

SCALE UP STUDY OF SALT ADDED BATCH DISTILLATION OF
AZEOTROPIC MIXTURE: ISOPROPANOL/WATER

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A thesis submitted in fulfilment
of the requirements for the degree of
Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

FEBRUARY 2013

SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Degree of Bachelor Engineering in Chemical Engineering.

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I hereby declare that the work of this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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*Dedicated to my beloved parents, siblings, and friends
for their love and encouragement*

ACKNOWLEDGEMENTS

First of all, I would like to acknowledge Universiti Malaysia Pahang for providing me with this opportunity to undertake this work and also for the financial support to undertake this study.

I place on record, my sincere thanks to my research supervisor, Professor Ir. Dr. Badhrulhisham bin Abdul Aziz, for his endless encouragement, invaluable advice and unwavering patience throughout the period of my final year project in the university. The constant guidance and assistance offered by my academic supervisor, Pn. Fatmawati binti Adam during this my academic year is also acknowledged. I will forever remain indebted.

I take this opportunity to record my sincere thanks to the Dean and all the staffs in the Faculty of Chemical Engineering and Natural Resources, Universiti Malaysia Pahang for their co-operation and help. I would like to put forward my gratitude to the technical staffs namely Mr. Muhammad Hairynizam bin Muhd Taib for his guidance and assistance carrying out pilot plant experiment.

Last but not least, I place my highest gratitude and appreciation towards my parents, siblings and friends for their unceasing encouragement and support throughout the course of this study. I also place my sense of appreciation to one and all who, directly or indirectly, have lent their helping hand in this research.

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LIST OF ABBREVIATIONS

CBD	Conventional Batch Distillation
DI	Deionised
DMSO	Dimethyl Sulfoxide
EMChiE	European Meeting on Chemical Industry and Environment
FKKSA	Faculty of Chemical & Natural Resources Engineering (Fakulti Kejuruteraan Kimia & Sumber Asli)
IL	Ionic Liquid
IPA	Isopropanol
RCM	Residue Curve Map
VLE	Vapour Liquid Equilibrium

LIST OF SYMBOLS

Symbols

n_j	Number of moles of components j
ν, ν_+, ν_-	Number of moles of ions, cation and anion
x_j	Mole fraction of component j in liquid
y_i	Mole fraction of solvent i in vapour
z_i	Mole fraction of solvent i in mixed solvent

Subscripts

1	Isopropanol (non-electrolyte)
2	Water
3	Salt
+, -	Cation and anion, respectively

Superscript

'	Vapour phase
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**KAJIAN PENSKALAAN UNTUK PROSES BATCH PENYULINGAN
DENGAN PENAMBAHAN GARAM BAGI CAMPURAN BERAZEOTROP:
ISOPROPANOL / AIR**

ABSTRAK

Pemulihan pelarut sering diamalkan dalam industri kimia terutamanya dalam industri pembuatan sarung tangan dan kateter. Walau bagaimanapun, fenomena azeotrop dalam campuran (campuran binari isopropanol-air) merumitkan proses pemisahan. Proses penyulingan secara 'batch' yang konvensional adalah tidak lagi berkesan dalam memulihkan pelarut. Selain itu, pelarut yang tidak dipulihkan akan berakhir sebagai sisa toksik dan pembuangan sisa toksik sebegini membawa impak yang amat bahaya kepada alam sekitar. Kerja eksperimen telah dijalankan dan kajian penskalaan telah dilakukan dalam usaha untuk menyelesaikan masalah ini. Dua objektif dalam kajian ini adalah: (a) menentukan kepekatan garam yang optimum untuk menyuling campuran berazeotrop IPA-air supaya memulihkan ketulenan IPA lebih daripada 90% mol dengan menggunakan penyulingan secara 'batch', dan (b) mengkaji tentang penskalaan unit penyulingan untuk sistem IPA-air dari skala makmal kepada skala loji pandu. Dua peringkat prosedur, iaitu eksperimen berskala makmal dan eksperimen loji pandu, telah dijalankan. Kepekatan garam yang optimum yang diperlukan untuk memecahkan titik azeotrop campuran IPA-air smekain berkurangan selaras dengan peningkatan z_1 . Untuk eksperimen berskala makmal, larutan garam dilarutkan dalam campuran IPA-air dan tiada nisbah refluks boleh dikawal kerana radas yang ringkas digunakan. Keputusan menunjukkan bahawa 2.5 mol% dan 1.57 mol% garam kalsium klorida telah berjaya memulihkan 91% mol IPA dari sistem IPA-air yang masing-masing bernilai $z_1 = 0.51$ dan 0.60. Titik azeotrop campuran binari telah berjaya dipecahkan oleh penambahan larutan garam kalsium klorida. Untuk loji pandu, larutan garam ditambahkan ke bahagian atas turus penyulingan dan nisbah refluks mutlak telah digunakan untuk meningkatkan kecekapan pemindahan haba dan jisim dalam proses penyulingan. Walau bagaimanapun, pendekatan ini tidak berjaya dalam memulihkan ketulenan IPA yang tinggi, di mana fenomena ini boleh dijelaskan oleh ralat dalam data sistem binari kepada sistem multikomponen.

