at different agitation levels for hydrodynamic ($d_o=3.0$ mm), circular downcomers

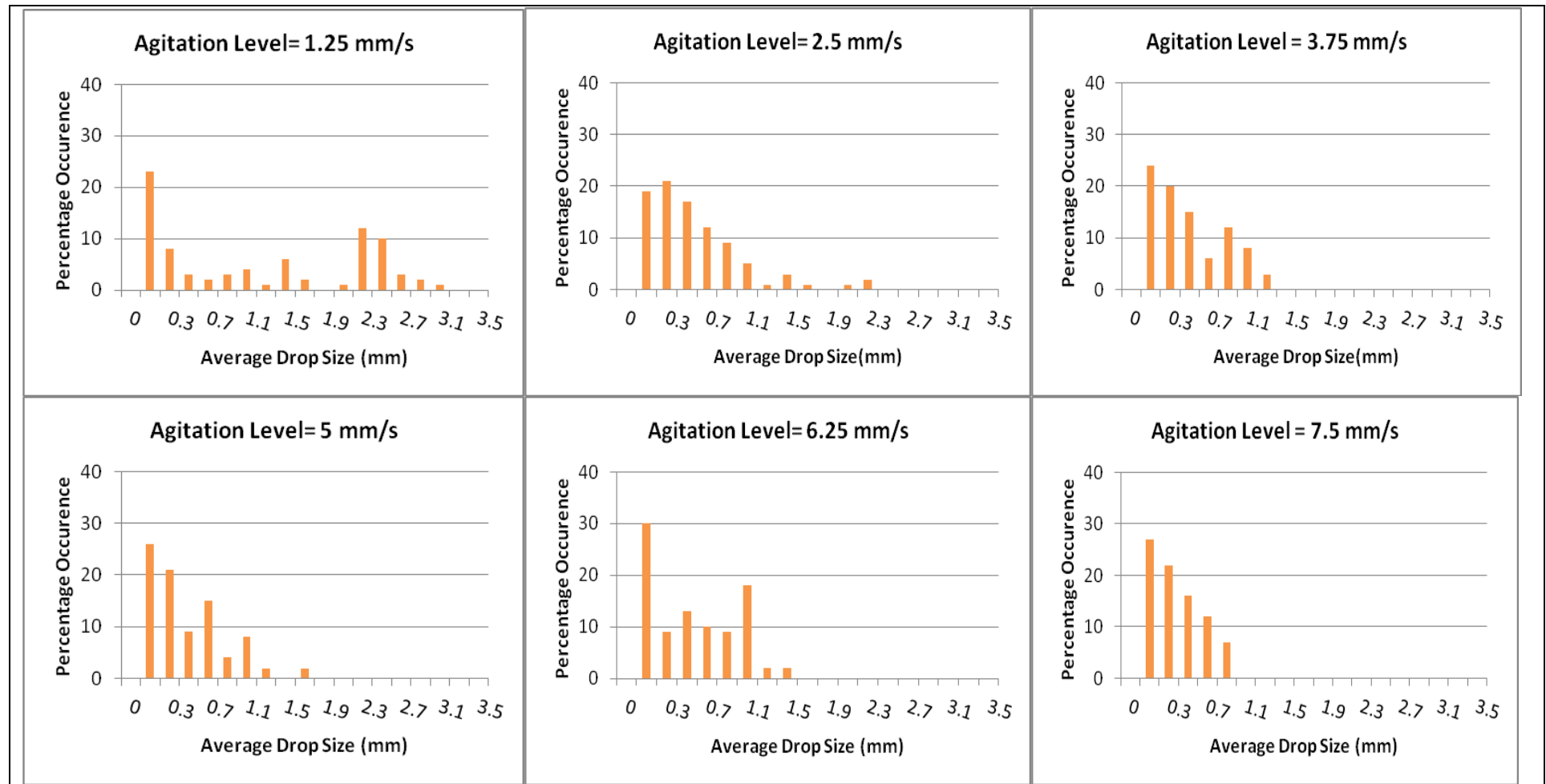


Figure 4.5 : dsd at different agitation levels for mass transfer ($d_o=3.0$ mm, Straight segment downcomer)

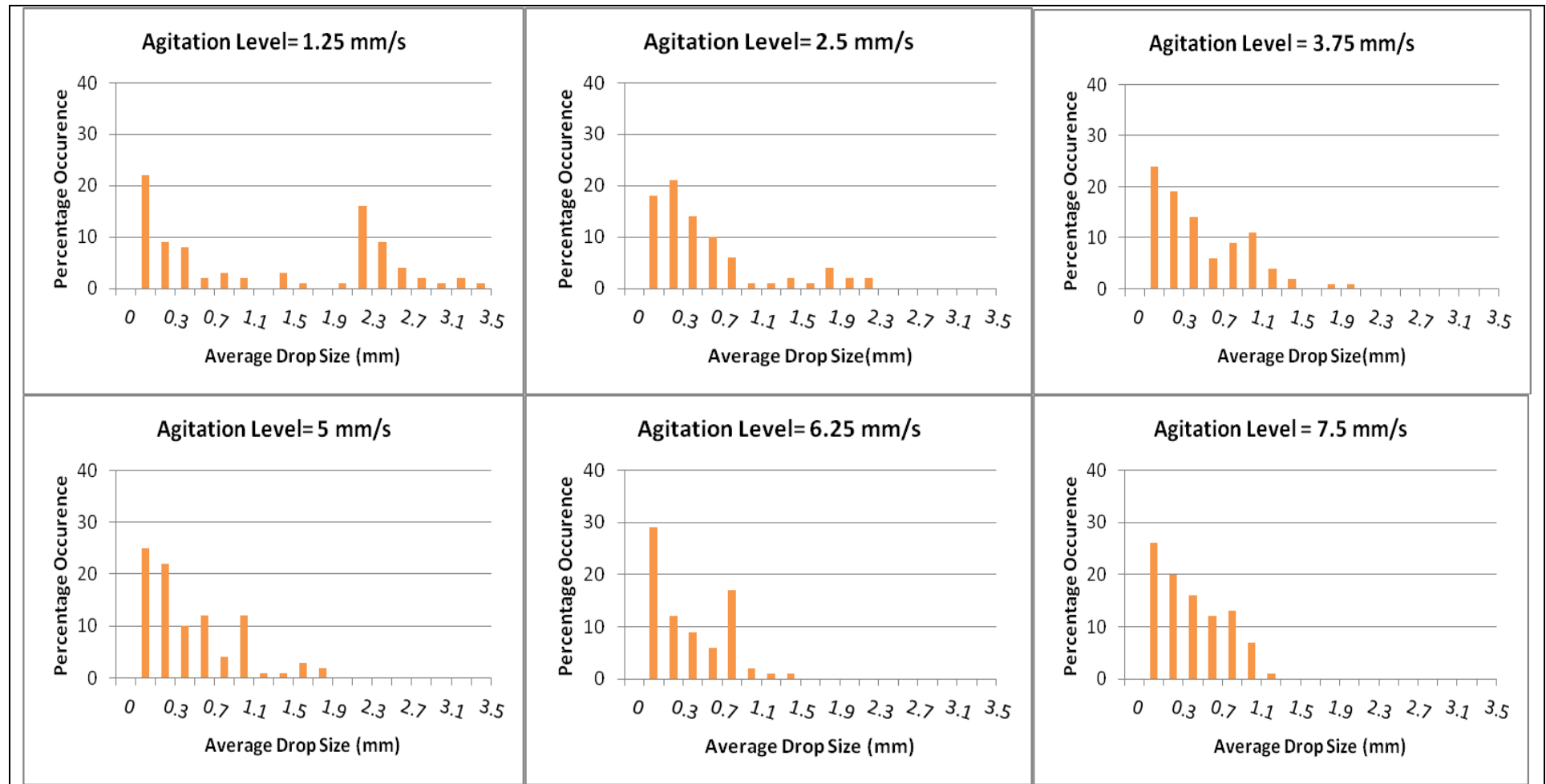


Figure 4.6 : dsd at different agitation levels for hydrodynamic ($d_o=3.0$ mm, straight segment downcomer)

4.1.3 Sauter Mean Diameter Results

The effect of mass transfer on the Sauter mean diameter was investigated using tray 1 and tray 2 to test the effects of mass transfer on the hydrodynamic conditions at a S/F ratio of 1:1, tray spacing of 150 mm and a total throughput of 30 l/hr. This effect is shown in Figure 4.7.

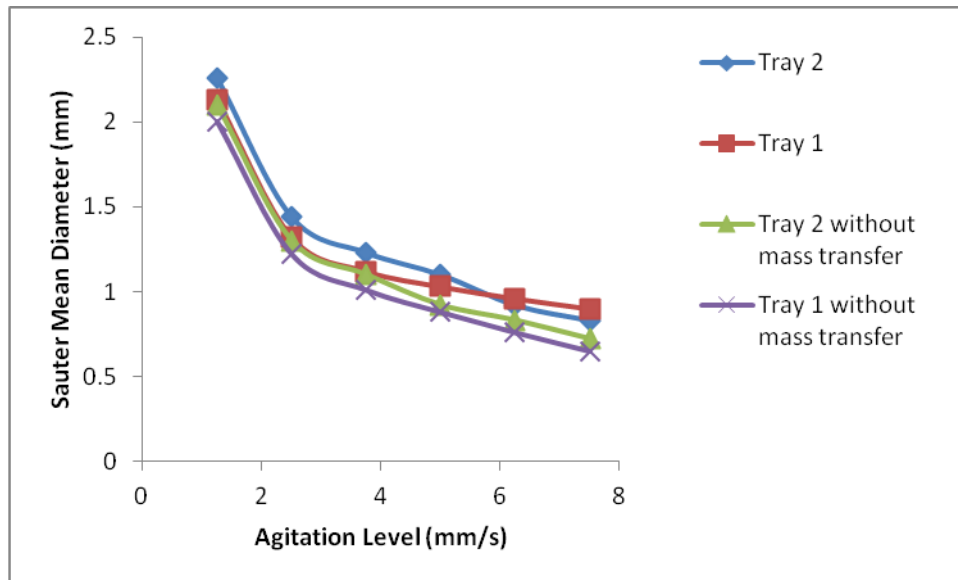


Figure 4.7: Effect of mass transfer on Sauter mean diameter

The Sauter mean diameters with mass transfer were found to be higher than those without mass transfer. This is due to the enhanced coalescence effects during mass transfer. As two drops approach each other, the surface tension is reduced due to the moving from the dispersed phase to the continuous phase. This causes the continuous phase between the drops to be drained and the drops coalesce forming bigger drops. This is consistent with literature Shen et al., (1985), and Aravamudan and Baird, (1999).

At low agitation levels there was not much difference between hydrodynamic and mass transfer experimental results. This can be contributed by small amount of acetone transfer from the dispersed phase to the continuous phase due to less energy that was added to the system.

This shows that mass transfer cannot be predicted using values of Sauter mean diameters in the absence of mass transfer as the values are affected by mass transfer. It is evident that there is a decrease in the Sauter mean diameter as the agitation level increases for both cases (with

and without mass transfer). This result is achieved since at higher agitation levels, the plates vibrated much faster. Thus more energy was dissipated to the fluid, producing much smaller size droplets.

Lo and Prochazka, 1983 noted that at higher agitation levels, the inertial and shear forces of the droplets increased resulting in the hindering of the coalescence of the droplets. This results in the smaller size droplets and a smaller Sauter mean diameter.

4.2 Mass Transfer Experimental Results

The Mass transfer experiments were conducted using acetone-water-toluene test system, 6 percent of acetone in toluene solution was used as a dispersed phase. The total throughput for the S/F ratio of 1:1 for all the experiments was kept constant at 30 l/h. The mass transfer experimental results are divided into two parts.

The first part shows the effect of tray downcomer types on the dispersed phase hold up and Sauter mean diameter. The second part shows the effect of hole diameter on the dispersed phase hold-up, the Sauter mean diameter and agitation levels on the extent of mass transfer using the circular downcomer. All experiments were repeated to ensure repeatability of results.

4.2.1 Effect of tray downcomer type on the dispersed phase holdup

Comparison of the dispersed phase holdup between the tray with the circular downcomers and the tray with straight segment downcomers is illustrated in Figure 4.8.

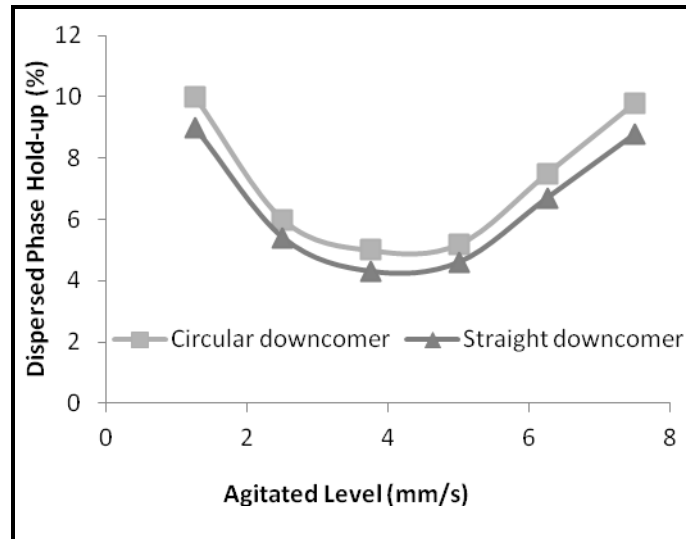


Figure 4.8: Holdup for different downcomer designs

Similar trends are seen for both tray designs. It also shows two distinct hydrodynamic flow regimes. The first flow regime is the mixer-settler regime where initially a high dispersed phase holdup was observed and as the agitation level increases the dispersed phase holdup decreased. This was attributed to the accumulation of the toluene layer under each tray in the column and as the agitation level increases this layer decreases, which subsequently corresponds to a decrease in the holdup.

The smallest dispersed phase holdup, which shows the transition from the mixer-settler flow regime to the dispersion regime, was observed at 3.375 mm/s for both tray designs. A further increase in the agitation level after the minimum holdup resulted in an increase in the dispersed phase holdup.

The increase in the agitation level caused the plates to vibrate faster, which resulted in turbulence and the formation of smaller droplets. The drag force acting on the droplets increased in relation to the buoyancy therefore the velocity of the drops decreased resulting in a higher residence time which causes a higher holdup (Carmurdan, 1986, and Taylor *et al.*, 1982).

Figure 4.8 shows a higher dispersed phase holdup when the tray with circular downcomers is used and low dispersed phase holdup when the tray with straight segment downcomers is used. This was attributed to more channelling that was observed when the tray with the

straight segment downcomers was used which resulted in the reduction of residence time of the droplets in the column.

4.2.2 Effect of tray downcomer type on drop size distribution

The drop size distribution results were used to determine the Sauter mean diameter. The Sauter mean drop diameter was established for each tray downcomer types at a S/F ratio of 1:1 as shown in table 4.2.2 in Appendix B1.2. The effect of tray downcomer types on the Sauter mean diameter is shown in Figure 4.9 (refer to Appendix B1.3 for detailed calculations of the Sauter mean diameter).

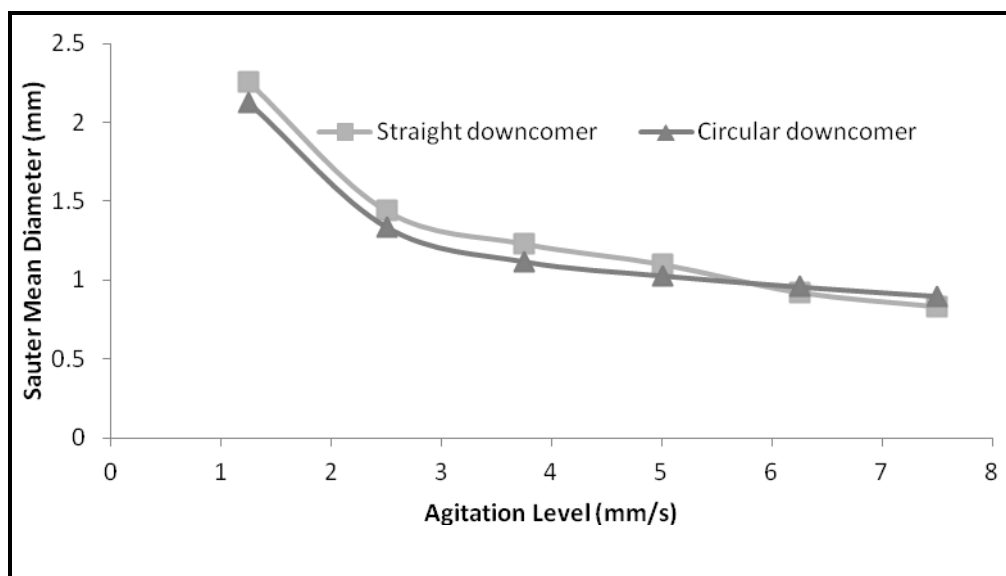


Figure 4.9: Effect of different tray downcomer type a Sauter mean diameter

Figure 4.9 shows no significant difference between the two designs. This means that the Sauter mean diameter is unaffected by downcomer type and may be mainly affected by the hole diameter. The sieve hole diameters in both trays were the same (3 mm).

The droplets are formed through the sieve tray hole diameter. It is evident that there is a decrease in the Sauter mean diameter as the agitation level increases.

4.2.3. Effect of tray design on the amount of acetone extracted

The percentage of acetone extracted was estimated from the feed and raffinate mass percentage. The percentage of acetone extracted was established for each tray design at the

S/F ratio of 1:1. The effect of tray downcomer types on the amount of acetone extracted is shown in Figure 4.10.

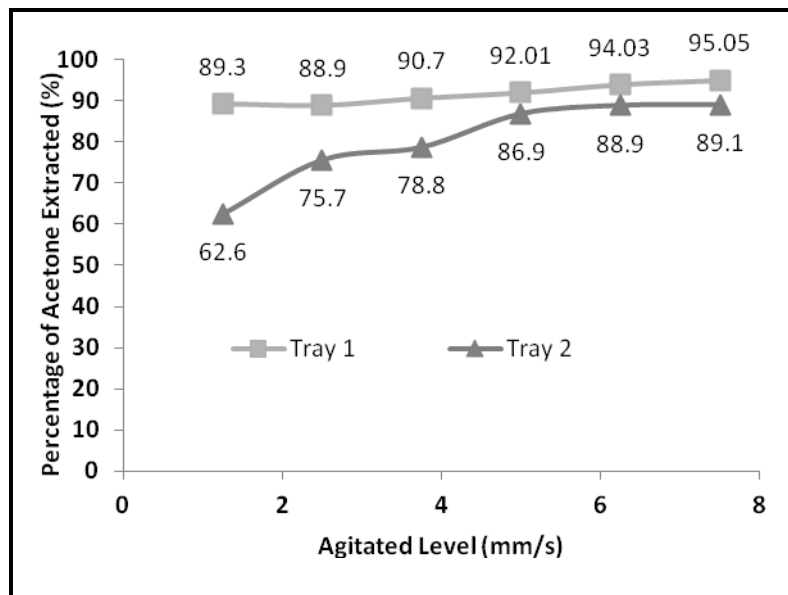


Figure 4.10: Effect of tray downcomer type on percentage acetone extracted

The interfacial area available for mass transfer plays an essential role in the extraction effectiveness of a vibrating plate extraction column. Aravamudan and Baird (1999) showed that the interfacial area is affected by the dispersed phase holdup.

Figure 4.10 shows the effect of tray downcomer types on the amount of acetone extracted which shows an increase in the amount of acetone extracted as the agitation level increases.

Figure 4.10 also shows a significant increase in the amount of acetone extracted when the tray with circular down comers is used. This effect may be the result of the mixing and the flow pattern of the circular downcomers and more channelling as observed when the tray with a straight segment downcomer was used causing the droplets to spend less time in the column.

From Figure 4.8 it was observed that the tray with circular downcomers have higher dispersed phase holdup which mean larger interfacial area available for mass transfer. This corresponds to the large amount of acetone extracted.

4.2.4. Effect of tray design (in terms of hole diameter) on the dispersed phase holdup

The effect of three sieve tray hole diameters was investigated at S/F ratio of 1:1. The circular downcomer design was chosen and sieve trays with three different hole diameters (4.5, 3.0 and 1.5 mm) were designed. The observations were compared to determine if the dispersed phase holdup is affected by the sieve tray hole diameter. The comparison of the dispersed phase holdup between the sieve tray hole diameters is displayed in Figure 4.11.

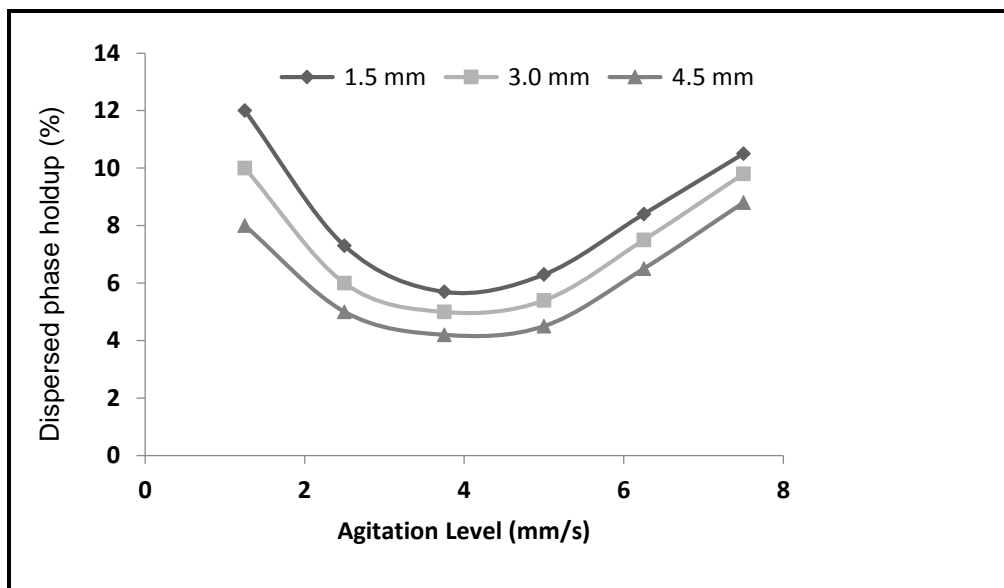


Figure 4.11: Effect of hole diameter on holdup

All tray types produced similar trends. Shown in Figure 4.11 are two distinct hydrodynamic flow regimes. Figure 4.11 also shows high values of dispersed phase holdup when the smallest sieve tray hole diameter is used. This is due to the fact that, as the hole diameter decreases smaller droplets are formed and more holes are available to form many small droplets. As the sieve tray hole diameter decreases the droplets become smaller and the smaller the droplets the longer the residence time and the higher the holdup.

4.2.5. Effect of tray design (in terms of hole diameter) on the drop size distribution

The drop size distribution was estimated for three hole diameters at various agitation levels. Three to five photographs for each run were taken and these photos were analysed using the Image Pro Plus software to determine the droplet sizes. Shown in Figures 4.12 are the comparison of the drop size distribution for hole diameters of 4.5, 3.0 and 1.5 mm respectively at different agitation levels.