SCALE UP STUDY OF SALT ADDED BATCH DISTILLATION OF AZEOTROPIC MIXTURE: ISOPROPANOL/WATER

ABSTRACT

Solvent recovery is often practised in chemical industry especially in glove and catheter manufacturing. However, azeotropic behaviour in mixture (isopropanol-water binary mixture) complicates the separation process. Conventional batch distillation is no longer effective in recovering the solvent. Moreover, the unrecovered solvent will end up as toxic waste and disposal of such toxic waste is jeopardising to the environment. Experimental works were carried out and scaling up study was performed in order to solve these problems. The research objectives are twofold: (a) to determine optimum salt concentration for distilling IPA-water azeotropic mixture by using salt added batch distillation to recover IPA for purity more than 90 mol%, and (b) to study scaling up of salt added batch distillation unit for IPA-water azeotropic mixture from a laboratory scale to pilot plant scale. Two stages of procedures, which are laboratory scale and pilot plant experiments, were carried out. The optimum salt concentration and the scaling up were studied. The optimum salt concentration needed to break the azeotropic point of IPA-water mixture is generally decreasing as z_1 is higher. Salt solution was dissolved to the still before distillation using laboratory scale and no reflux ratio can be controlled due to simpler apparatus. Results showed that 2.5 mol% and 1.57 mol% of calcium chloride salt has successfully recover 91 mol% of IPA from a IPA-water system with $z_1 = 0.51$ and 0.60 , respectively. The binary mixture azeotropic point is successfully broken down by calcium chloride salt addition. In pilot plant, salt solution was added near to the top of column and total reflux ratio was applied to enhance mass and heat transfer in salt effect distillation. However, it did not succeeded in recovering high purity of IPA, probably due to errors in the extension of binary data to multi-component systems.

CHAPTER 1

INTRODUCTION

1.1 Background

Distillation is the most commonly used separation process in the chemical industries. Distillation separates two or more liquid components in a mixture using the principle of relative volatility or boiling points of each component. However, some mixtures exhibit one or more azeotropes, a state at which the coexisting liquid and vapour phases are of equal compositions (Vivek, Madhura, & Lionel, 2009). In the organic chemical industry, separation of azeotropic and close-boiling point mixtures is frequently encountered (Skouras, Kiva & Skogestad, 2005). Therefore, separate homogenous azeotropic mixtures using distillation is viable by adding an extraneous component, referred to as an entrainer or mass separating agent, to facilitate the separation process (Vivek et al, 2009). This is significant because it is a common solvent recovery technology that is typically used in processing industries, such as pharmaceutical, food and specialty chemical industries (Ma řa & Tah, 2010).

This research was conducted in Universiti Malaysia Pahang, Gambang campus for the Final Year Project. This research studied on the scaling up of a salt-added batch distillation for azeotropic IPA-water mixtures. Experimental works were carried out in Faculty of Chemical and Natural Resources Engineering (FKKSA) laboratory by distilling an azeotropic IPA-water mixture using a batch distillation unit. The optimum salt concentration needed to give 'salting out' effect to IPA-water mixture for distillation was determined. Scaling up study of such experiments from laboratory scale to pilot plant scale was conducted. This research was completed within two semesters.