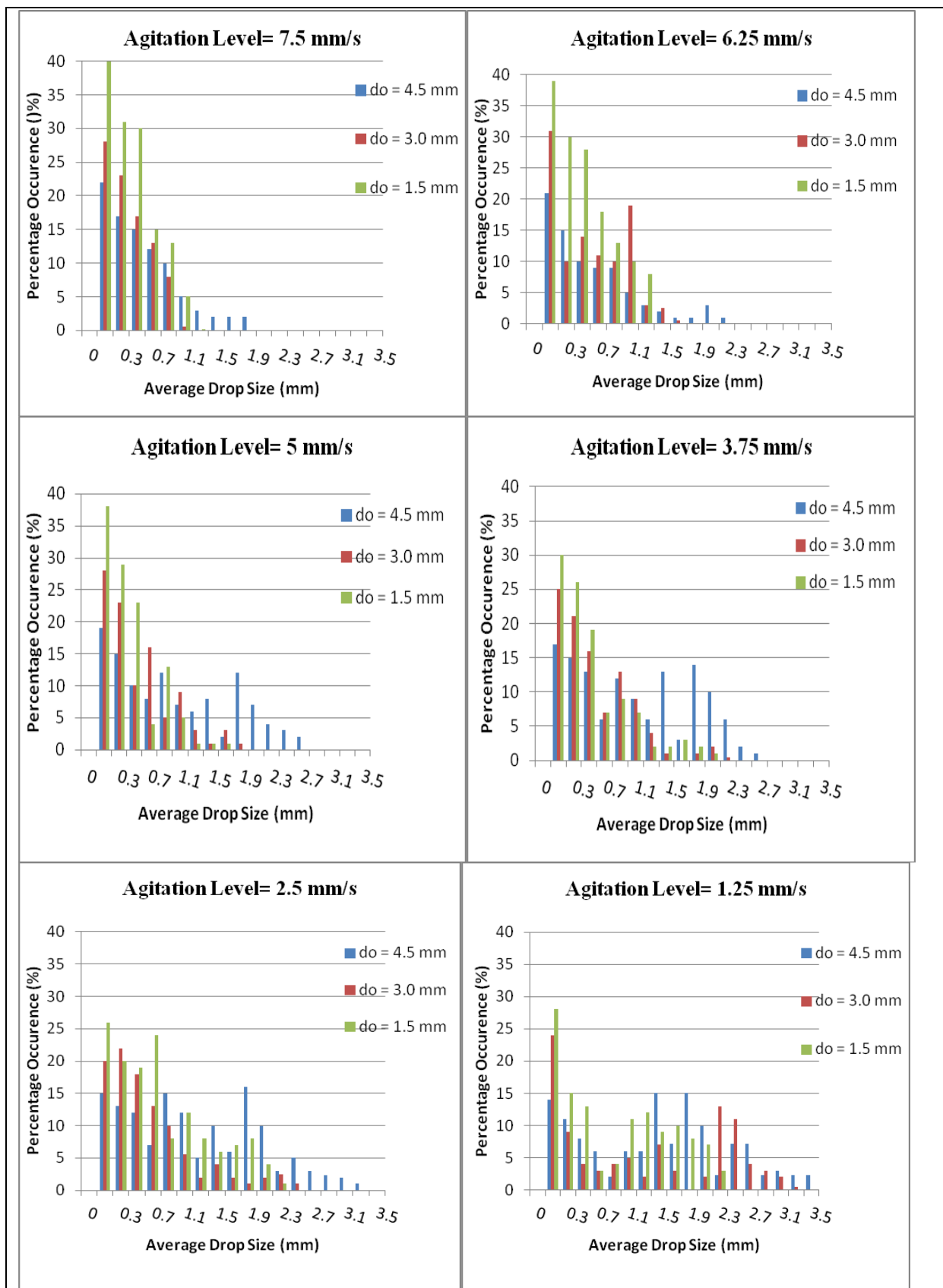


Figure 4.12: dsd graphs at different agitation levels for mass transfer experiments ($d_o = 4.5, 3.0$ and 1.5 mm), circular downcomers

In Figures 4.12 a multimodal drop size distribution is observed at a low agitation levels corresponding to mixer-settler region. With an increase in agitation rate the distribution turns to anti-modal as the system changes from the mixer-settler to dispersion regime. An increase in agitation level narrows the drop size distribution. The tray geometry affects the drop size distribution. As the tray hole size diameter decreases from 4.5mm to 1.5mm more occurrence of smaller was observed.

4.2.6. Effect of tray design (in terms of hole diameter) on Sauter mean diameter

The Sauter mean diameter was calculated from the drop size distribution. This was done in order to find the sieve hole diameter that can produce the smallest droplets with a large interfacial area.

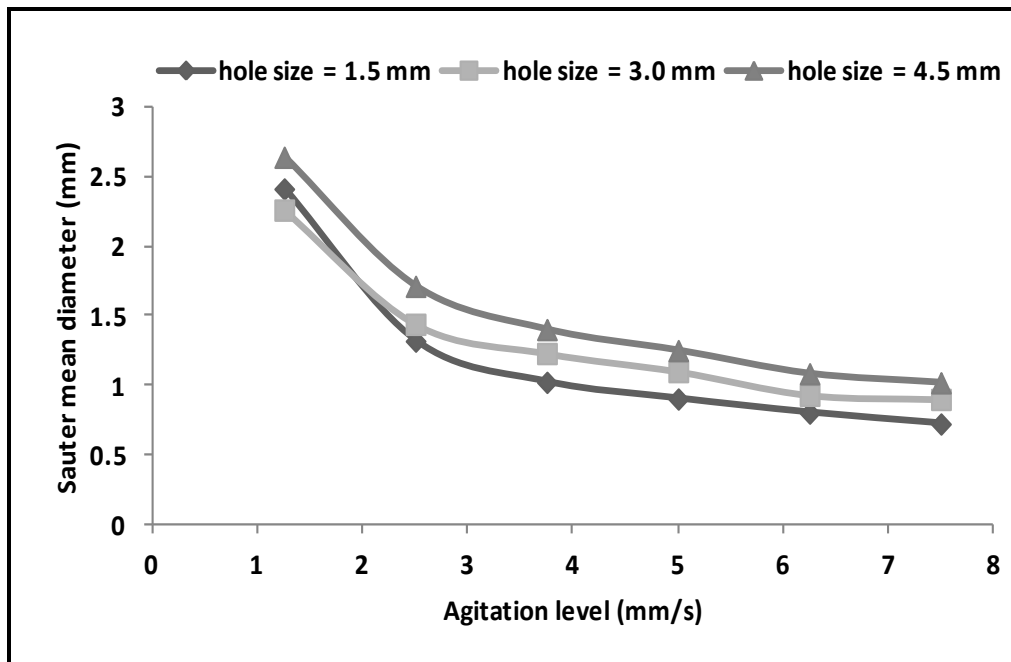


Figure 4.13: Effect of hole diameter on Sauter mean diameter

Figure 4.13 also shows lower values of Sauter mean diameters when the smaller hole diameter is used. This means that the Sauter mean diameter is affected by the sieve hole diameter and the droplets are developed through the sieve hole diameter.

4.2.7. Effect of tray design (in terms of hole diameter) on the extent of mass transfer

The amount of the acetone extracted was determined using the difference of acetone concentration between raffinate and feed and divided by the acetone concentration of the feed and expressed in the mass percentage form.

$$\text{Percentage amount of acetone extracted} = \frac{x_f - x_r}{x_f} \times 100 \quad .$$

Where x_f and x_r are the mass concentrations of acetone in the feed and raffinate respectively

The concentration of acetone was determined by analysing the raffinate and feed samples using the gas chromatograph. The effect of mass transfer on the extent of mass transfer was determined by plotting the amount of acetone extracted versus the agitation level which is shown in Figure 4.14.

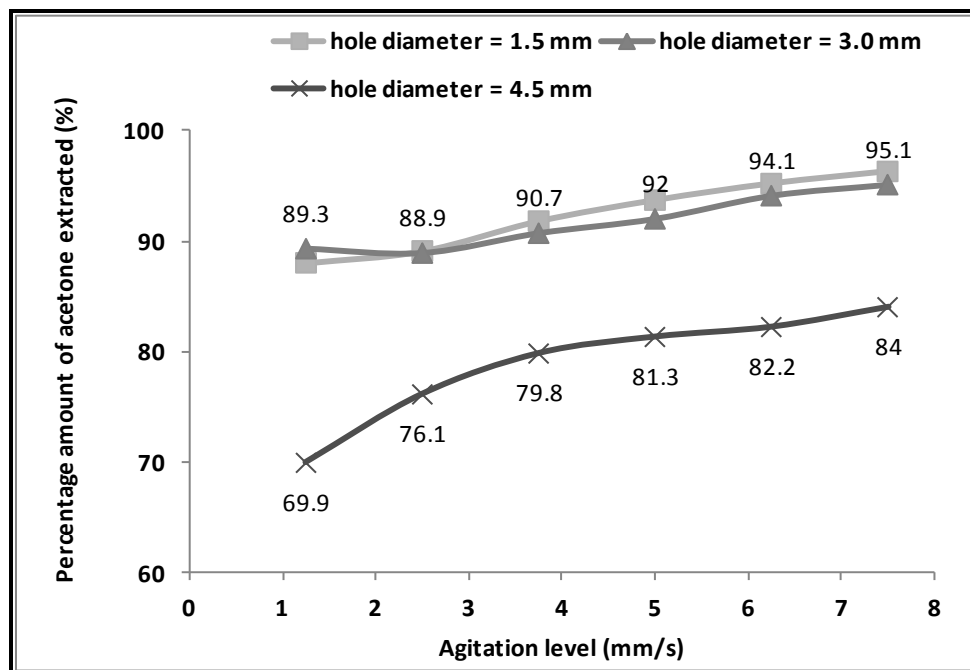


Figure 4.14: Effect of hole diameter on the amount of acetone extracted

Figure 4.14 shows an increase in the amount of acetone extracted as the agitation level increases and shows a significant decrease in the amount of acetone extracted when the sieve tray hole diameter of 4.5 mm is used. Figure 4.14 also shows no significant increase of acetone extracted when the sieve tray hole diameter of 1.5 mm was used even though the dispersed phased holdup was high and the Sauter mean diameter was small.

To drill the sieve tray hole diameter of 1.5 mm is not easy because many holes are needed which makes it more expensive.

The sieve tray hole diameter of 3.0 mm has marginally lower values of acetone extracted but it is easy to drill 3.0 mm and less holes are required. Moreover, the 3.0 mm hole diameter sieve tray is cheaper compared to 1.5 mm hole diameter trays. In conclusion sieve tray hole diameter of 3.0 mm is recommended.

4.2.8. Mass transfer coefficient results

The mass transfer coefficient was calculated using Equation 2.24, where N_{ox} was used as equivalent to NTU (Rathilal *et al.*, 2010). Where N_{ox} for tray spacing of 150 mm was 30, active height of the column (H) was 4.76 m and superficial velocity (U_d) of the dispersed phase was 0.002 m/s.

$$K_{oxm} = \frac{N_{ox} U_d d_{32}}{6\phi H} \quad (2.24)$$

Mass transfer coefficient correlation was estimated using the model that was developed by Rathilal *et al.* (2010) as shown in Equation 2.25, where the tray spacing is in mm, and the agitation level in mm/s. The following model only applies to the dispersion flow regime and emulsion flow regime, due to the fact that the dispersed phase holdup in the mixer settler flow regime includes the coalesced dispersed phase layer accumulated underneath the perforated plates. The prediction was for a hole diameter of 3 mm and for circular downcomers.

$$K_{ox} = -0.007(af) + 0.06 + \frac{h}{10000} \quad (2.25)$$

Table 4.1 illustrates the results for the mass transfer coefficients and also includes the values for the predicted mass transfer coefficients for the different tray downcomers, hole diameters and agitation levels tested. The predicted mass transfer coefficients were estimated using the model developed by Rathilal *et al.* (2010) that relates the mass transfer coefficient to the tray spacing and the agitation level (as a product of amplitude and frequency of vibrations).

Refer to Appendix B1.5 for detailed sample calculations concerning determination of the measured mass transfer coefficient. Comparison could only be done on the agitation levels equal to or greater than 3.75 mm/s due to the application conditions of Equation (4.2), which is only valid in the dispersion and emulsion flow regimes.

Table 4.1: Comparison between measured and predicted mass transfer coefficients

S/F = 1:1 h = 150 mm								
af (mm/s)	Tray 1		Tray 2		Tray 3		Tray 4	
	Circular downcomers		Straight segment		Circular downcomers		Circular downcomers	
	h_d = 3.0 mm		h_d = 3.0 mm		h_d = 1.5 mm		h_d = 4.5 mm	
	k_{oxm}	k_{oxp}	k_{oxm}	k_{oxp}	k_{oxm}	k_{oxp}	k_{oxm}	k_{oxp}
1.25	0.0663	0.0471	0.0663	0.0523	0.0663	0.0445	0.0663	0.0730
2.5	0.0575	0.0494	0.0575	0.0549	0.0575	0.0400	0.0575	0.0757
3.75	0.0488	0.0495	0.0488	0.0576	0.0488	0.0397	0.0488	0.0738
5	0.0400	0.0422	0.0400	0.0455	0.0400	0.0317	0.0400	0.0615
6.25	0.0313	0.0283	0.0313	0.0303	0.0313	0.0215	0.0313	0.0369
7.5	0.0225	0.0203	0.0225	0.0199	0.0225	0.0152	0.0225	0.0028

A fairly close approximation exists between the predicted mass transfer coefficient values and the measured mass transfer coefficient values for the different hole diameter, downcomer types and agitation levels greater than or equal to 3.75 mm/s investigated. Thus the correlation developed by Rathilal, 2010, seems to predict the mass transfer coefficient using the agitation level and the solvent to feed ratio suitably for the data collected, with a maximum error of approximately.

4.2.9. Number of equilibrium stages with and without forward mixing and back mixing

The McCabe Thiele method was used to establish the number of equilibrium stages with and without back mixing and forward mixing. The data for acetone-toluene-water system at equilibrium was used in plotting the equilibrium line and linear relationship was determined with a gradient of 0.832 (Saïen *et al.*, 2006, and Lisa *et al.*, 2003).

The actual operating line was constructed using the acetone concentrations from the gas chromatograph analysis for the raffinate sample, extract sample and samples along the length of the extraction column. The measured number of equilibrium stages without back mixing was determined by stepping off between the equilibrium line and the ideal operating line (straight line) while the actual number of equilibrium stages with back mixing was established by stepping off between the equilibrium line and the actual operating line. Refer to Appendix B1.4 for detailed sample calculation concerning the establishment of the ideal number of equilibrium stages.

The stepping off of the equilibrium stages using the McCabe Thiele method is illustrated in Figure 4.15 for a tray with circular downcomers and a hole diameter of 3 mm at a spacing of 150 mm for lowest agitation level of 1.25 mm/s.

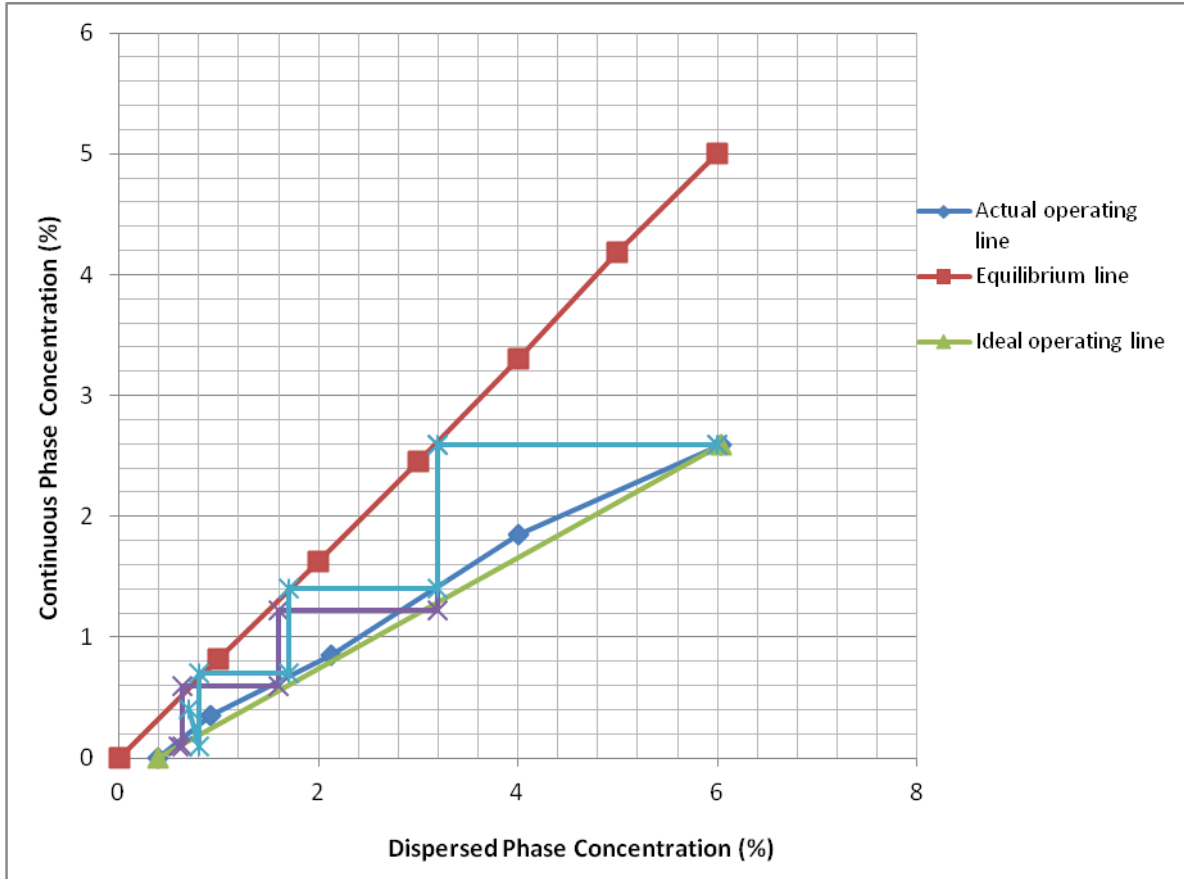


Figure 4.15: Number of stages, for circular downcomers, $d_o = 3.0$ mm and $af = 1.25$ mm/s

Figure 4.15 shows a relatively linear operating line for the lowest agitation level tested, thus showing negligible back mixing occurring in the dispersed phase compared to Figure 4.16 indicating a nonlinear operating line for the highest agitation level tested, showing the occurrence of back mixing and the forward mixing in the dispersed phase. This outcome was also confirmed by Rathilal *et al.* (2010).

Forward mixing occurred when the dispersed phase droplets flow through downcomers fixed on the perforated plates above, thus permitting the droplets to pass a stage and circulate within the next stage (Rathilal *et al.*, 2010). Back mixing in the dispersed phase caused by the decrease in the accumulation of the dispersed phase beneath each perforated plate as the agitation level is increased. Thus, the dispersed phase droplets re-enter the preceding stage it came from.

The stepping off of the equilibrium stages using the McCabe Thiele method is illustrated in Figure 4.16 below that is for a tray with a circular downcomers and hole diameter of 3 mm for the tray spacing of 150 mm at a solvent/feed ratio of 1:1, for the highest agitation level of 7.5 mm/s tested

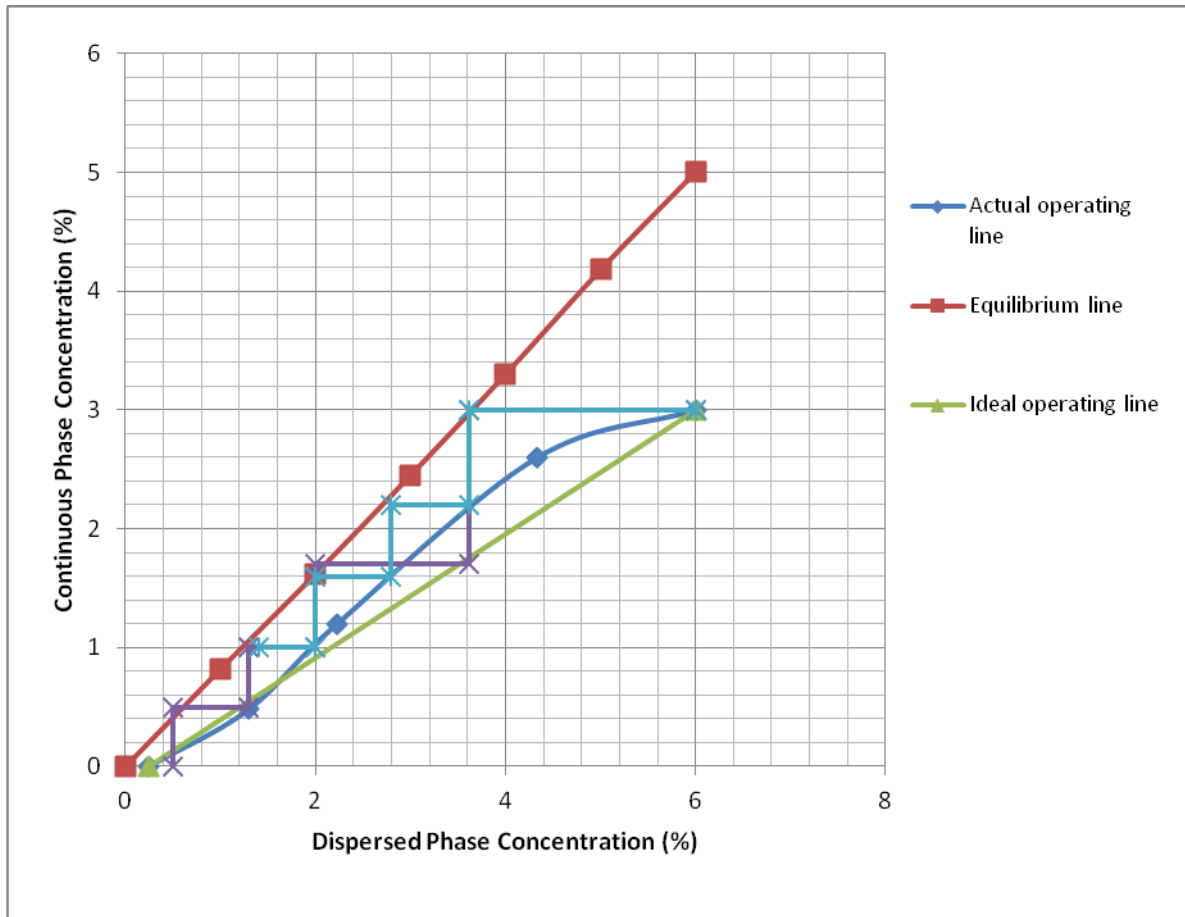


Figure 4.16: Number of stages, for circular downcomers, $d_o = 3.0$ mm and $af = 7.5$ mm/s

From Figure 4.15 and 4.16 it can be seen that the back mixing in the dispersed phase increases as the agitation level increases, leading to the achievement of the higher number of equilibrium stages without back mixing, as illustrated in Table 4.2.

Appendix E1 illustrates the stepping off process to find the number stages using the McCabe Thiele method for other plate designs.

Table 4.2 below lists the number of equilibrium stages with and without back mixing achieved from stepping off using McCabe Thiele technique, for various plate downcomer types, hole diameters and agitation levels tested. The results illustrate that the values of actual and ideal number of stages without back mixing is higher than the number of stages with back mixing.

Table 4.2: Number of equilibrium stages with and without backmixing

S/F = 1:1 h = 150 mm									
Tray 1			Tray 2		Tray 3		Tray 4		
Circular downcomers			Straight segment		Circular downcomers		Circular downcomers		
$d_o = 3.0$ mm			$d_o = 3.0$ mm		$d_o = 1.5$ mm		$d_o = 4.5$ mm		
af (mm/s)	Actual N_{ox}	Ideal N_o	Actual N_{ox}	Ideal N_{ox}	Actual N_{ox}	Ideal N_{ox}	Actual N_{ox}	Ideal N_{ox}	Ideal N_{ox}
1.25	4	4	3	3	3	3	3	3	
2.5	4	4	3	3	3	4	3	4	
3.75	4	4	3	3	4	4	4	4	
5	4	4	3	4	4	4	4	4	
6.25	6	6	3	4	4	4	4	4	
7.5	6	6	4	4	5	5	5	5	

The agitation level affects the rate of back mixing in the dispersed phase resulting in the decline in the accumulation of the dispersed phase under each perforated plate as the agitation level is increased. Similar results were reported by Rathilal *et al.* (2010). During lower agitation levels, the dispersed phase layer accumulated under the perforated plate, works as a barrier stopping the dispersed phase droplets from returning to the previous stage it came from. In this manner back mixing is prevented from happening.

The hole diameters investigated also have an effect on the back mixing in the dispersed phase taking place. For the smallest hole diameter of 1.5 mm it was seen that negligible back mixing occurred as there was constantly the occurrence of a dispersed phase layer accumulated under each perforated plate, consequently not permitting the dispersed phase droplets from re-entering into the previous stage. For the larger hole diameter of 4.5 mm a considerable amount of back mixing took place as a result of larger dispersed phase droplets present to re-circulating within the stages and the re-entering of the dispersed phase droplets into the previous stage.

Table 4.3 indicates the results obtained from comparison between the predicted number of stages using the model established by Rathilal *et al.* (2010) and the measured number of equilibrium stages (with back mixing) from stepping off. The model established to predict the number of equilibrium stages for the vibrating plate extraction column. Refer to Equation 2.21.

Table 4. 3: Comparison between measured and predicted number of equilibrium stages

$S/F = 1:1$								
$af(\text{mm/s})$	Tray 1 Circular downcomers $d_o = 3.0 \text{ mm}$		Tray 2 Straight segment $d_o = 3.0 \text{ mm}$		Tray 3 Circular downcomers $d_o = 1.5 \text{ mm}$		Tray 4 Circular downcomers $d_o = 4.5 \text{ mm}$	
	Noxm	Noxp	Noxm	Noxp	Noxm	Noxp	Noxm	Noxp
1.25	-	4	-	3	-	3	-	3
2.5	-	4	-	3	-	3	-	3
3.75	5	4	5	3	5	4	5	4
5	6	4	6	3	6	4	6	4
6.25	8	6	8	3	8	4	8	4
7.5	10	6	10	4	10	5	10	5

A close estimation exists for hole diameter of 3.0 mm at each plate downcomer type investigated between predicted number of stages and the measured number of equilibrium stages, as compared to the other hole diameters investigated. Therefore indicating that the correlation developed by Rathilal *et al.* (2010), predicts the number of stages quite well for all the plate downcomer types at the hole diameter of 3.0 mm investigated, for both plate with circular downcomers and with straight downcomers as well as the agitation level between 1.25 and 5 mm/s.

CHAPTER 5: CONCLUSION

The main objective of this research was to investigate the effect of different tray designs in order to enhance separation process efficiency for the vibrating plate extraction column. All tests were conducted using the toluene-acetone-water system. The experiments were conducted to test the effect of mass transfer on the hydrodynamics of the system while changing the agitation levels, downcomers and hole diameters. The total throughput for the S/F ratio of 1:1 for all the experiments was kept constant at 30 l/h.

The dispersed phase hold-up, Sauter mean diameter and drop size distribution was determined for each tray design (tray 1 with circular down-comers and tray 2 with straight segment downcomers) for various agitation levels in order to select the more effective downcomer. Then the selected downcomer was used to investigate the optimum hole diameter. Three hole diameters (4.5, 3.0, and 1.5 mm) were investigated.

It was noted that initially in the mixer-settler regime a high dispersed phase holdup was observed. The dispersed phase holdup decreased as the agitation level increases. This was attributed to the accumulation of the toluene layer under each tray in the column and as the agitation level increases this layer decreased, which subsequently corresponds to a decrease in the holdup.

Comparison of the effect of the tray downcomer types on the amount of acetone extracted was utilised to select the optimum downcomer. The circular downcomer was the more effective compared to the straight segment downcomer, since it had a higher dispersed phase holdup and the highest percentage amount of acetone extracted (95.05%). Circular downcomers have larger interfacial area available for mass transfer and high dispersed phase holdup hence the larger amount of acetone was extracted.

The holdup during mass transfer and in the absence of mass transfer decreased initially as the agitation level was increased until it reached a minimum at an agitation level of 3.75 mm/s. The holdup decreased as the hole diameter increased and since it was found that there was a strong relationship between hole diameter and the holdup, it was concluded that the holdup increased with a decrease in the hole diameter.

The holdup during mass transfer was lower than that in the absence of mass transfer due to the solute being continuously removed from the dispersed phase resulting in there being lesser dispersed phase and a lower holdup during mass transfer.

From the differences in the measurements during hydrodynamics and mass transfer it was concluded that the measurements during hydrodynamics cannot be used for the prediction of the performance of the vibrating plate extractor since the process of mass transfer affects the hydrodynamics of the column.

Drop size analysis showed that there was a multi modal distribution of sizes at low agitation levels which became narrower as the agitation level increased. Due to enhanced coalescence effects during mass transfer there were a few large drops that were observed even at high agitation levels. A much higher Sauter mean diameter for all the agitation rates is achieved as the tray hole diameter is increased, since the distribution is wider. This is due to the breakup of the drops and reduced coalescence which occur mainly in the locality of the plates.

The correlation developed by Rathilal et al., 2010 seems to predict the mass transfer coefficients for the experimental data correctly especially for the dispersion regime. Thus the predicted mass transfer coefficient values are appropriately close to the measured values.

The occurrence of backmixing in the dispersed phase is proportional to the agitation level. Thus at low agitation level there is negligible backmixing and at high agitation level there is significant occurrence of back and forward mixing. The McCabe Thiele method shows that a higher number of equilibrium stages without backmixing was obtained as compared to the number of equilibrium stages with backmixing.

The tray hole diameters were also found to have an effect on the backmixing in the dispersed phase, with a decrease in the hole diameter causing an increase in the backmixing in the dispersed phase, which resulted in an increase in mass transfer.

The 3.0 mm tray hole diameter was the most favourable with regard to mass transfer efficiency compared to 1.5 and 4.5 mm. There was insignificant difference in the amount of acetone extracted between 1.5 and 3.0 mm tray hole diameter ($<1\%$), whilst 4.5 mm had the

least amount acetone extracted (84%) compared to 95.1% and 94.6% of 3mm and 1.5mm respectively. The 3.0 mm hole diameter is easier and cheaper to manufacture compared to 1.5 mm hole diameter, thus the 3.0 mm diameter is recommended.

After a thorough investigation of different tray designs the tray design with circular downcomers and hole diameter of 3 mm was selected as an effective tray design that can be used in the vibrating plate extraction column.

CHAPTER 6: RECOMMENDATIONS

- An installation of the flow controller in the feed is recommended because of fluctuations encountered.
- Experimental work need to be done on the emulsion flow regime concerning interfacial area of mass transfer.
- This research was conducted varying only the frequency of vibration and keeping the amplitude of the vibration constant, so other experiments should be done to investigate the effect of changing the amplitude of vibration.

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APPENDICES

APPENDIX A: EQUIPMENT CALIBRATION GRAPHS

Appendix A1.1: Vibration motor calibration graph

The perforated sieve plates inside the vibrating plate extraction column are connected to a central shaft, driven by a variable speed motor that is attached to a yoke at the top of the extraction column. The calibration of the motor carried out in order to establish the relation between the variable speeds motor's controller readings and the frequency. Figure A1 illustrates the relation between the motor controller's settings and the vibration motor speed in units of number of revolutions per minute.

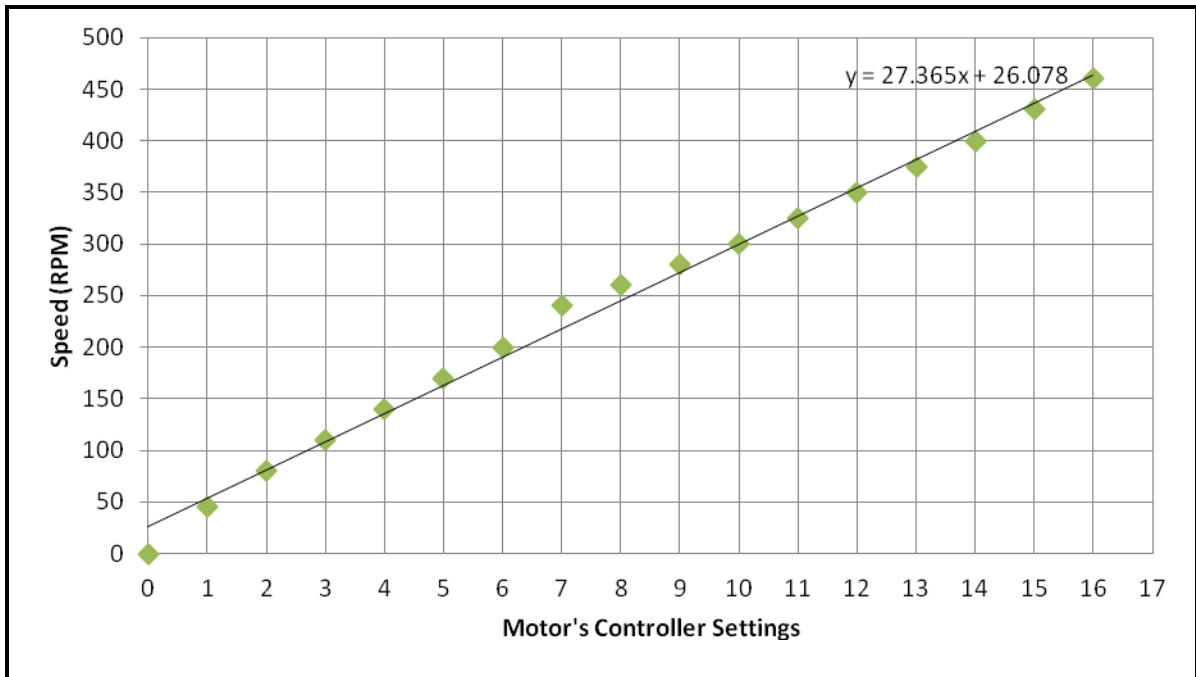


Figure A1.1: Graph of speed (RPM) versus motor's controller settings

Appendix A1.2: GC calibration for acetone in water

The standard solution was prepared with the precise amounts of acetone in water. Then these samples were tested in the GC to achieve the standard areas of each constituent. The calibration curve was plotted using these standard areas and the precise amounts of acetone in water. Since Flame ionization detector (FID) is the instrument used for detecting the presence of hydrocarbon only one peak (acetone) was obtained from GC. A calibration equation that was achieved from the chart was used to calculate the concentrations of the unknown samples of the extract phase.

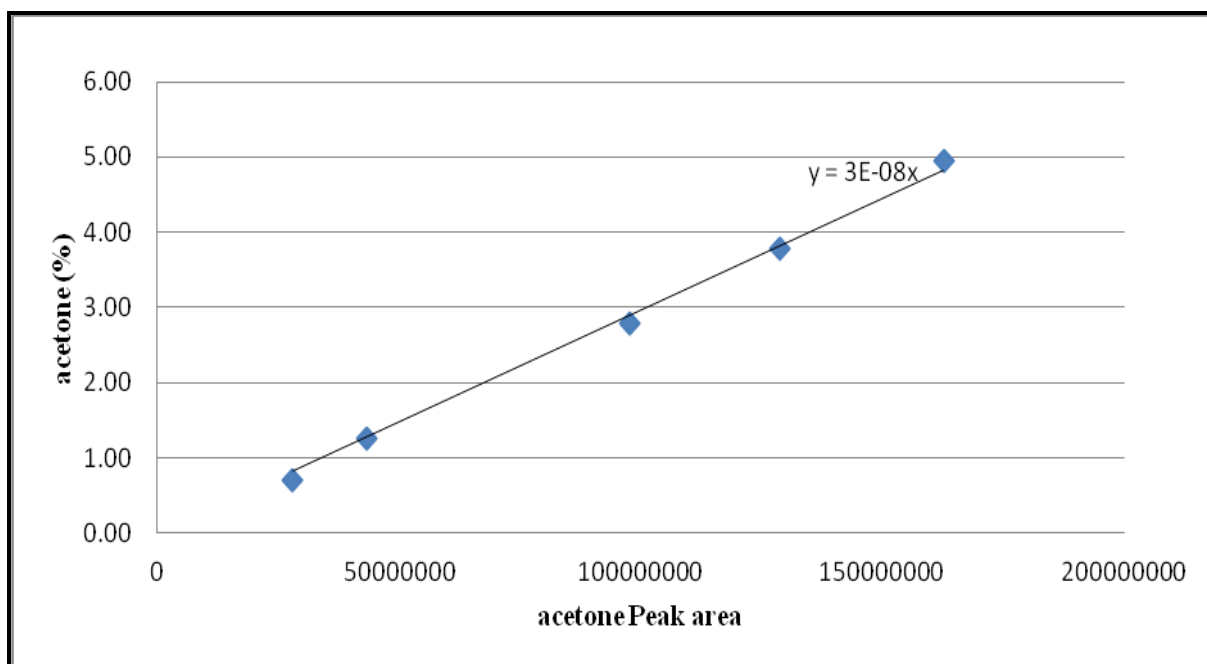


Figure A1.2: GC calibration curve for acetone amount in water

Appendix A1.3: GC calibration for acetone in toluene

The standard solution was prepared with the precise amounts of acetone in toluene. Then these samples were tested in the GC to achieve the standard areas of each constituent. The calibration curve was plotted using these standard areas.

Two peaks were obtained, one for acetone and one for toluene. Here the peak area ratio of acetone to toluene was expressed as % and was plotted against the precise amounts of acetone in toluene. A calibration equation that was achieved from the chart was used to calculate the concentrations of the unknown samples of the raffinate phase.

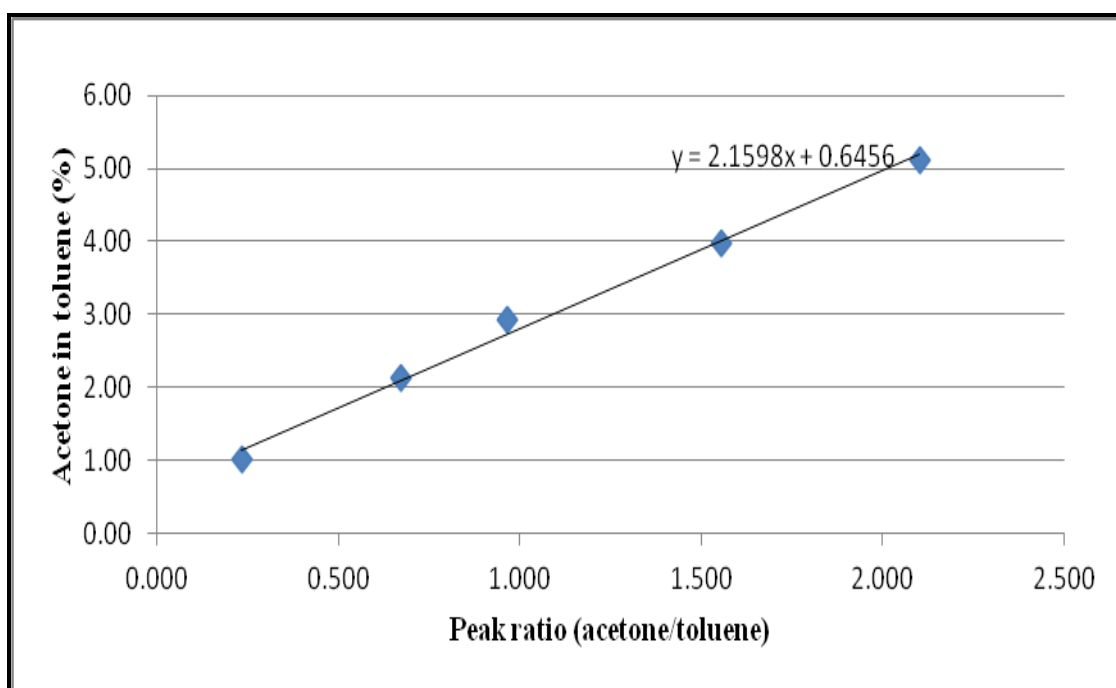


Figure A 1.3: GC calibration curve for acetone amount in toluene

APPENDIX B: SAMPLE CALCULATIONS

The following Sample Calculations were performed for Run 1 of S/F=1:1 at h=150 mm

Appendix B1.1: Dispersed phase hold-up

The active volume of the column and the dispersed phase volume were used to calculate the dispersed phase hold-up percentage. The dispersed phase hold-up (height of the raffinate below the interface level) was used to determine the volume of the raffinate as follows:

Table B1. 1: Data used for calculations

Cross-Sectional area of top settling tank (m ²) (A)	0.01767
Active Volume at a tray spacing of 150 mm (mm ³)	7.9

Volume of raffinate = Height of raffinate below interface level * Cross Sectional area of top settling tank

$$= (54 \text{ mm} / 1000) * 0.01767 \text{ m}^2$$

$$= \underline{0.0009542 \text{ m}^3}$$

The dispersed phase hold up was calculated and illustrated as a percentage as follows.

Dispersed Phase Hold-up = (Volume of Raffinate / Active Volume) * 100

$$= (0.0009542 / 7.9 * 10^{-3}) * 100$$

$$= \underline{12.08 \%}$$

Appendix B1.2: Drop size distribution

The drop size and number of droplets were calculated using the Image Pro plus software. Table B1.2 below represents the number of droplets for different size ranges.

Table B1.2: Drop size distribution

size range	average drop size (mm)	no of drop	Fraction of drop size (%)	nd ² (mm ²)	nd ³ (mm ³)
0 - 0.2	0.1	0	0		
0.2 - 0.4	0.3	5	13.5	0.45	0.135
0.4 - 0.6	0.5	15	40.5	3.75	1.875
0.6 - 0.8	0.7	6	16.2	2.94	2.058
0.8 – 1	0.9	5	13.5	4.05	3.645
1 - 1.2	1.1	2	5.4	2.42	2.662
1.2 - 1.4	1.3	4	10.8	6.76	8.788
Total		37	100	20.37	19.163

The fraction of drops in a size range is represented as the percentage occurrence therefore the drop size distribution results are displayed by plotting the percentage occurrence versus agitation level. The following calculation is a sample for the size range 0.2-0.4

$$\begin{aligned}\text{Fraction of Drops} &= (\text{Number of drops per size range} / \text{Total number of drops}) * 100 \\ &= (5/37) * 100 \\ &= 13.51 \%\end{aligned}$$

Appendix B1.3: Sauter mean diameter

The drop size distribution was used to calculate the Sauter mean diameter. The following calculation was performed for a size range of 0.2-0.4 mm, using the data contained in Table B1.2:

$$\begin{aligned}\text{Nd}^2 &= (5) (0.3)^2 \\ &= 0.45 \text{ mm}^2\end{aligned}$$

$$Nd^3 = (5) (0.3)^3$$

$$= 0.135 \text{ mm}^3$$

$$\Sigma n d_i^2 = 0.45 + 3.75 + 2.94 + 4.05 + 2.42 + 6.76$$

$$= 20.37 \text{ mm}^2$$

$$\Sigma n d_i^3 = 0.135 + 1.875 + 2.058 + 3.645 + 2.662 + 8.788$$

$$= 19.163 \text{ mm}^3$$

The Sauter Mean Diameter, d_{32} , was calculated using the following equation:

$$d_{32} = \frac{\sum_{i=1}^N n_i d_i^3}{\sum_{i=1}^N n_i d_i^2}$$

$$d_{32} = \frac{19.163 \text{ mm}^3}{20.37 \text{ mm}^2}$$

$$d_{32} = 0.941 \text{ mm}$$

Appendix B1.4: Number of equilibrium stages without back-mixing

The sample calculation shows how the ideal number of equilibrium stages was calculated for mass transfer experiments conducted at $h = 150 \text{ mm}$, $S/F = 1:1$ and Agitation level = 1.25 mm/s , the slope of the equilibrium line, m , was 0.832 .

$$\begin{aligned} \text{Data:} \quad y_i &= 0 & y_o &= x_{\text{extract}} = 0.0259 \\ x_i &= x_{\text{feed}} = 0.0604 & x_o &= x_{\text{raffinate}} = 0.0069 \end{aligned}$$

$$N_{\text{exp}} = \frac{1}{\left[\frac{(y_1 - y_o)}{m(x_o - x_i)} \right] - 1} \ln \left(\frac{y_1 - mx_o}{y_o - mx_i} \right)$$

$$N_{\text{exp}} = \frac{1}{\left[\frac{(0 - 0.0259)}{0.832(0.0069 - 0.0604)} \right] - 1} \ln \left(\frac{0 - 0.832 * 0.0069}{0.0259 - 0.832 * 0.0604} \right) \setminus$$

$$N_{\text{exp}} = 1.47$$

Appendix B1.5: Measured mass transfer coefficient

The following sample calculation indicates how the mass transfer coefficient was established using the following Equation below that combines Equation 17 and $N_{ox} = \frac{k_{ox} a H}{U_d}$, (Aravamudan and Baird, 1999). The mass transfer experiments' results for h= 150 mm, agitation level of 1.25 mm/s, circular downcomers, and do = 3.0 mm.

$$N_{ox} = 30 \text{ for a tray spacing of 150 mm}$$

$$U_d = 0.0021 \text{ m/s}$$

$$d_{32} = 2.092 \text{ m}$$

$$\phi = 0.1$$

$$H = 4.76 \text{ m}$$

$$\begin{aligned} k_{ox} &= \frac{N_{ox} U_d d_{32}}{6 \phi H} \\ &= \frac{30 \times 0.0021 \times 2.092}{6 \times 0.1 \times 4.76} \\ &= \underline{0.046 \text{ m/s}} \end{aligned}$$