1.2 Problem Statements

IPA is widely used in the semi-conductor industry as a cleaning agent. Pure IPA is recovered from the waste IPA-water mixture containing water (Luybern & Chien, 2010). The recovery and reuse of organic solvent is generally practised in processing industries because of the increasing solvent cost and potential solvent shortages. In most industries, however, azeotropy in distillation complicates the recovery of solvents. This in turn results in the unrecovered solvents end up as toxic chemical waste. Disposal of such toxic waste is often results in violation of air-, water-, or land-pollution regulation (Hilmen, 2000).

On the other hand, the scale-up of the distillation process of IPA-water binary system from laboratory scale to pilot plant scale and commercial scale is still an unsolved problem due to the insufficient of scaling up data. One of the most essential

questions is whether the performance of large scale equipment can be predicted with the same approach as applied in the laboratory scale (Sundmacher & Kienle, 2003).

IPA recovery is a real industrial problem in rubber industry, such as glove and catheter manufacturing. However, azeotropy has complicated the solvent recovery. A solution from experiment and pilot plant study is needed.

1.3 Research Objectives

The research objectives were twofold:

- i. To determine optimum salt concentration needed for distilling IPA-water azeotropic mixture having an industrial IPA-water composition by using salt added batch distillation to recover IPA for purity more than 90 mol%.
- ii. To study scaling up of salt added batch distillation unit for IPA-water azeotropic mixture from a laboratory scale to pilot plant scale.

1.4 Scope of Study

There were three main scopes for this research. There are a few of separation processes that can be used to separate azeotropic mixture, and salt added batch distillation is chosen for this study. Apart from that, calcium chloride (CaCl_2), a common type of salt, is chosen as the entrainer because it has a high availability and good separation performance. On the other hand, there are two types of scale up parameters: design and operation. Design parameters such as reflux ratio, number of stages and concentration of CaCl_2 salt to be introduced to the distillation column, as well as operation parameters such as salt addition method are also being studied.

1.5 Significance of Study

This study has several significances from the economy, environment and engineering aspects. Firstly, this research can help in saving solvent cost by successfully separating azeotropic IPA-water mixture using salt-added batch distillation to recover IPA more than 90 mol%. Apart from that, this research reduces the unrecovered solvents which are a toxic chemical waste that is harmful to the environment.

1.6 Summary

Optimum salt concentration needed for distilling IPA-water azeotropic mixture has been determined and scaling up study of salt added batch distillation unit from a laboratory scale to pilot plant scale has been performed. Optimum salt concentration was determined through experimental works, data collection and analysis. Salt-added batch distillation, IPA-water azeotropic mixture, and calcium chloride₂ salt are the scopes for this research. This research has successfully recovers the IPA for a purity more than 90 mol%. It helps in saving a significant solvent cost and reduces the disposal of toxic waste.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Industrial production of chemicals involves purification and recovery of the products, by-products and unreacted raw materials. Therefore, solvent recovery is becoming a major issue in the pharmaceutical and specialty chemical industries. However, solvent recovery by conventional batch distillation (CBD) is limited by the frequent presence of azeotropes in the used solvent mixtures. As a result, most distillation processes for the separation of azeotropic or difficult zeotropic mixtures involve the addition of an entrainer (homogeneous and heterogeneous azeotropic distillation or extractive distillation).

In a European meeting on Chemical Industry and Environment (EMChiE), Van Baelen et al. (2010) pinpointed that the strongest commercial argument for recovering solvents is the direct cost savings. In some processes with intensive solvent use, the cost of the solvent can be a significant proportion of the overall

product cost. They further explained that another compelling reason for recovering solvents is the increasing environmental legislation against emissions.

2.2 Batch Distillation

Mujtaba (2004) stated that batch distillation is possibly the most widely used operation for liquid mixtures separation. Batch distillation is commonly utilized for the production of fine chemicals and specialized products such as essential oils, alcoholic beverages, perfume, pharmaceutical and petroleum products. Mujtaba's study (as cited in Lucet, Charamel, Champuis, Guido & Loreau, 1992) found that batch distillation is the most frequent separation method in batch process.