Appendix B1.6: Percentage acetone extracted

The extent of mass transfer was displayed graphically by plotting the percentage acetone extracted versus the agitation level. The percentage extracted sample calculation was calculated using the feed and raffinate concentrations for a tray spacing of 150 mm, S/F =1:1 and Agitation level of 1.25 mm/s

$$\text{Data} \quad X_{\text{feed}} = 6.04 \quad X_{\text{raffinate}} = 0.69$$

$$\text{Percentage Acetone Extracted} = \frac{X_{\text{feed}} - X_{\text{raffinate}}}{X_{\text{feed}}} \times 100$$

$$\text{Percentage Acetone Extracted} = \frac{6.04 - 0.69}{6.04} \times 100$$

$$\text{Percentage Acetone Extracted} = 88.58\%$$

APPENDIX C: HYDRODYNAMIC RESULTS FOR DROP SIZE DISTRIBUTION FOR THREE VARIOUS HOLE DIAMETERS

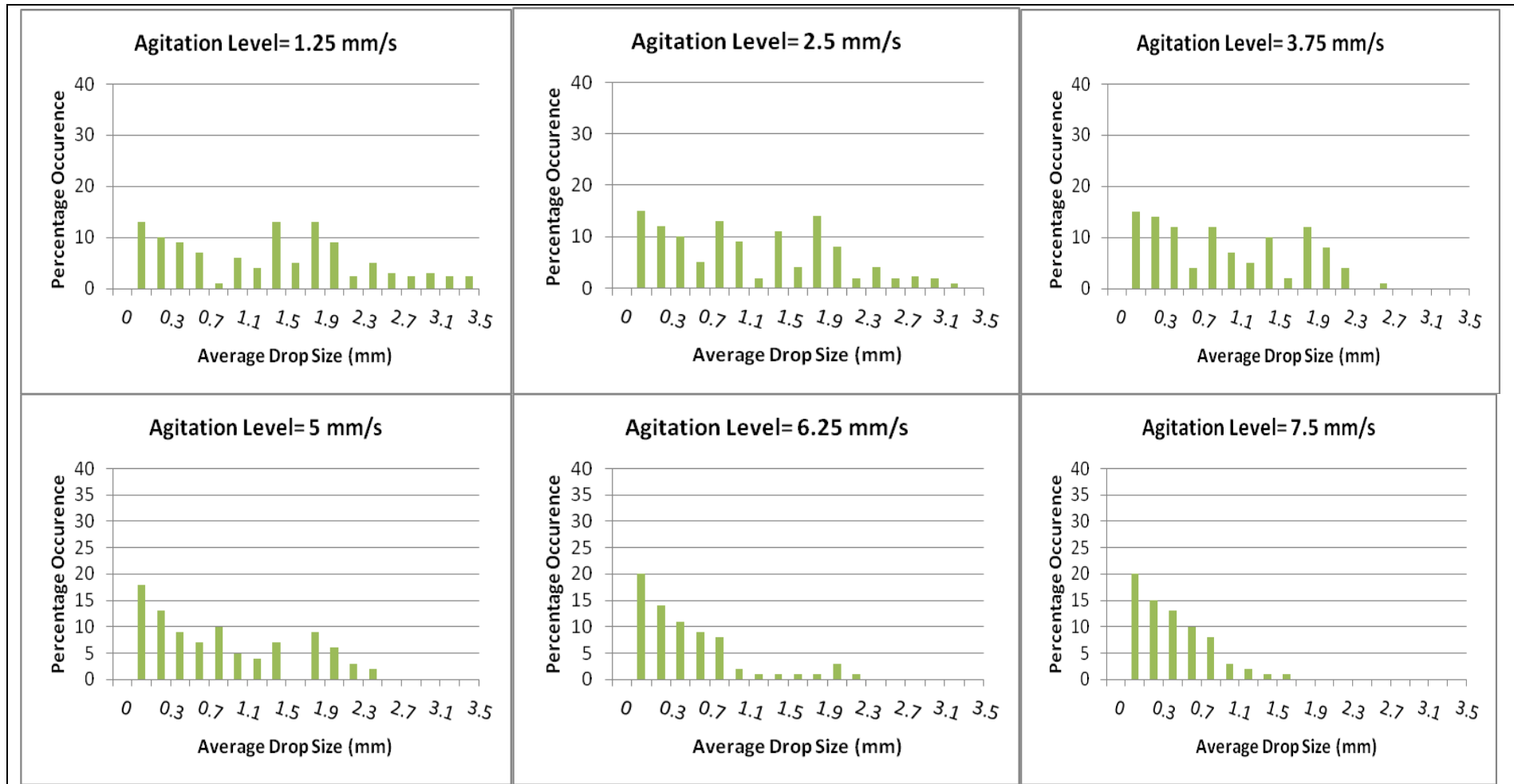


Figure C1.1: Hydrodynamic for dsd at a various agitation level (4.5 mm hole diameter)

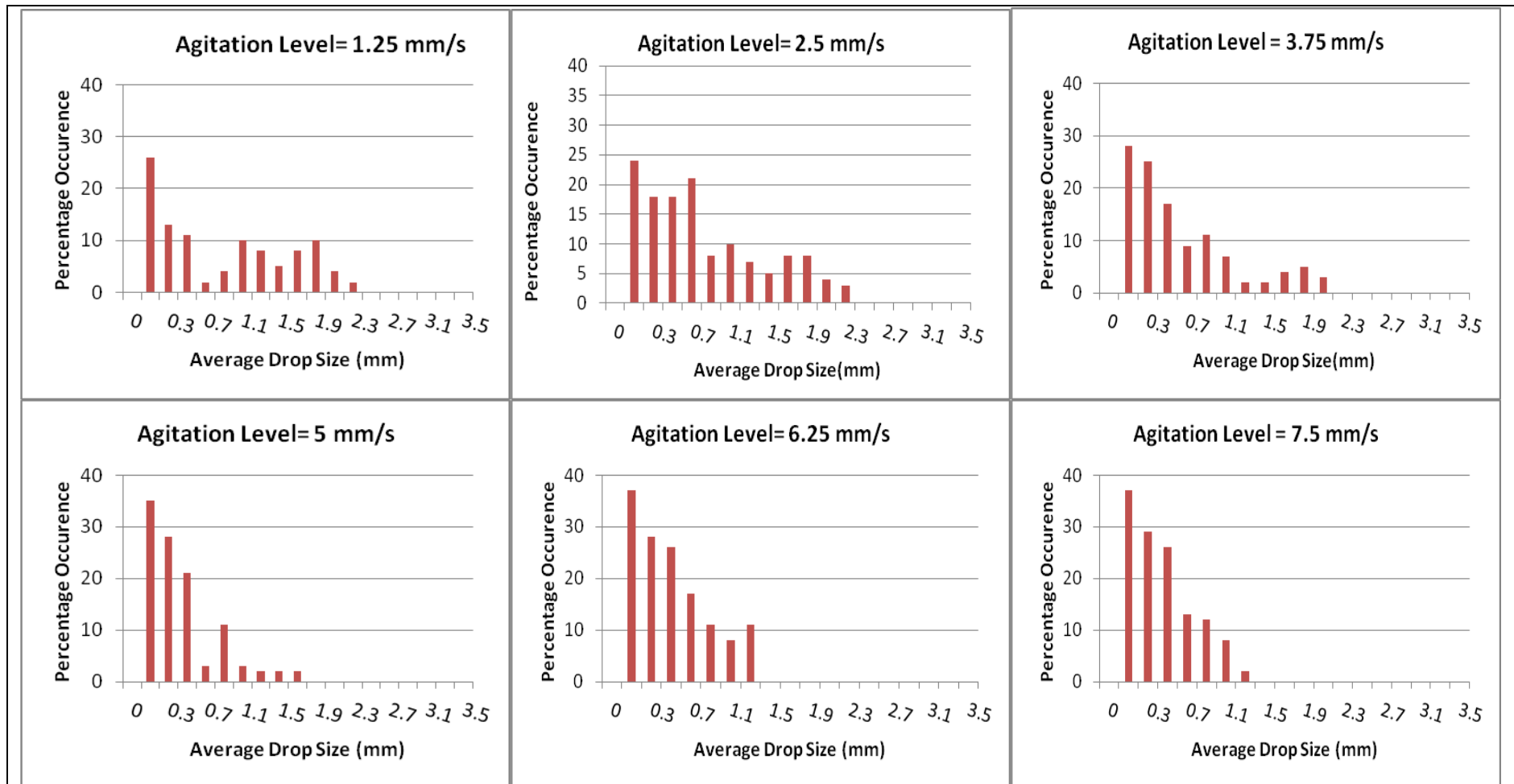


Figure C 1.2: Hydrodynamic for dsd at a various agitation level (1.5 mm hole diameter)

APPENDIX D: RAW DATA

Table D1. 1: Percentage of acetone extracted plate with straight segment, $d_o = 3.0$ mm, $S/F = 1:1$, $H = 150$ mm segment downcomer

Agitation level (mm/s)	Hold-up (mm)	x_f	x_r	x_e	d_1	C_1	d_2	C_2	d_3	C_3	Acetone Extracted (%)
1.25	50	6.00	2.23	2.08	1.71	0.99	1.57	0.57	1.07	0.23	62.3
			2.30	2.03	1.61	1.09	1.45	0.56	0.96	0.19	
			2.26	2.05	1.66	1.04	1.51	0.57	1.02	0.21	
1.25	48	5.98	2.22	2.04	1.66	1.04	1.50	0.57	1.01	0.20	63.0
Average	49										62.6
2.5	26	5.99	1.47	2.96	2.15	1.85	1.41	0.97	1.07	0.40	75.6
			1.45	2.99	2.08	1.78	1.35	0.95	1.00	0.45	
			1.46	2.98	2.12	1.82	1.38	0.96	1.04	0.43	
2.5	28	6.02	1.46	3.00	2.10	1.91	1.36	0.95	1.06	0.43	75.7
Average	27										75.7
3.75	24	5.93	1.24	2.82	1.97	2.07	1.45	1.09	0.87	0.53	79.1
			1.24	2.81	2.19	2.02	1.45	1.12	0.87	0.51	
			1.24	2.82	2.08	2.04	1.45	1.10	0.87	0.52	
3.75	22	5.98	1.28	2.91	2.09	2.04	1.45	1.13	0.90	0.52	78.6
Average	23										78.8
5	33	6.09	0.79	2.93	1.90	1.97	1.26	0.96	0.77	0.37	87.0
			0.79	2.86	1.90	1.99	1.26	0.96	0.81	0.38	
			0.79	2.89	1.90	1.98	1.26	0.96	0.79	0.38	
5	35	5.98	0.79	2.91	1.91	2.01	1.25	0.95	0.79	0.38	86.8
Average	34										86.9
6.25	41	6.04	0.67	3.07	1.89	2.00	0.83	0.52	0.65	0.14	88.9
			0.67	3.06	1.77	1.95	0.80	0.55	0.64	0.12	
			0.67	3.06	1.83	1.98	0.82	0.54	0.64	0.13	
6.25	42	6.00	0.67	3.07	1.88	1.20	0.82	0.54	0.64	0.13	88.8
Average	41.5										88.9
7.5	65	5.98	0.65	3.32	2.00	1.73	0.65	0.24	0.65	0.03	89.1
			0.65	3.29	1.96	1.69	0.65	0.24	0.63	0.02	
			0.65	3.30	1.98	1.71	0.65	0.24	0.64	0.02	
	64	6.01	0.65	3.30	1.96	1.70	0.64	0.23	0.65	0.30	89.1
Average	64.5										89.1

Table D1.2: Percentage of acetone extracted plate with circular downcomers, $d_o = 4.5$ mm, $S/F = 1:1$, $H = 150$ mm

Agitation level	Holdup	x_f	x_r	x_e	d₁	C₁	d₂	C₂	d₃	C₃	Acetone extracted
(mm/s)	(mm)										(%)
1.25	50	6.04	0.36	1.29	3.06	1.02	1.35	0.33	0.61	0.14	
1.25	48	6.01	0.37	1.3	3.08	1.02	1.33	0.34	0.59	0.15	69.9
2.5	26	6.02	0.27	1.3	3.12	0.96	1.50	0.34	0.63	0.15	
2.5	28	6.04	0.29	1.32	3.10	0.95	1.49	0.34	0.64	0.16	76.1
3.75	24	6.03	0.23	1.31	3.38	0.92	1.67	0.46	0.82	0.2	
3.75	23	5.99	0.24	1.30	3.36	0.91	1.65	0.43	0.85	0.22	79.8
5.0	33	6.01	0.19	1.33	3.24	0.92	1.57	0.28	0.44	0.11	
5.0	34	5.97	0.21	1.32	3.26	0.92	1.58	0.24	0.44	0.13	81.3
6.25	41	6.03	0.17	1.35	2.87	0.87	1.27	0.22	0.32	0.09	
6.25	43	6.01	0.19	1.34	2.88	0.86	1.27	0.19	0.33	0.1	81.2
7.5	53	6.05	0.16	1.44	2.71	0.82	1.17	0.13	0.32	0.08	
7.5	55	6.01	0.15	1.45	2.70	0.83	1.16	0.15	0.31	0.08	84.0

Table D1.3: Percentage of acetone extracted plate with circular downcomers, $d_o = 3.0$ mm., $S/F = 1:1$, $H = 150$ mm

Agitation Level	Dispersed Phase Holdup			Percentage Acetone (%)										
	(mm/s)	mm	m ³	(%)	x _f	x _r	x _e	d ₁	c ₁	d ₂	c ₂	d ₃	c ₃	Extracted
1.25	43	0.00076	9.6	6.04	0.65	2.59	3.23	1.95	2.03	1.24	0.86	0.59	89.2	89.1
1.25	41	0.00072	9.2	6.01	0.67	2.62	3.30	2.03	2.09	1.19	0.82	0.56	88.9	
2.5	25	0.00044	5.6	6.02	0.68	2.66	3.35	1.96	2.10	1.25	0.72	0.62	88.7	88.9
2.5	23	0.00041	5.1	6.04	0.66	2.68	3.39	1.94	2.15	1.27	0.75	0.64	89.1	
3.75	20	0.00035	4.5	6.03	0.59	2.71	3.41	2.09	2.16	1.33	0.65	0.66	90.2	90.4
3.75	22	0.00039	4.9	5.99	0.57	2.69	3.40	2.12	2.19	1.37	0.62	0.66	90.5	
5	34	0.0006	7.6	6.01	0.51	2.82	3.52	2.15	2.31	1.38	0.58	0.68	91.5	91.8
5	36	0.00064	8.1	5.97	0.47	2.86	3.56	2.16	2.29	1.40	0.56	0.68	92.1	
6.25	43	0.00076	9.6	6.03	0.36	2.95	3.57	2.19	2.42	1.46	0.42	0.69	94.0	93.9
6.25	45	0.0008	10.1	6.01	0.37	2.91	3.60	2.21	2.46	1.43	0.40	0.69	93.8	
7.5	52	0.00092	11.6	6.05	0.32	3.22	3.68	2.24	2.62	1.53	0.31	0.70	94.7	94.9
7.5	48	0.00085	10.7	6.01	0.29	3.23	3.72	2.26	2.58	1.61	0.32	0.72	95.2	

Table D1.4: Percentage of acetone extracted plate with circular downcomers, $d_o = 1.5$ mm, $S/F = 1:1$, $H = 150$ mm

Agitation level	Holdup	x_f	x_r	x_e	d₁	C₁	d₂	C₂	d₃	C₃	Acetone extracted
(mm/s)	(mm)										(%)
1.25	50	5.98	1.4	2.75	4.17	1.8	2.20	1	1.4	0.34	79.2
1.25	48	6	1.42	2.78	4.19	1.82	2.21	1.01	1.42	0.33	
2.5	26	6.01	0.98	2.98	4.30	1.87	2.27	1.10	0.97	0.39	83.7
2.5	28	5.99	0.96	2.96	4.32	1.85	2.29	1.11	0.98	0.4	
3.75	24	5.99	0.86	3.1	4.43	1.90	2.50	1.04	0.98	0.41	85.8
3.75	23	5.98	0.84	3.0	4.45	1.93	2.48	1.04	0.98	0.41	
5.0	33	6.03	0.79	3.18	4.48	1.98	2.46	0.98	1.28	0.46	86.7
5.0	34	6.01	0.8	3.2	4.45	1.96	2.45	0.97	1.26	0.47	
6.25	41	5.99	0.75	3.2	4.33	1.86	2.23	0.9	1.3	0.48	86.9
6.25	43	6.04	0.74	3.22	4.32	1.85	2.23	0.91	1.33	0.48	
7.5	53	6	0.65	3.26	4.09	1.98	2.03	0.83	0.43	0.15	89.2
7.5	55	6.01	0.66	3.25	4.1	1.99	2.05	0.82	0.42	0.14	

APPENDIX E: STEPPING OFF STAGES

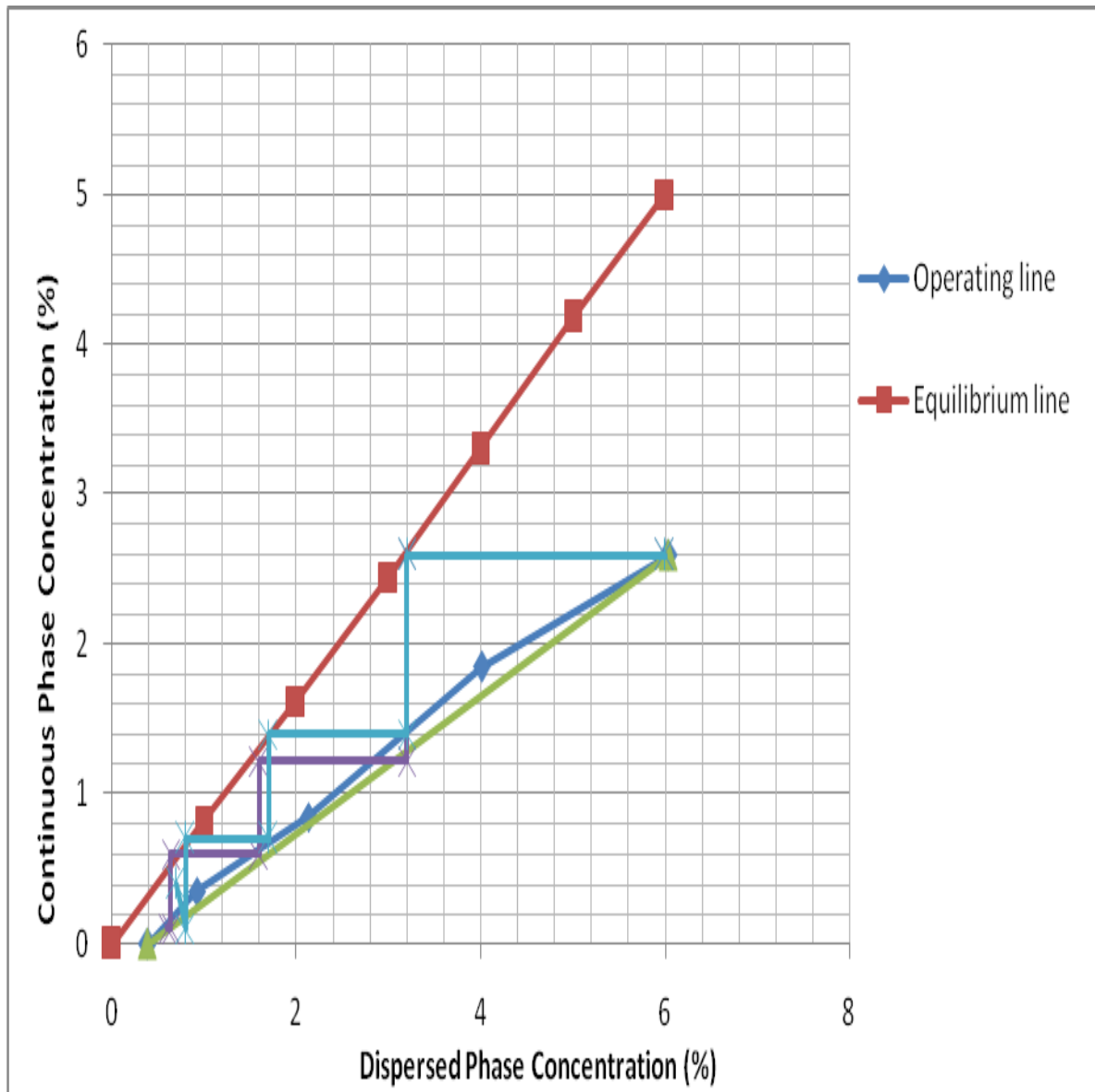
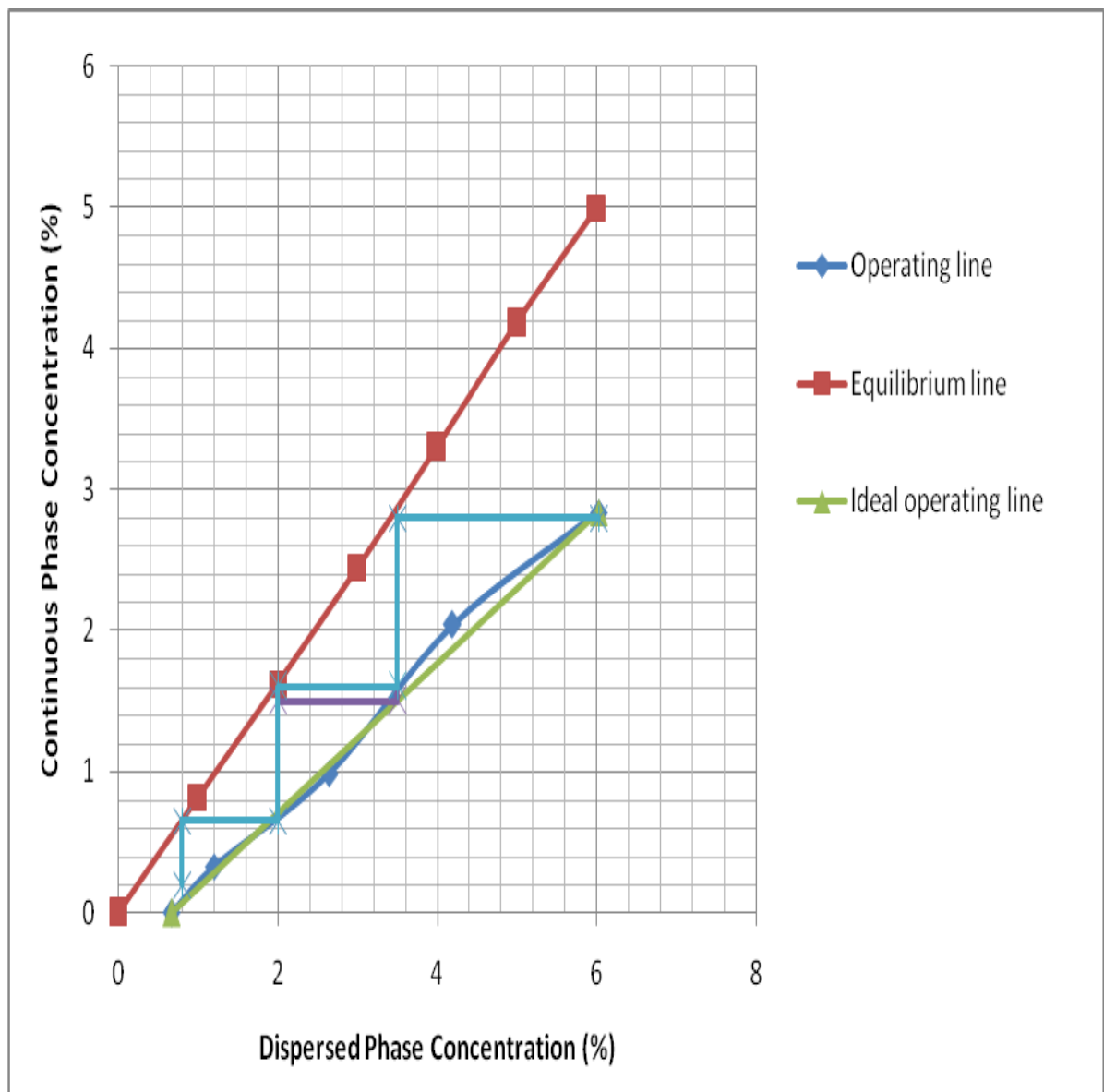
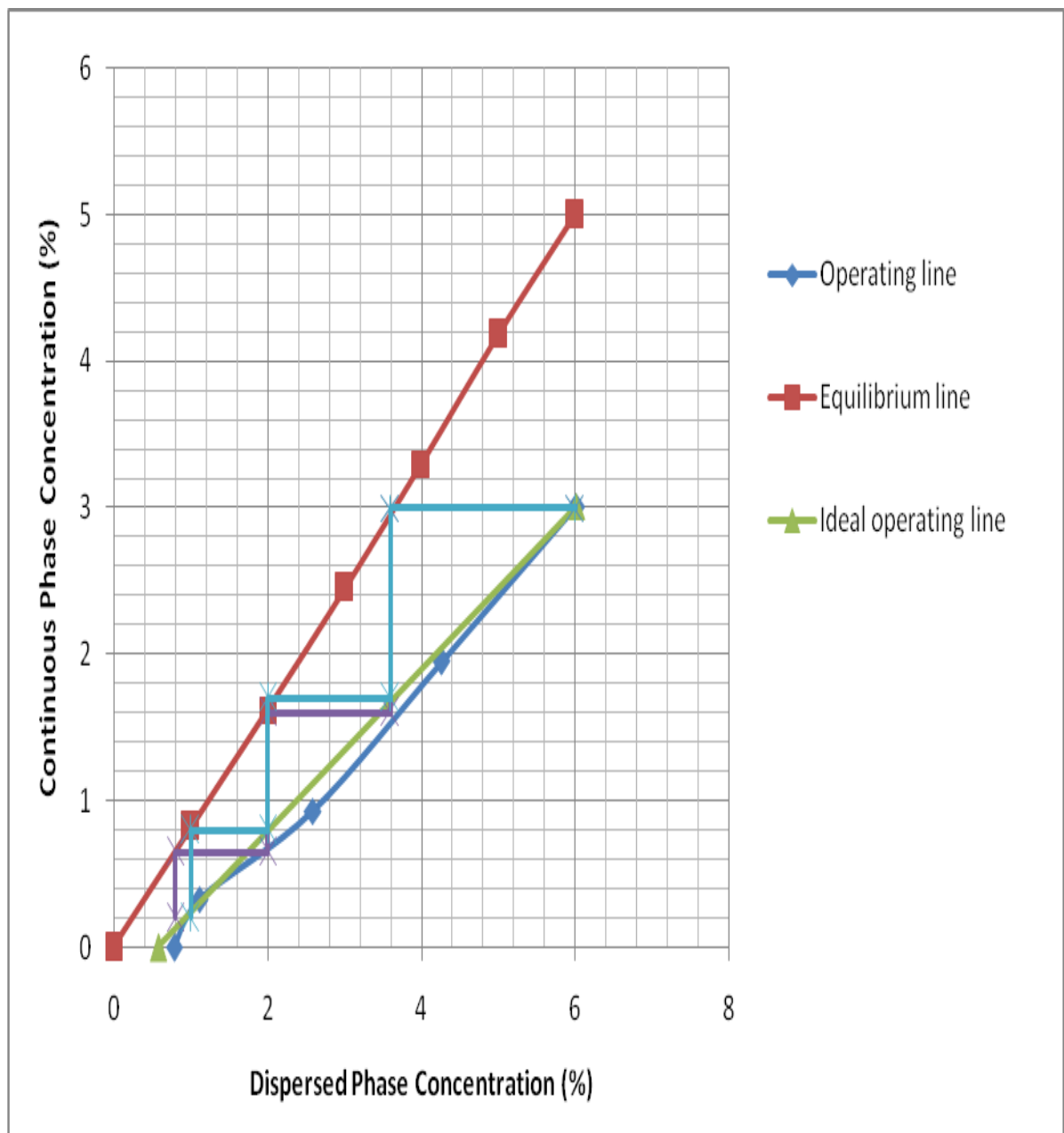


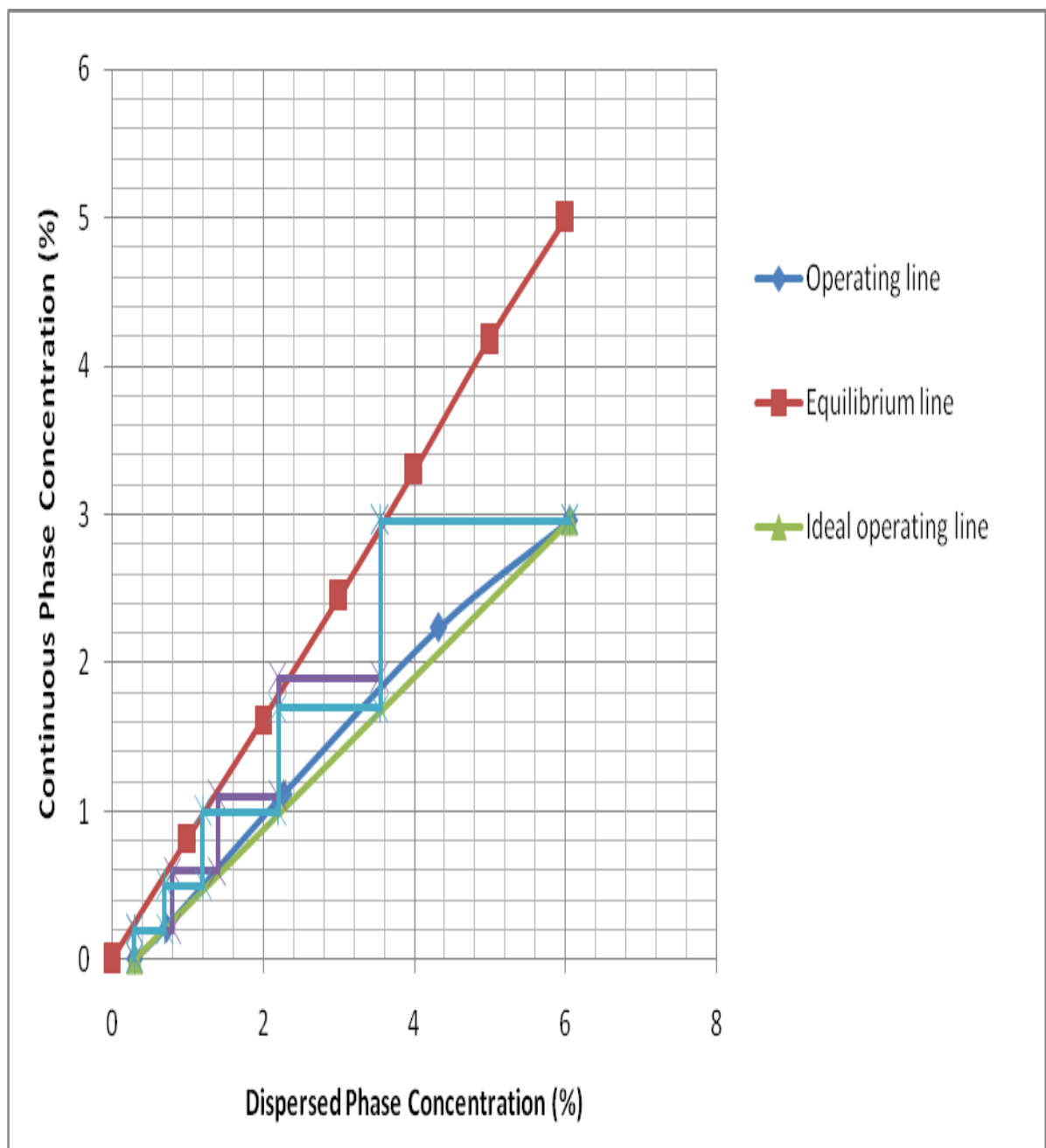
Figure E1. 1: Equilibrium stages $af = 1.25$ mm/s, $d_o = 3.0$ mm (circular downcomers)



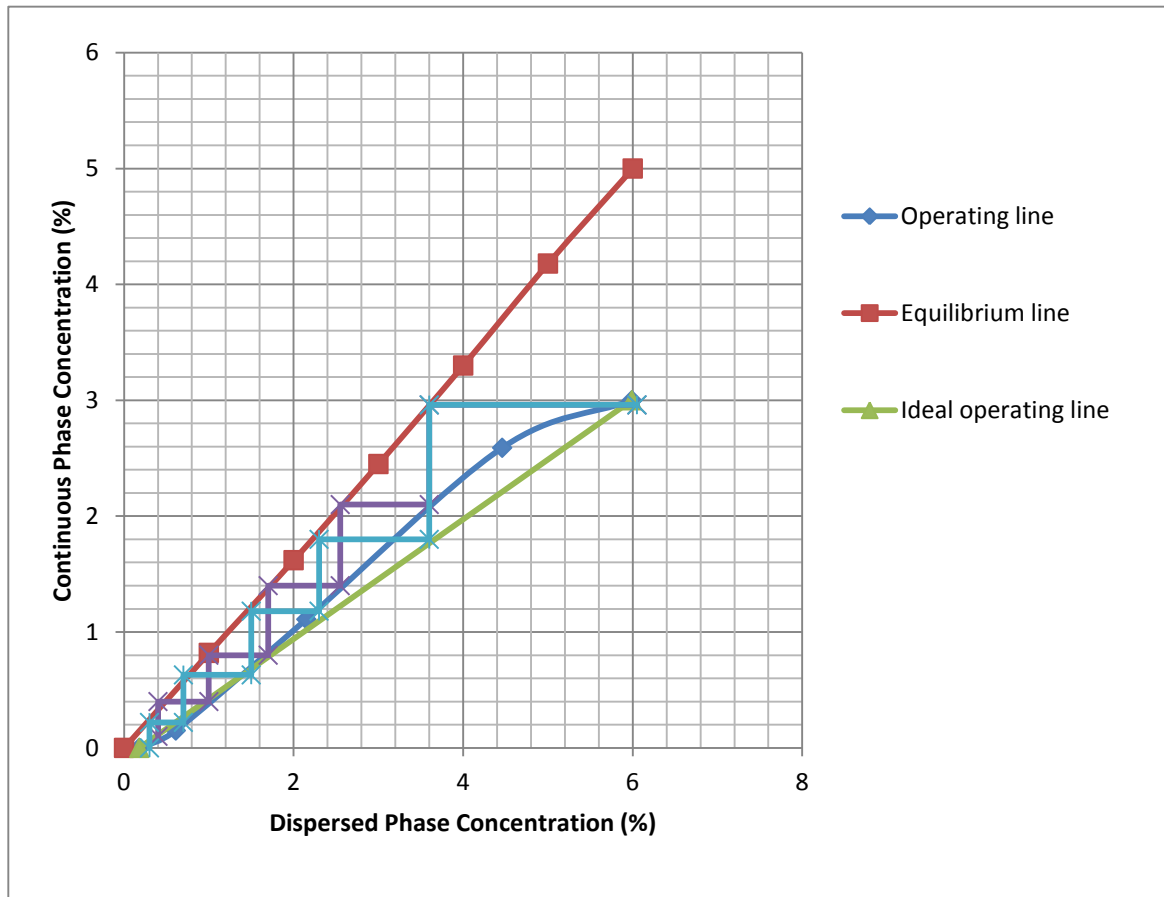
Equilibrium stages with and without backmixing for $h = 150$ mm, $S/F = 1:1$, $af = 2.5$ mm/s, $do = 3.0$ mm, and for circular downcomers



Equilibrium stages with and without backmixing for $h = 150$ mm, $S/F = 1:1$, $af = 3.75$ mm/s, $do = 3.0$ mm, and for circular downcomers



Equilibrium stages with and without backmixing for $h = 150$ mm, $S/F = 1:1$, $af = 6.25$ mm/s, $do = 3.0$ mm, and for circular downcomers



Equilibrium stages with and without backmixing for $h = 150$ mm, $S/F = 1:1$, $af = 7.5$ mm/s, $do = 3.0$ mm, and for circular downcomers

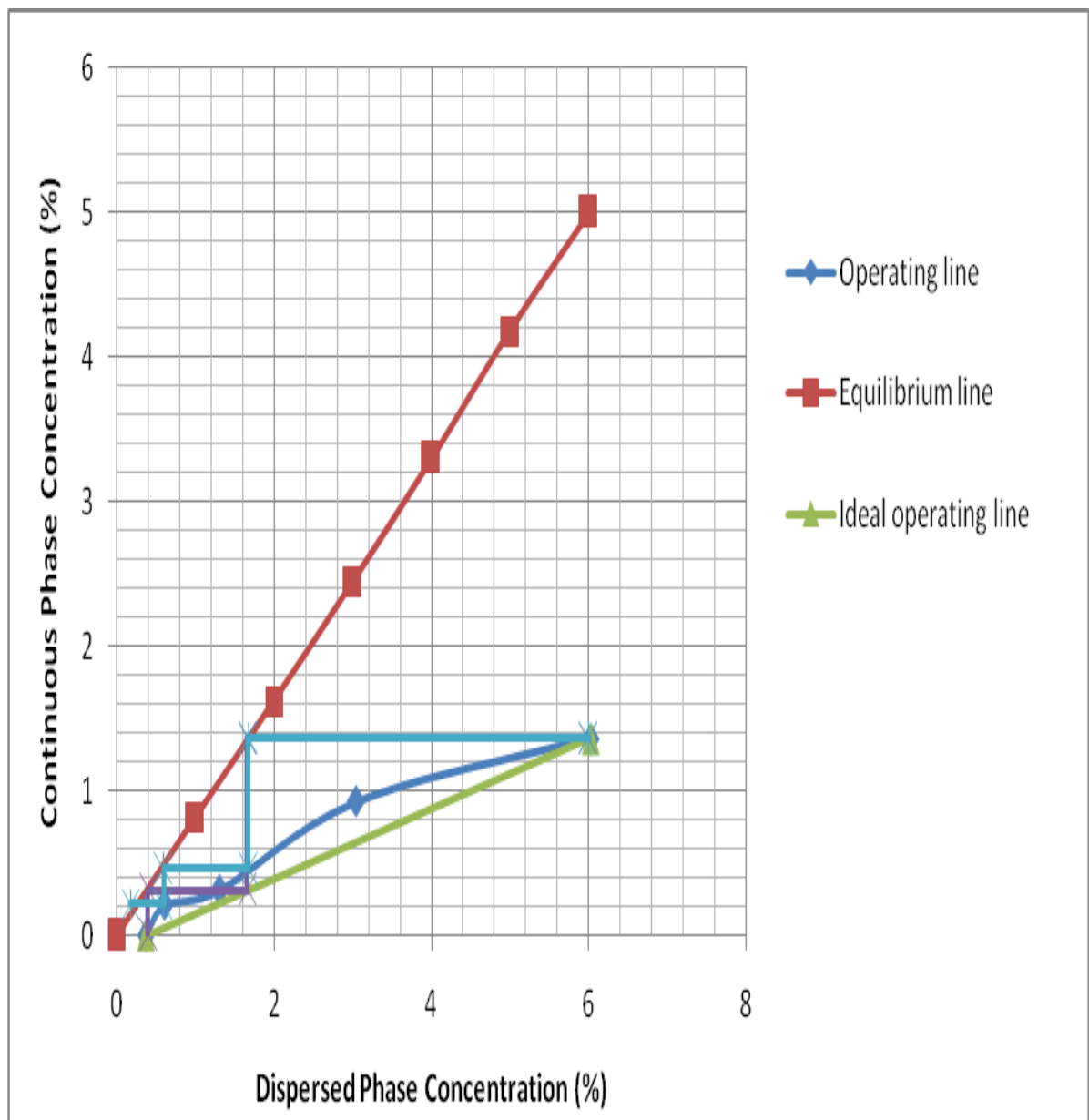
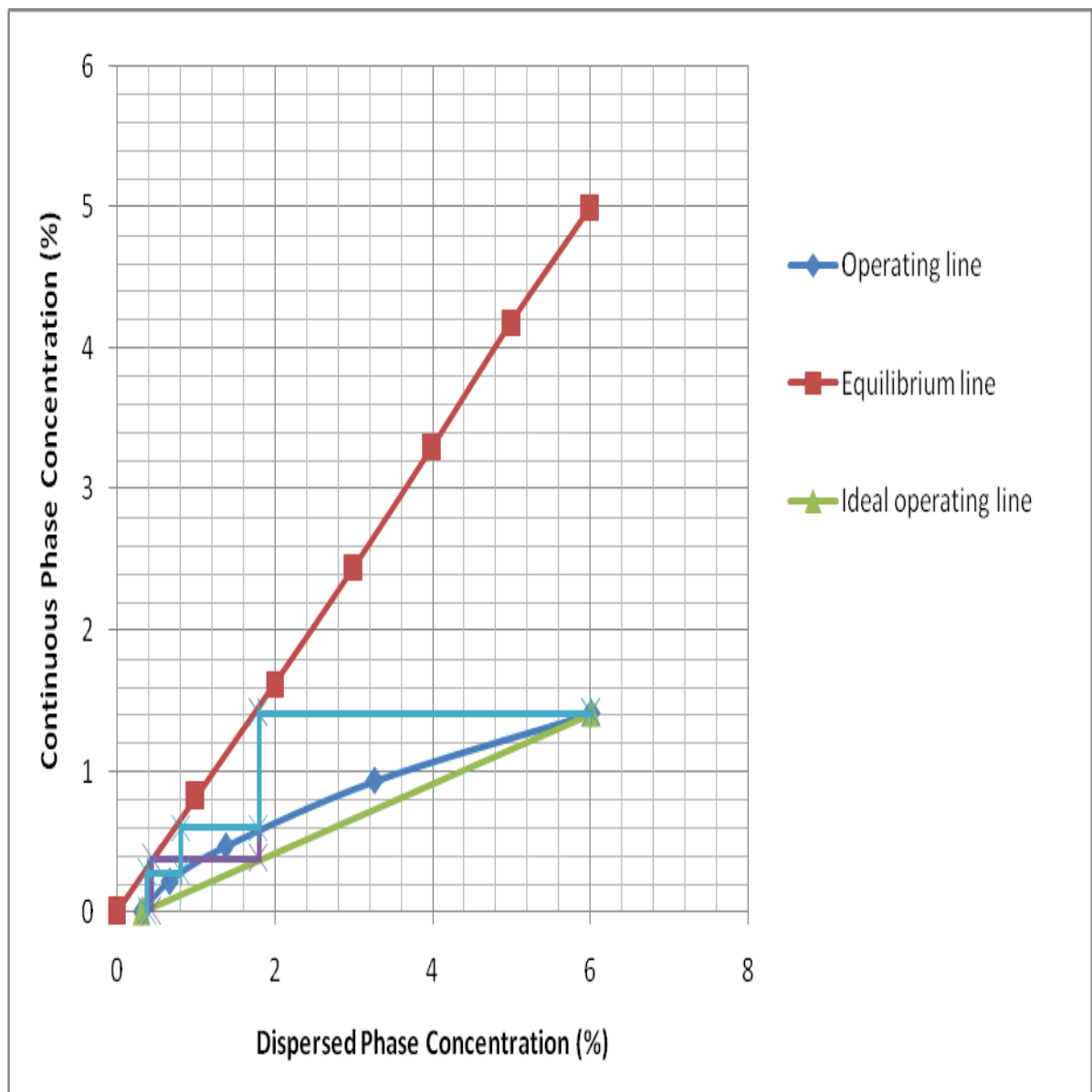
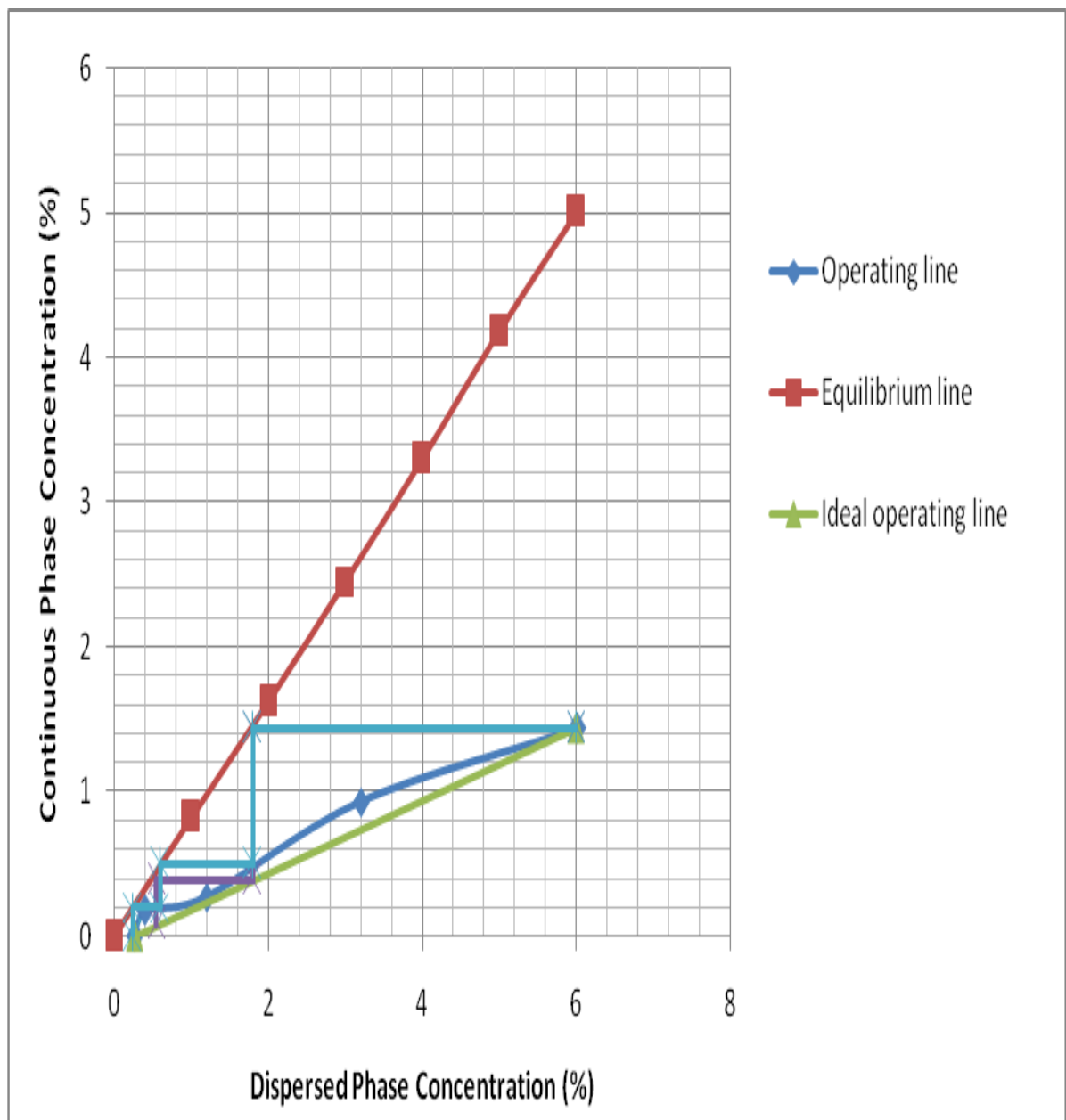


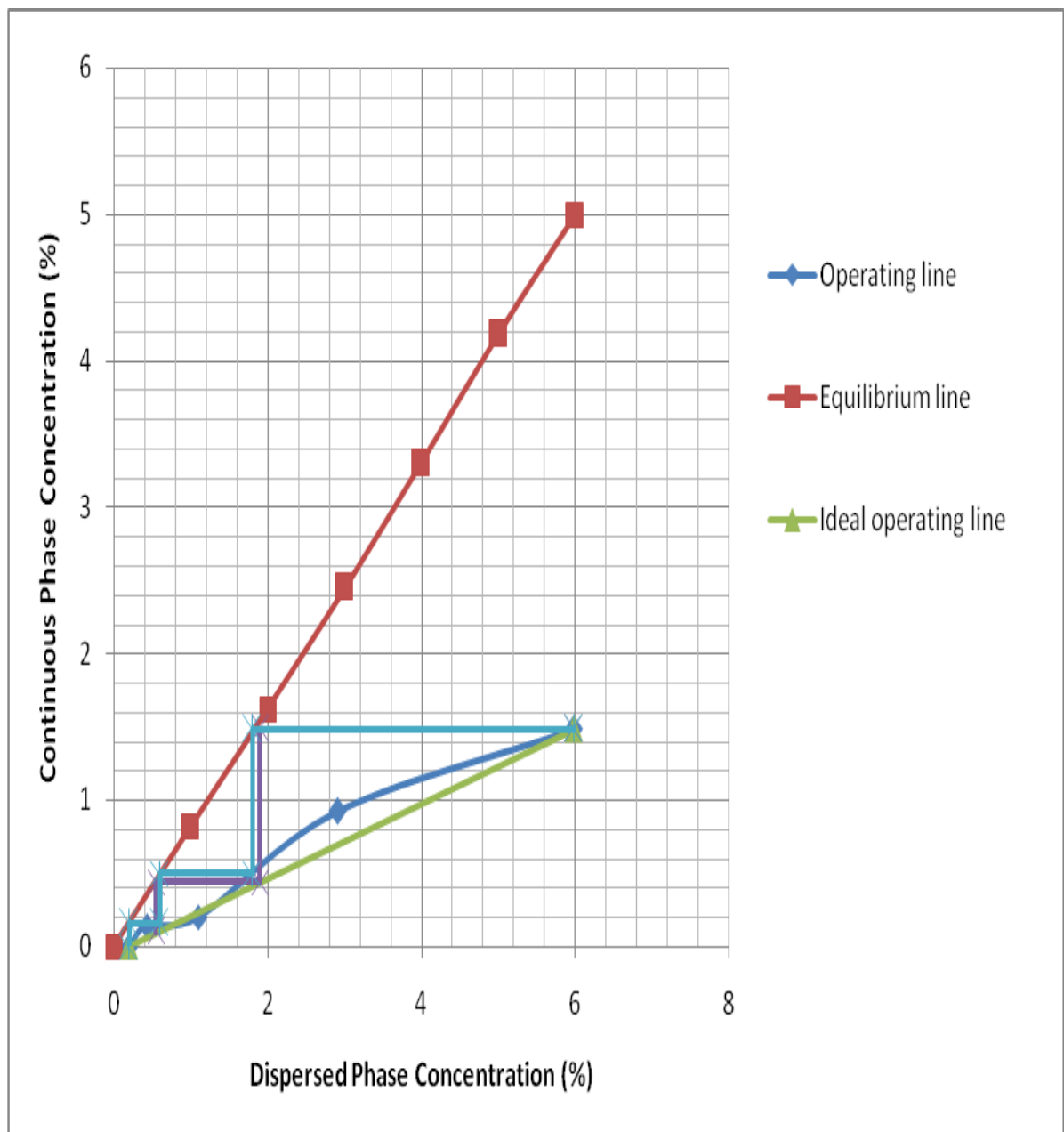
Figure E 1. 2: Equilibrium stages, $af = 2.5$ mm/s, $do = 3.0$ mm (straight segment.)



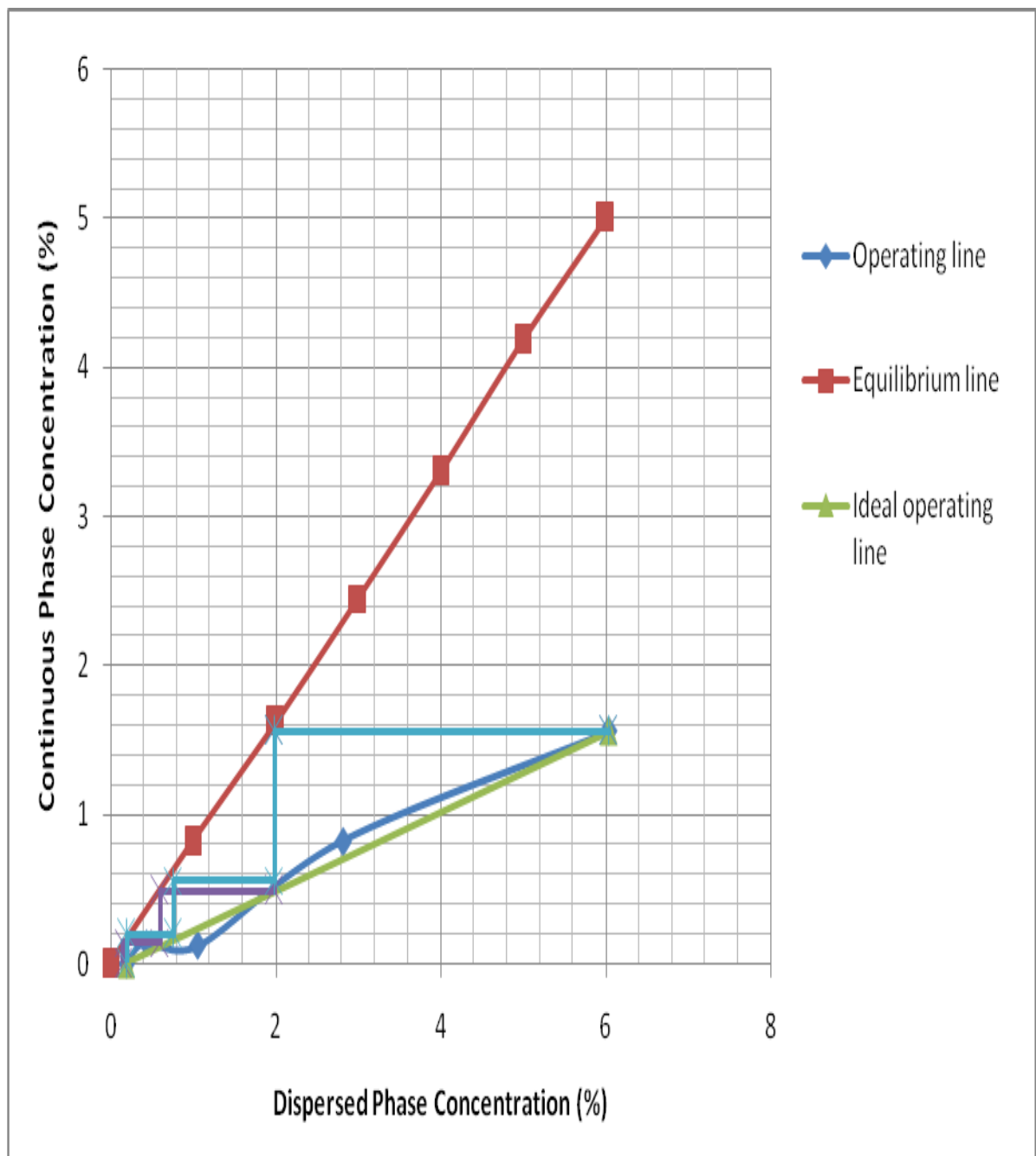
Equilibrium stages ' $af = 3.75$ mm/s, $do = 3.0$ mm, and for straight segment



Equilibrium stages $af = 5.0$ mm/s, $do = 3.0$ mm, and for straight segment.



Equilibrium stages, $af = 6.25$ mm/s, $do = 3.0$ mm, and for straight segment.



Equilibrium stages, $af = 7.5$ mm/s, $do = 3.0$ mm, and for straight segment.

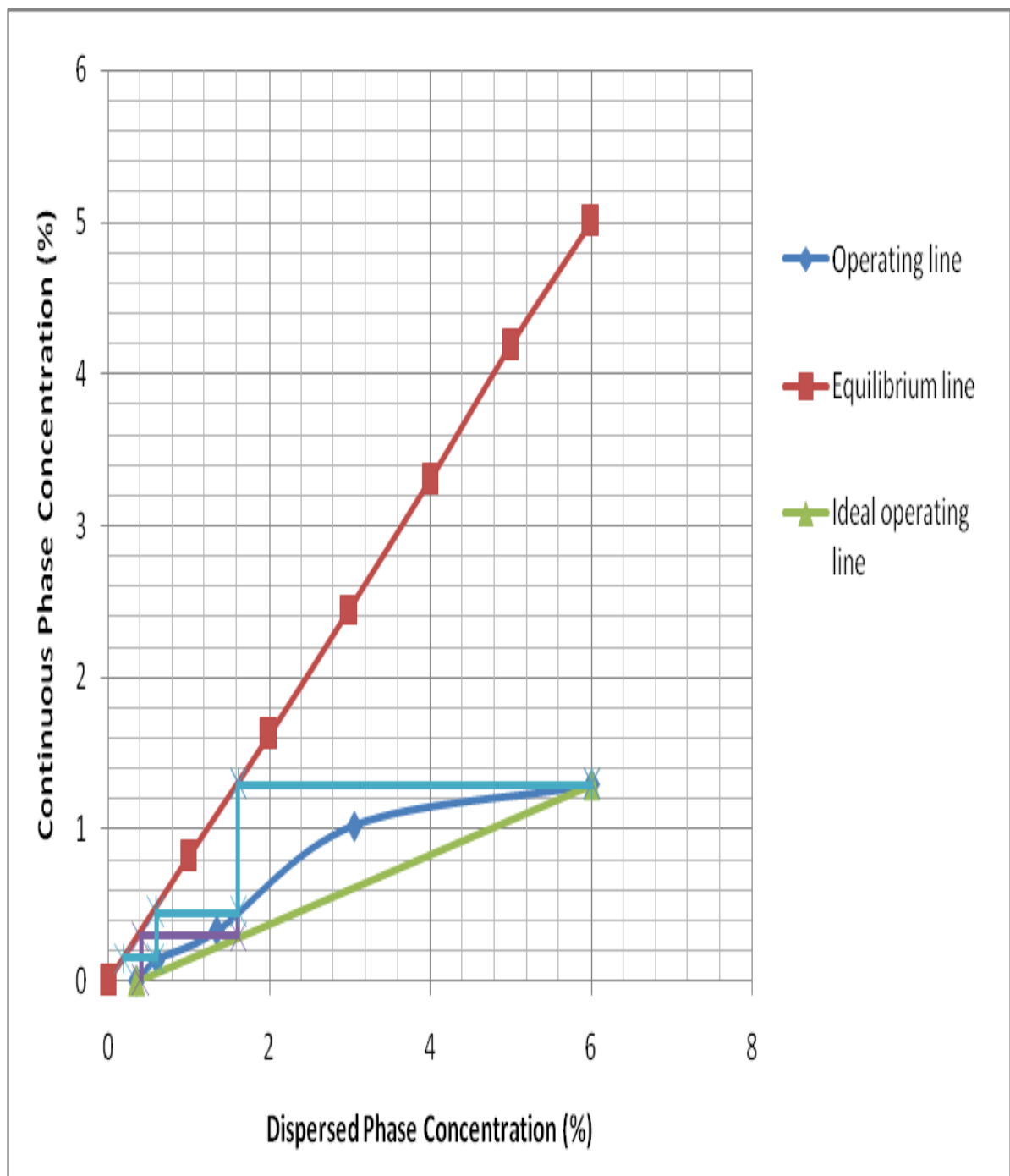
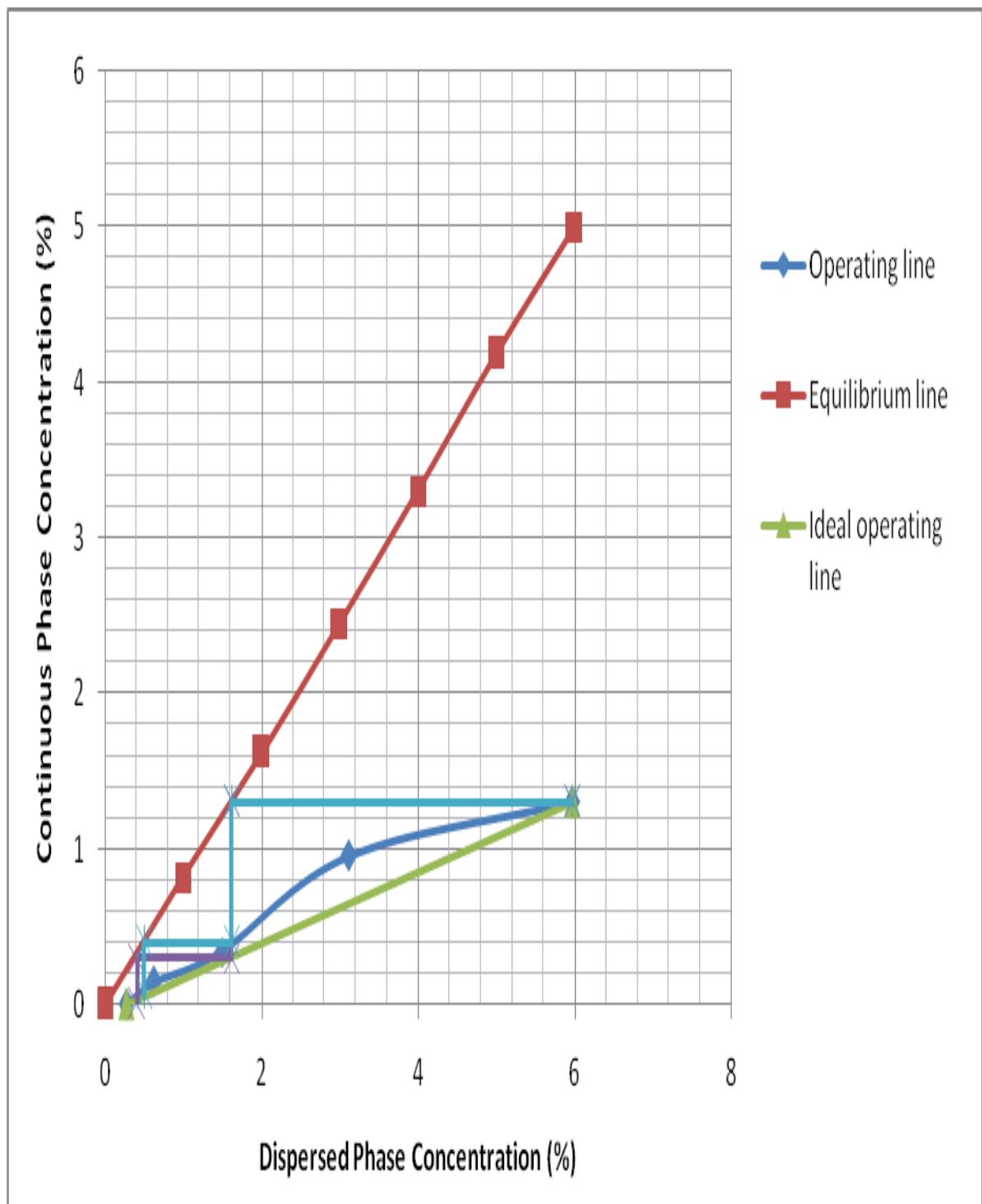
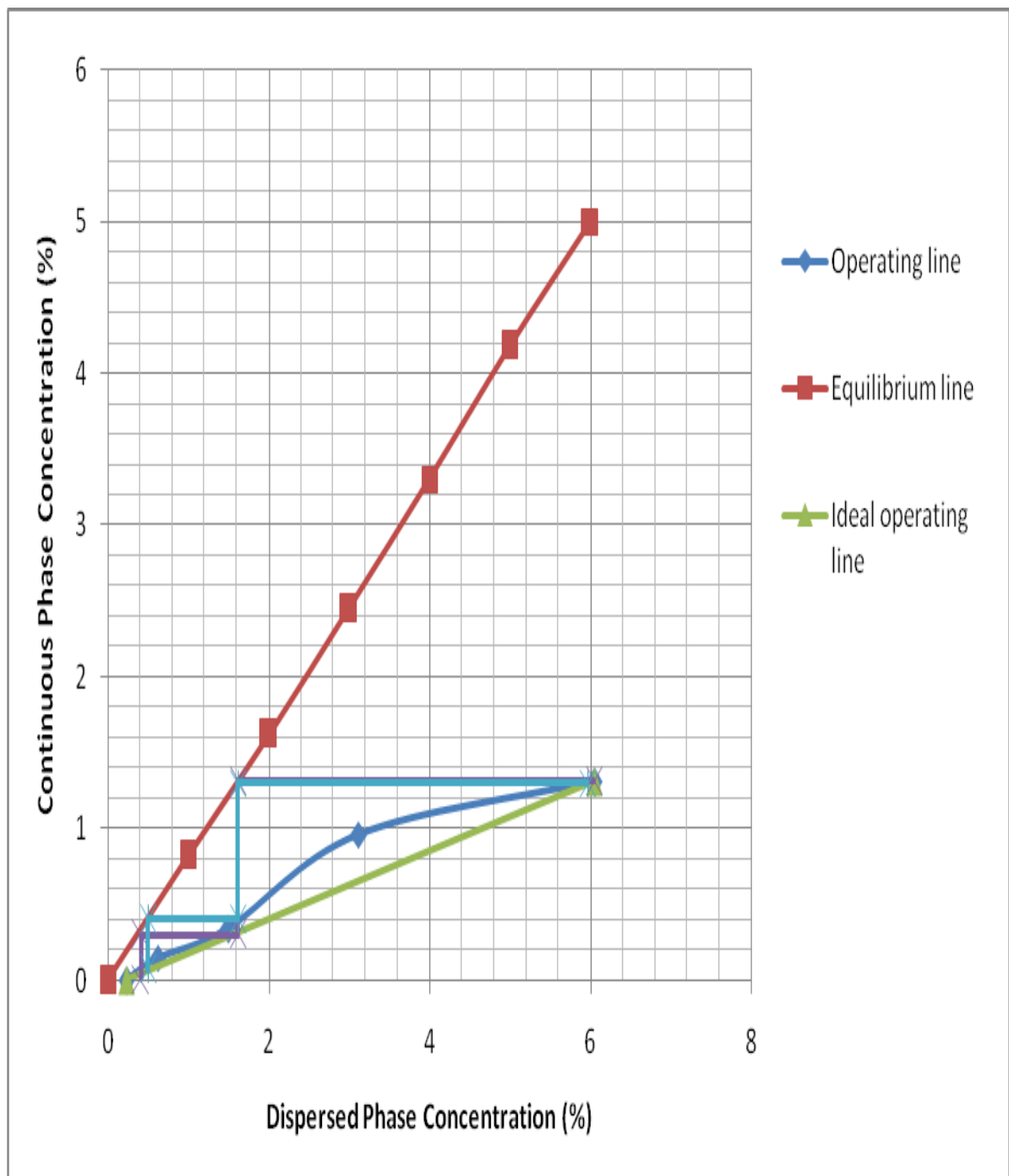


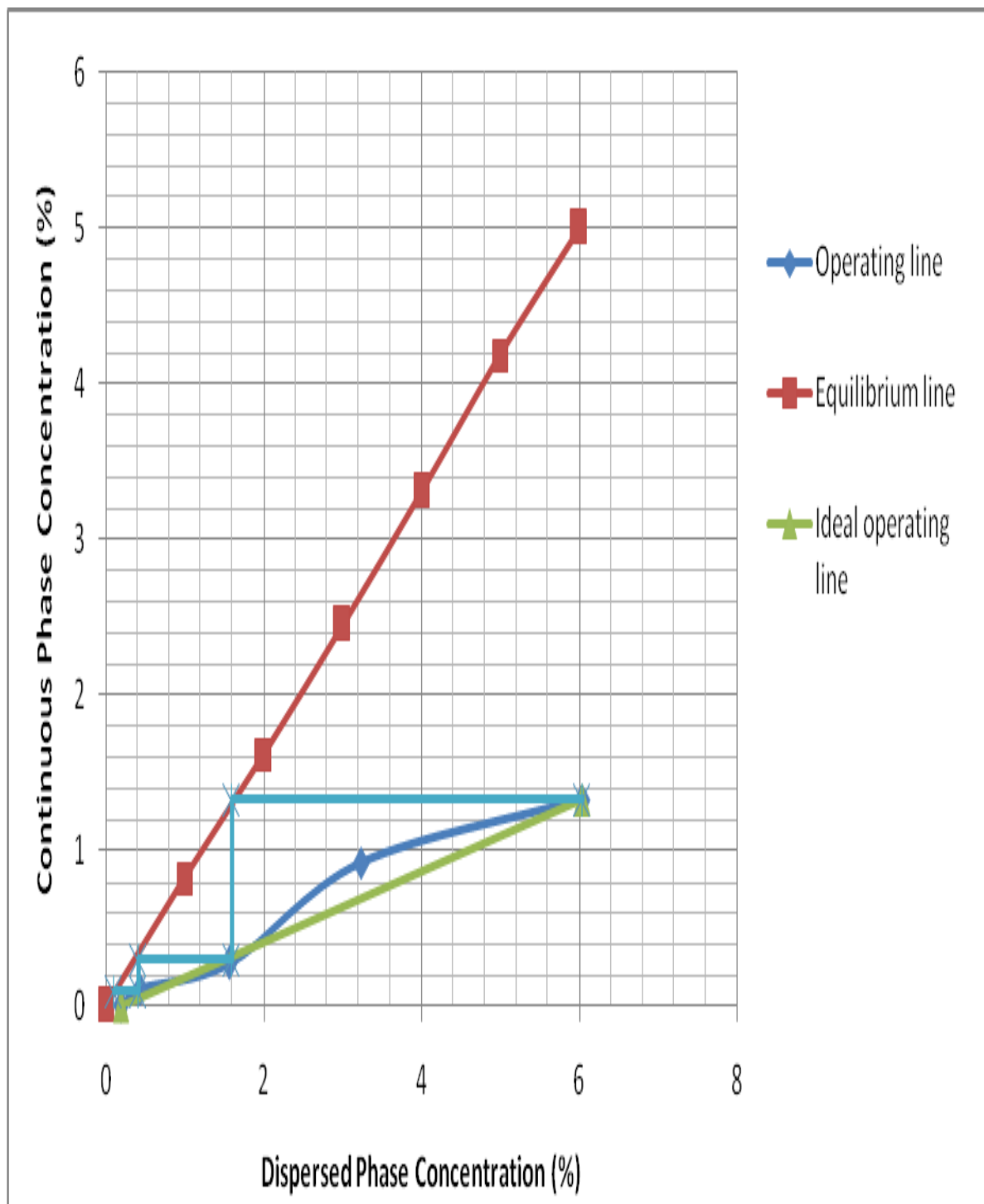
Figure E 1. 3: Equilibrium stages, $af = 1.25$ mm/s, $do = 4.5$ mm, circular downcomers.



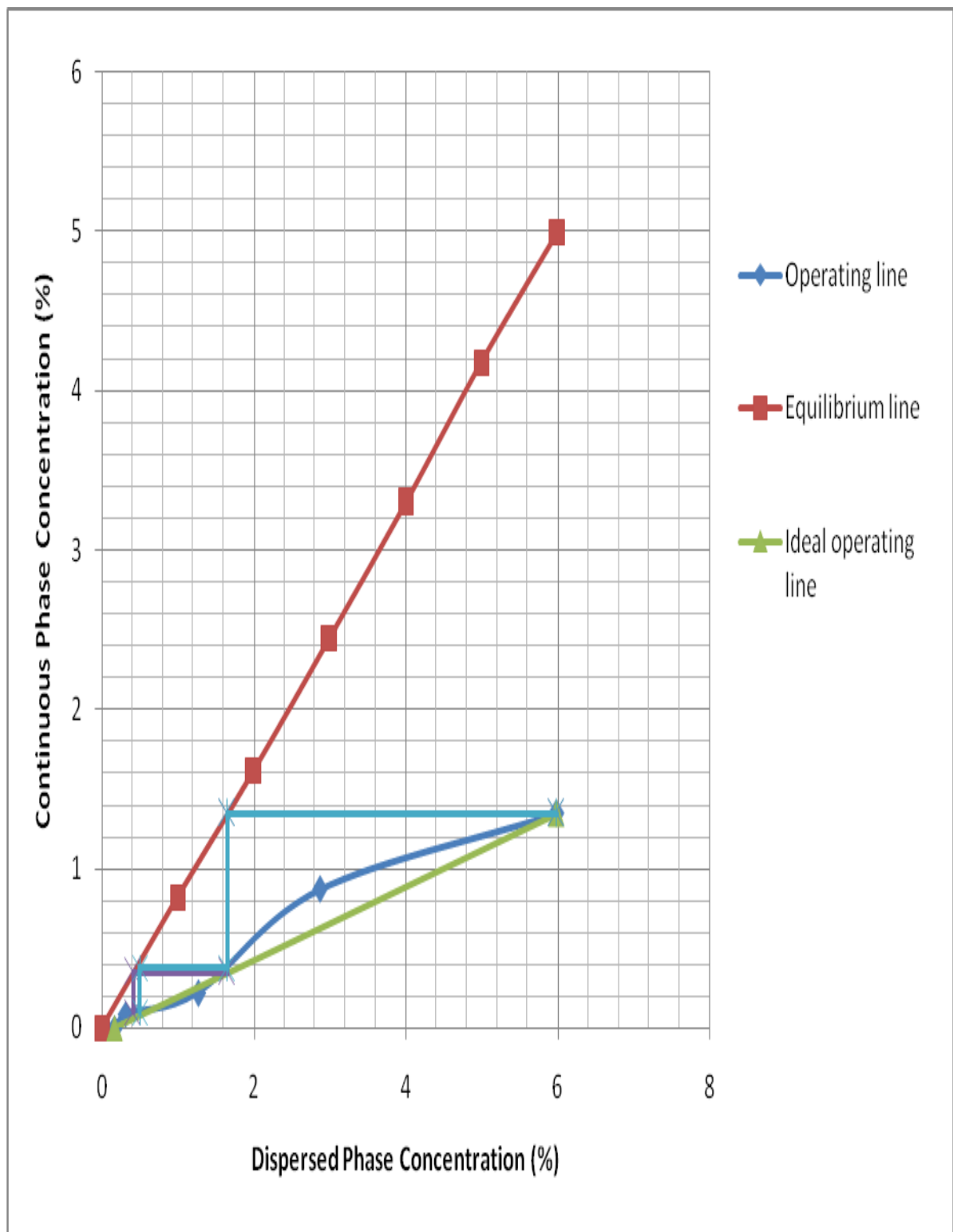
Equilibrium stages, $af = 2.5$ mm/s, $do = 4.5$ mm, circular downcomers.



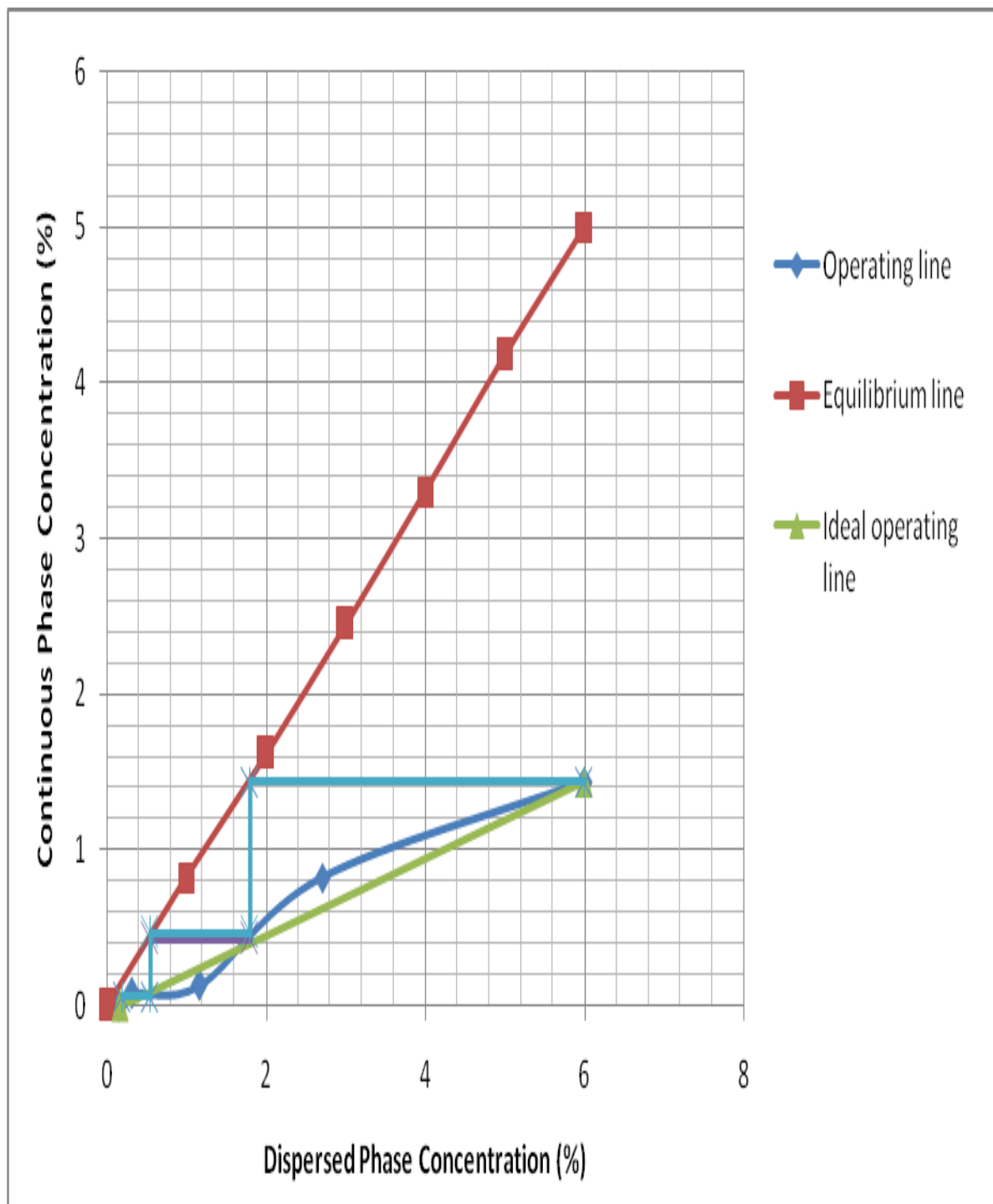
Equilibrium stages with, $af = 3.75$ mm/s, $do = 4.5$ mm, circular downcomers.



Equilibrium stages, $af = 5.0$ mm/s, $do = 4.5$ mm, circular downcomers.



Equilibrium stages, $af = 6.25$ mm/s, $do = 4.5$ mm, circular downcomers.



Equilibrium stages , $af = 7.5$ mm/s, $do = 4.5$ mm, circular downcomers.

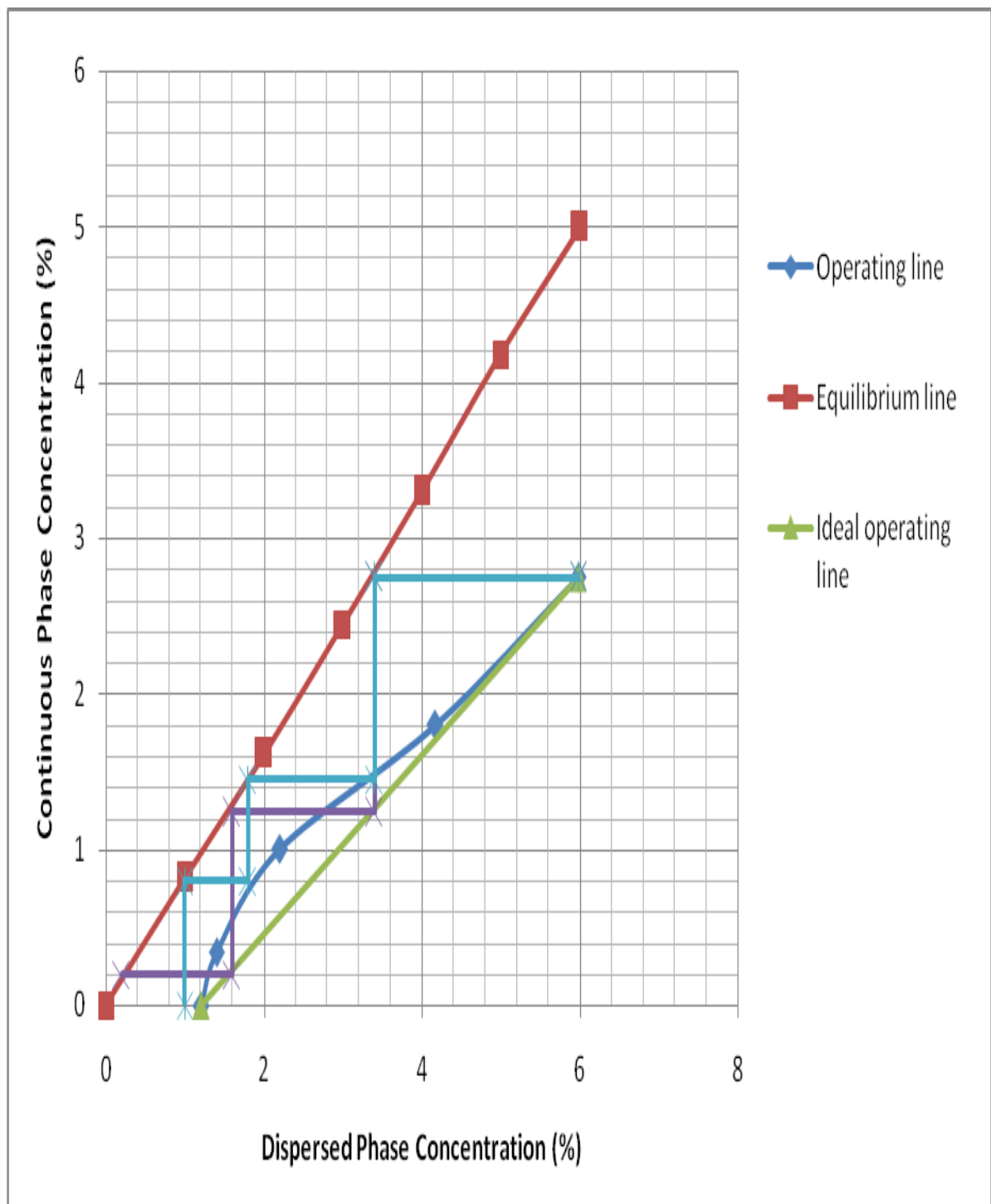
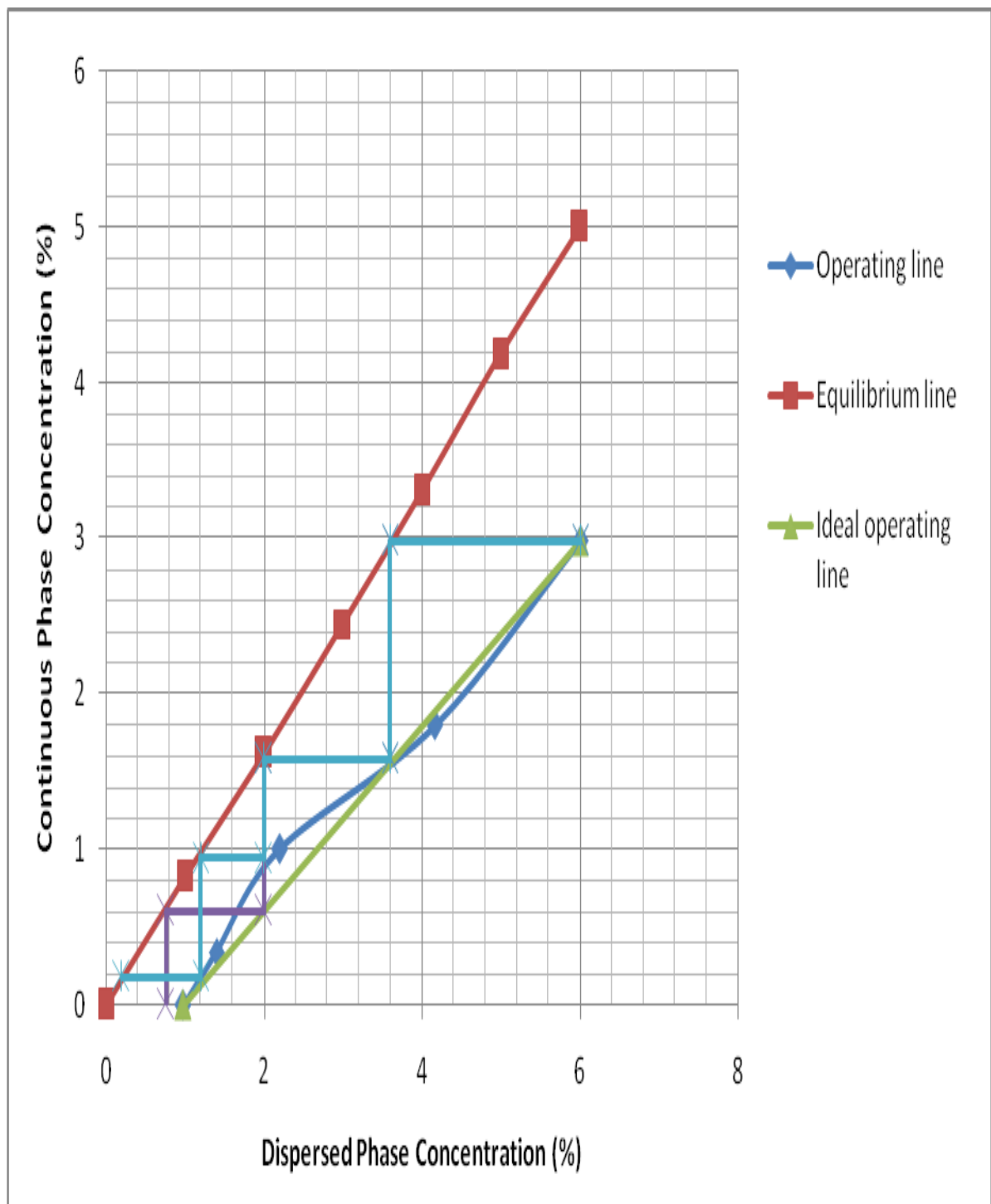
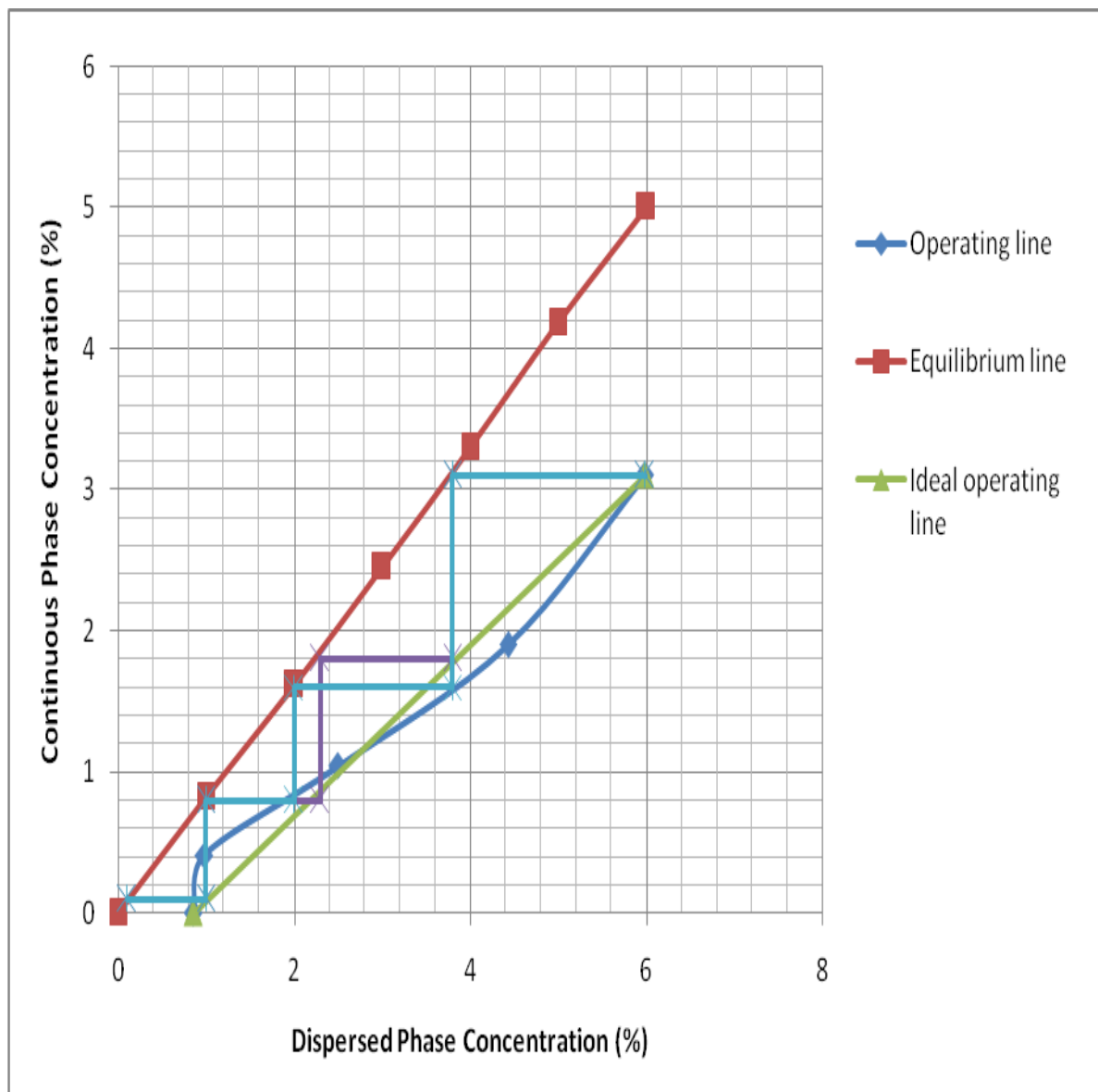


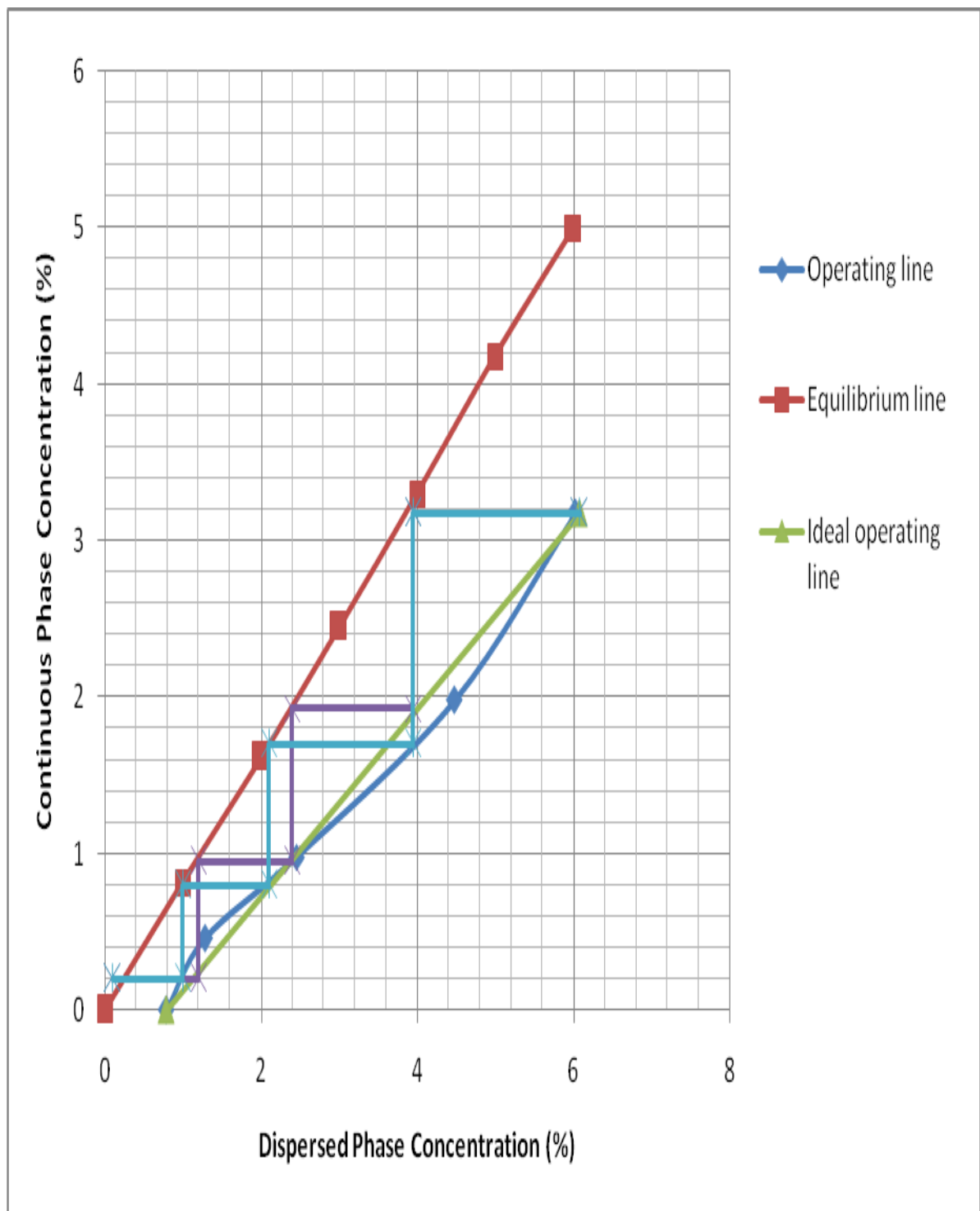
Figure E 1. 4: Equilibrium stages, $af = 1.25$ mm/s, $do = 1.5$ mm, circular downcomers.



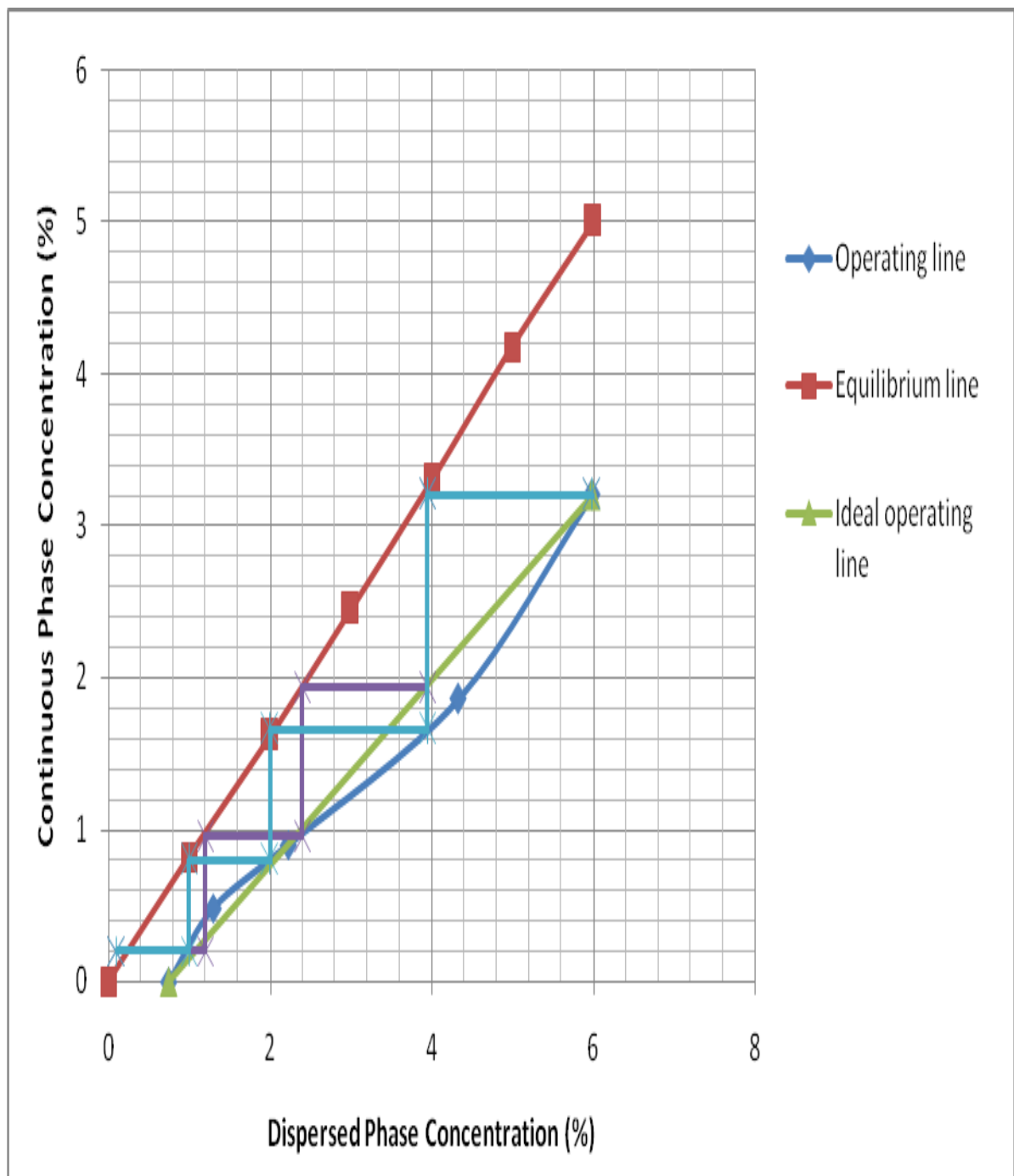
Equilibrium $af = 2.5$ mm/s, $do = 1.5$ mm, circular downcomers.



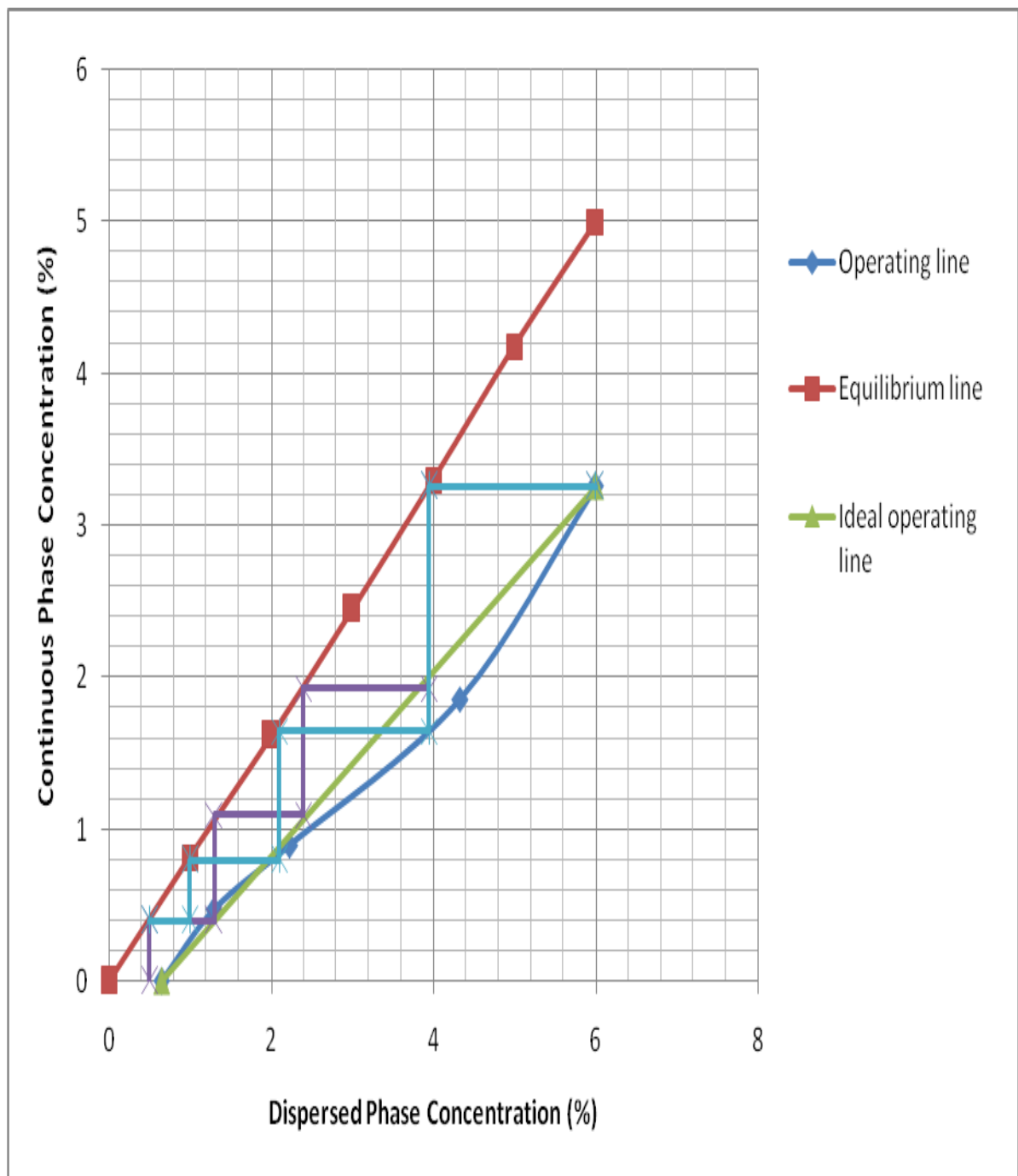
Equilibrium stages, $af = 3.75$ mm/s, $do = 1.5$ mm, and for circular downcomers.



Equilibrium stages $af = 5.0$ mm/s, $do = 1.5$ mm, and for circular downcomers.



Equilibrium stages $af = 6.25$ mm/s, $do = 1.5$ mm, and for circular downcomers.



Equilibrium stages, $af = 7.5$ mm/s, $do = 1.5$ mm, and for circular downcomers.

APPENDIX F: MATERIAL SAFETY DATA SHEETS (MSDS)



Health	2
Fire	3
Reactivity	0
Personal Protection	H

Appendix F.1: Material safety data sheet toluene-d8 MSDS

Section 1: Chemical Product and Company Identification	
Product Name: Toluene Synonym: Toluol, Tolu-Sol; Methylbenzene; Methacide; Phenylmethane; Methylbenzol Chemical Name: Toluene Chemical Formula: C ₆ -H ₅ -CH ₃ or C ₇ -H ₈	
Section 2: Composition and Information on Ingredients	
Molecular weight: 94 Toxicological Data on Ingredients: Toluene: ORAL (LD50): Acute: 636 mg/kg [Rat]. DERMAL (LD50): Acute: 14100 mg/kg [Rabbit]. VAPOR (LC50): Acute: 49000 mg/m 4 hours [Rat]. 440 ppm 24 hours [Mouse].	
Hazards Identification	
Potential Acute Health Effects: Potential Chronic Health Effects: CARCINOGENIC EFFECTS: MUTAGENIC EFFECTS: Not available. The substance may be toxic to blood, kidneys, the nervous system, liver, brain, central nervous system (CNS).	Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator). A4 (Not classifiable for human or animal.) by ACGIH, 3 (Not classifiable for human.) by IARC. Repeated or prolonged exposure to the substance can produce target organs damage.
First Aid Measures	
Eye Contact:	Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention.
Skin Contact:	In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.
Serious Skin Contact:	Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:	If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.
Serious Inhalation:	Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek medical attention.
Ingestion:	Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. If large quantities of this material are swallowed, call a physician immediately. Loosen tight clothing such as a collar, tie, belt or waistband.
Fire and Explosion Data	
Flammability of the Product:	Flammable.
Auto-Ignition Temperature:	480°C (896°F)
Flash Points:	CLOSED CUP: 4.4444°C (40°F). (Seta flash) OPEN CUP: 16°C (60.8°F).
Flammable Limits:	LOWER: 1.1% UPPER: 7.1%
Products of Combustion:	These products are carbon oxides (CO, CO ₂).
Fire Hazards in Presence of Various Substances:	Flammable in presence of open flames and sparks, of heat. Non-flammable in presence of shocks.
Explosion Hazards in Presence of Various Substances:	Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.
Fire Fighting Media and Instructions:	Flammable liquid, insoluble in water. SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray or fog.
Special Remarks on Fire Hazards:	Not available.
Special Remarks on Explosion Hazards:	Toluene forms explosive reaction with 1,3-dichloro-5,5-dimethyl-2,4 imidazolididione; dinitrogen tetra oxide; concentrated nitric acid, sulfuric acid + nitric acid; N ₂ O ₄ ; AgClO ₄ ; BrF ₃ ; Uranium hexafluoride; sulfur dichloride. Also forms an explosive mixture with tetranitromethane.

Section 6: Accidental Release Measures	
Small Spill:	Absorb with an inert material and put the spilled material in an appropriate waste disposal.
Large Spill:	Toxic flammable liquid, insoluble or very slightly soluble in water. Keep away from heat. Keep away from sources of ignition. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Do not touch spilled material. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.
Section 7: Handling and Storage	
Precautions:	Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/vapor/spray. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents.
Storage:	Store in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame).

Exposure Controls/Personal Protection	
Engineering Controls:	Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.
Personal Protection:	Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.
Personal Protection in Case of a Large Spill:	Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.
Exposure Limits:	TWA: 200 STEL: 500 CEIL: 300 (ppm) from OSHA (PEL) [United States] TWA: 50 (ppm) from ACGIH (TLV) [United States] SKIN TWA: 100 STEL: 150 from NIOSH [United States] TWA: 375 STEL: 560 (mg/m3) from NIOSH [United States] Consult local authorities for acceptable exposure limits.

Physical and Chemical Properties			
Physical state:	Liquid.	Vapor Density:	3.1 (Air = 1)
Odor:	Sweet, pungent, Benzene-like.	Boiling Point:	110.6°C (231.1°F)
Color:	Colorless	Melting Point:	-95°C (-139°F)
Odor Threshold:	1.6 ppm	Critical Temperature:	318.6°C (605.5°F)
Solubility:	Soluble in diethyl ether, acetone. Practically insoluble in cold water. Soluble in ethanol, benzene, chloroform, glacial acetic acid, carbon disulfide. Solubility in water: 0.561 g/l @ 25 deg. C.	Specific Gravity:	0.8636 (Water = 1)
Molecular Weight:	92.14 g/mole	Vapor Pressure:	3.8 kPa (@ 25°C)
Stability and Reactivity Data			
Stability:.	The product is stable.		
Instability Temperature:	Not available		
Conditions of Instability:	Heat, ignition sources (flames, sparks, static), incompatible materials		
Incompatibility with various substances:	Reactive with oxidizing agents.		
Corrosivity:	Non-corrosive in presence of glass.		
Special Remarks on Reactivity:	Incompatible with strong oxidizers, silver perchlorate, sodium difluoride, Tetranitromethane, Uranium Hexafluoride. Frozen Bromine Trifluoride reacts violently with Toluene at -80 deg. C. Reacts chemically with nitrogen oxides, or halogens to form nitro toluene, nitrobenzene, and nitro phenol and halogenated products, respectively.		
Special Remarks on Corrosivity:.	Not available		

Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals: WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE.

Acute oral toxicity (LD50): 636 mg/kg [Rat].

Acute dermal toxicity (LD50): 14100 mg/kg [Rabbit].

Acute toxicity of the vapor (LC50): 440 24 hours [Mouse].

Chronic Effects on Humans: CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH, 3 (Not classifiable for human.) by IARC. May cause damage to the following organs: blood, kidneys, the nervous system, liver, brain, central nervous system (CNS).

Other Toxic Effects on Humans:

Hazardous in case of skin contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (premotor).

Special Remarks on Toxicity to Animals:

Lowest Published Lethal Dose:

LDL [Human] - Route: Oral; Dose: 50 mg/kg

LCL [Rabbit] - Route: Inhalation; Dose: 55000 ppm/40min

Special Remarks on Chronic Effects on Humans:

Detected in maternal milk in human. Passes through the placental barrier in human. Embryo toxic and/or foetotoxic in animal. May cause adverse reproductive effects and birth defects (teratogenic). May affect genetic material (mutagenic)

Special Remarks on other Toxic Effects on Humans:

Acute Potential Health Effects:

Skin: Causes mild to moderate skin irritation. It can be absorbed to some extent through the skin.

Eyes: Causes mild to moderate eye irritation with a burning sensation. Splash contact with eyes also causes conjunctivitis, blepharospasm, corneal edema, and corneal abrasions. This usually resolves in 2 days.

Inhalation: Inhalation of vapor may cause respiratory tract irritation causing coughing and wheezing, and nasal discharge. Inhalation of high concentrations may affect behavior and cause central nervous system effects characterized by nausea, headache, dizziness, tremors, restlessness, lightheadedness, exhilaration, memory loss, insomnia, impaired reaction time, drowsiness, ataxia, hallucinations, somnolence, muscle contraction or spasticity, unconsciousness and coma. Inhalation of high concentration of vapor may also affect the cardiovascular system (rapid heartbeat, heart palpitations, increased or decreased blood pressure, dysrhythmia,), respiration (acute pulmonary edema, respiratory depression, apnea, asphyxia), cause vision disturbances and dilated pupils, and cause loss of appetite.

Ingestion: Aspiration hazard. Aspiration of Toluene into the lungs may cause chemical pneumonitis. May cause irritation of the digestive tract with nausea, vomiting, pain. May have effects similar to that of acute inhalation. Chronic Potential Health Effects:

Inhalation and Ingestion: Prolonged or repeated exposure via inhalation may cause central nervous system and cardiovascular symptoms similar to that of acute inhalation and ingestion as well liver damage/failure, kidney damage/failure (with hematuria, proteinuria, oliguria, renal tubular acidosis), brain damage, weight loss, blood (pigmented or nucleated red blood cells, changes in white blood cell count), bone marrow changes, electrolyte imbalances (Hypokalemia, Hypophosphatemia), severe, muscle weakness and Rhabdomyolysis.

Skin: Repeated or prolonged skin contact may cause defatting dermatitis.

Ecological Information
<p>Eco toxicity: Eco toxicity in water (LC50): 313 mg/l 48 hours [Daphnia (daphnia)]. 17 mg/l 24 hours [Fish (Blue Gill)]. 13 mg/l 96 hours [Fish (Blue Gill)]. 56 mg/l 24 hours [Fish (Fathead minnow)]. 34 mg/l 96 hours [Fish (Fathead minnow)]. 56.8 ppm any hours [Fish (Goldfish)].</p> <p>BOD5 and COD: Not available.</p> <p>Products of Biodegradation: Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.</p> <p>Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.</p> <p>Special Remarks on the Products of Biodegradation: Not available.</p>
Disposal Considerations
<p>Waste Disposal: Waste must be disposed of in accordance with federal, state and local environmental control regulations</p>
Transport Information
<p>DOT Classification: CLASS 3: Flammable liquid.</p> <p>Identification: : Toluene UNNA: 1294 PG: II</p> <p>Special Provisions for Transport: Not available</p>
Other Regulatory Information
<p>OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).</p> <p>Health Hazard: 2</p>

Fire Hazard: 3

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 3

Reactivity: 0

Specific hazard:

Protective Equipment: Gloves, lab coat, vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator



Health	2
Fire	3
Reactivity	0
Personal Protection	H

Appendix F.2: Material safety data sheet acetone MSDS

Chemical Product	
Product Name: Acetone Synonym: 2-propanone; Dimethyl Ketone; Dimethyl formaldehyde; Pyro acetic Acid Chemical Name: Acetone Chemical Formula: C ₃ -H ₆ -O	
Section 2: Composition and Information on Ingredients	
Toxicological Data on Ingredients: Acetone: ORAL (LD ₅₀): Acute: 5800 mg/kg [Rat]. 3000 mg/kg [Mouse]. 5340 mg/kg [Rabbit]. VAPOR (LC ₅₀): Acute: 50100 mg/m 8 hours [Rat]. 44000 mg/m 4 hours [Mouse].	
Hazards Identification	
Potential Acute Health Effects: Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator). Potential Chronic Health Effects: CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Classified Reproductive system/toxin/female, reproductive system/toxin/male [SUSPECTED]. The substance is toxic to central nervous system (CNS). The substance may be toxic to kidneys, the reproductive system, liver, skin. Repeated or prolonged exposure to the substance can produce target organs damage.	
First Aid Measures	
Eye Contact:	Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention.
Skin Contact:	In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.
Serious Skin Contact:	Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.
Inhalation:	If inhaled, remove to fresh air. If not breathing, give artificial respiration. If

Serious Inhalation:	breathing is difficult, give oxygen. Get medical attention.
	Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.
Ingestion:	Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. If large quantities of this material are swallowed, call a physician immediately. Loosen tight clothing such as a collar, tie, belt or waistband.
Fire and Explosion Data	
Flammability of the Product:	Flammable.
Auto-Ignition Temperature:	465°C (869°F)
Flash Points:	CLOSED CUP: -20°C (-4°F). OPEN CUP: -9°C (15.8°F) (Cleveland).
Flammable Limits:	LOWER: 2.6% UPPER: 12.8%
Products of Combustion:	These products are carbon oxides (CO, CO ₂).
Fire Hazards in Presence of Various Substances:	Highly flammable in presence of open flames and sparks, of heat..
Explosion Hazards in Presence of Various Substances:	Risks of explosion of the product in presence of mechanical impact: Not available. Slightly explosive in presence of open flames and sparks, of oxidizing materials, of acids.
Fire Fighting Media and Instructions:	Flammable liquid, soluble or dispersed in water. SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use alcohol foam, water spray or fog
Special Remarks on Fire Hazards:	Vapor may travel considerable distance to source of ignition and flash back.
Special Remarks on Explosion Hazards:	Forms explosive mixtures with hydrogen peroxide, acetic acid, nitric acid, nitric acid + sulfuric acid, chromic anhydride, chromyl chloride, nitrosyl chloride, hexachloromelamine, nitrosyl perchlorate, nitryl perchlorate, permonosulfuric acid, thiodiglycol + hydrogen peroxide, potassium ter-butoxide, sulfur dichloride, 1-methyl-1,3-butadiene, bromoform, carbon, air, chloroform, thitriazyl perchlorate
Section 6: Accidental Release Measures	
Small Spill:	Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container.
Large	Flammable liquid. Keep away from heat. Keep away from sources of ignition.

Spill:	Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not touch spilled material. Prevent entry into sewers, basements or confined areas; dike if needed. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.
Section 7: Handling and Storage	
Precautions:	Keep locked up. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, reducing agents, acids, alkalis.
Storage:	Store in a segregated and approved area (flammables area) . Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Keep away from direct sunlight and heat and avoid all possible sources of ignition (spark or flame).

Exposure Controls/Personal Protection			
Engineering Controls:		Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.	
Personal Protection:		Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.	
Personal Protection in Case of a Large Spill:		Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.	
Exposure Limits:		TWA: 500 STEL: 750 (ppm) from ACGIH (TLV) [United States] TWA: 750 STEL: 1000 (ppm) from OSHA (PEL) [United States] TWA: 500 STEL: 1000 [Austalia] TWA: 1185 STEL: 2375 (mg/m3) [Australia] TWA: 750 STEL: 1500 (ppm) [United Kingdom (UK)] TWA: 1810 STEL: 3620 (mg/m3) [United Kingdom (UK)] TWA: 1800 STEL: 2400 from OSHA (PEL) [United States] Consult local authorities for acceptable exposure limits.	
Physical and Chemical Properties			
Physical state:	Liquid.	Vapor Density:	2 (Air = 1)
Odor:	Fruity. Mint-like. Fragrant.	Boiling Point:	56.2°C (133.2°F)

	Ethereal		
Color:	Colorless, Clear	Melting Point:	-95.35 (-139.6°F)
Taste:	Pungent, Sweetish	Critical Temperature:	235°C (455°F)
Solubility:	Easily soluble in cold water, hot water.	Specific Gravity:	0.79 (Water = 1)
Molecular Weight:	58.08 g/mole	Vapor Pressure:	24 kPa (@ 20°C)
Stability and Reactivity Data			
Stability:	The product is stable.		
Instability Temperature:	Not available		
Conditions of Instability:	Excess heat, ignition sources, exposure to moisture, air, or water, incompatible materials		
Incompatibility with various substances:	Reactive with oxidizing agents, reducing agents, acids, alkalis		
Corrosivity:	Non-corrosive in presence of glass.		
Special Remarks on Reactivity:	Not available.		
Special Remarks on Corrosivity:	Not available		
Polymerization:	Will not occur.		

Toxicological Information
<p>Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation.</p> <p>Toxicity to Animals: WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 3000 mg/kg [Mouse]. Acute toxicity of the vapor (LC50): 44000 mg/m³ 4 hours [Mouse].</p> <p>Chronic Effects on Humans: CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH. DEVELOPMENTAL TOXICITY: Classified Reproductive system/toxin/female, Reproductive system/toxin/male [SUSPECTED]. Causes damage to the following organs: central nervous system (CNS). May cause damage to the following organs: kidneys, the reproductive system, liver, skin.</p>

Other Toxic Effects on Humans:

Hazardous in case of skin contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator).

Special Remarks on Toxicity to Animals:

Not available.

Special Remarks on Chronic Effects on Humans:

May affect genetic material (mutagenicity) based on studies with yeast (*S. cerevisiae*), bacteria, and hamster fibroblast cells. May cause reproductive effects (fertility) based upon animal studies. May contain trace amounts of benzene and formaldehyde which may cancer and birth defects. Human: passes the placental barrier.

Special Remarks on other Toxic Effects on Humans:

Acute Potential Health Effects: Skin: May cause skin irritation. May be harmful if absorbed through the skin.

Eyes: Causes eye irritation, characterized by a burning sensation, redness, tearing, inflammation, and possible corneal injury.

Inhalation: Inhalation at high concentrations affects the sense organs, brain and causes respiratory tract irritation. It also may affect the Central Nervous System (behavior) characterized by dizziness, drowsiness, confusion, headache, muscle weakness, and possibly motor incoordination, speech abnormalities, narcotic effects and coma. Inhalation may also affect the gastrointestinal tract (nausea, vomiting). Ingestion: May cause irritation of the digestive (gastrointestinal) tract (nausea, vomiting). It may also affect the Central Nervous System (behavior), characterized by depression, fatigue, excitement, stupor, coma, headache, altered sleep time, ataxia, tremors as well as the blood, liver, and urinary system (kidney, bladder, ureter) and endocrine system. May also have musculoskeletal effects. Chronic

Potential **Health Effects: Skin:** May cause dermatitis.

Eyes: Eye irritation.

Ecological Information

Eco toxicity: Eco toxicity in water (LC50): 5540 mg/l 96 hours [Trout]. 8300 mg/l 96 hours [Bluegill]. 7500 mg/l 96 hours [Flathead Minnow]. 0.1 ppm any hours [Water flea].

BOD5 and COD: Not available.

Products of Biodegradation: Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The product itself and its products of

degradation are not toxic.

Special Remarks on the Products of Biodegradation: Not available..

Disposal Considerations

Waste Disposal: Waste must be disposed of in accordance with federal, state and local environmental control regulations

Transport Information

DOT Classification: CLASS 3: Flammable liquid.

Identification: : Acetone UNNA: 1090 PG: II

Special Provisions for Transport: Not available.

Other Regulatory Information

OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Health Hazard: 2

Fire Hazard: 3

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 1

Flammability: 3

Reactivity: 0

Specific hazard:

Protective Equipment: Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Splash goggles.