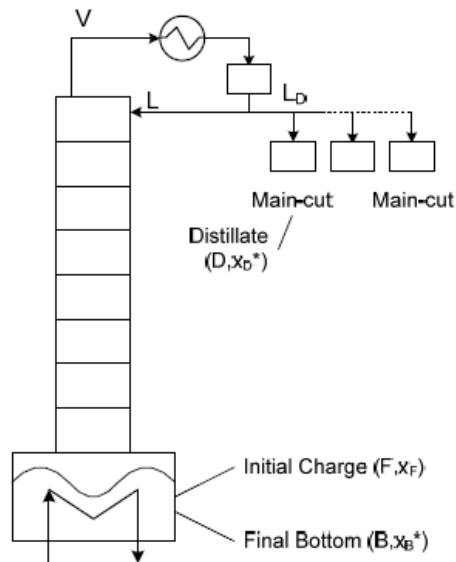


Figure 2.1 A conventional batch distillation (CBD) unit

(Source: Mujtaba, 2004)

Figure 2.1 shows a conventional batch distillation (CBD) unit. The essential parts of a CBD column are as followed:

- i. A bottom receiver/reboiler which is charged with the feed to be processed and which provides the heat transfer surface.
- ii. A rectifying column (either a tray or packed column) superimposed on the reboiler, coupled with either a total condenser or a partial condenser system.
- iii. A series of product accumulator tanks connected to the product streams to collect the main and/or the intermediate distillate fractions.

Batch distillation is usually started by charging a batch of liquid feed into the tank. The more volatile component in the mixture will be vapourised and rising through a column above the tank combines with reflux coming down the column to effect concentration. Operation of such a column involves running the fractionation until a required amount has been distilled off. During the operation, the overhead composition varies and generally a number of cuts are made. Some of the cuts are desired products (main-cuts) while others are intermediate fractions (off-cuts) that can be recycled to subsequent batches to obtain further separation. A residual bottom fraction may or may not be recovered as product (Mujtaba, 1989; 2004).

2.2.1 Advantages of Batch Distillation

Although in the process industries, most distillation systems are continuous, batch systems are preferred for the distillation of relatively small quantities of solvents. Ma řa and Tah's journal (as cited in Kim & Diwekar, 2000) stated that the most outstanding feature of batch distillation is its flexibility in design and operation. Its high flexibility enables it can be used to recover plenty of different solvents. It is also very practical in pilot-plant operations and in processes where the compositions of the materials to be separated vary widely or the main product contains only small amounts of impurities (as cited in Kister, 1992). Apart from that, multi-component mixtures can be separated using one single batch column (as cited in Stichlmair & Fair, 1998). A batch system can often separate many components in one column, albeit with a premium on utilities. Nevertheless, in order to separate a multi-component mixture of n components by continuous distillation, a minimum of $n-1$ separate columns are required, which involves a significantly higher capital cost. While a batch system is more energy intensive than a continuous system, steam costs generally are less significant on a small operation.

2.3 Azeotropy

All liquid mixtures have intermolecular forces of attraction. When two or more liquid components are mixed, the intermolecular interactions may cause the mixture to form certain "inseparable" compositions where the vapour and liquid compositions at equilibrium are equal within a given pressure and temperature range.

These specific mixture compositions are termed azeotropes, and the phenomena is called azeotropy.

2.3.1 IPA-Water Binary System

At atmospheric condition, a binary mixture of IPA–water forms a homogeneous minimum-boiling azeotrope at 87.4–87.7 mass% and 80.3–80.4 °C. Some investigations have reported that IPA–water azeotrope can also be broken with other azeotropic distillations to form heterogeneous azeotropic systems by adding one of the following entrainers: isopropyl ether, benzene, methyl ethyl ketone, and isopropyl acetate.

2.4 Vapour Liquid Equilibrium (VLE) Diagram

To understand the nature of simple distillation, fractional distillation and azeotropes, a vapour/liquid diagram for pairs of solvents is studied. Figure 2.2 shows a typical VLE diagram for 2 solvents, A and B where A is the lower boiling material. The bottom of the graph shows the liquid state and the top of the graph shows the vapour state. The area in between the two curves shows what is happening in the distillation column. The x-axes at the top and bottom represent the composition of component A and B in the mixture, respectively. The y-axis is the temperature of the mixture.

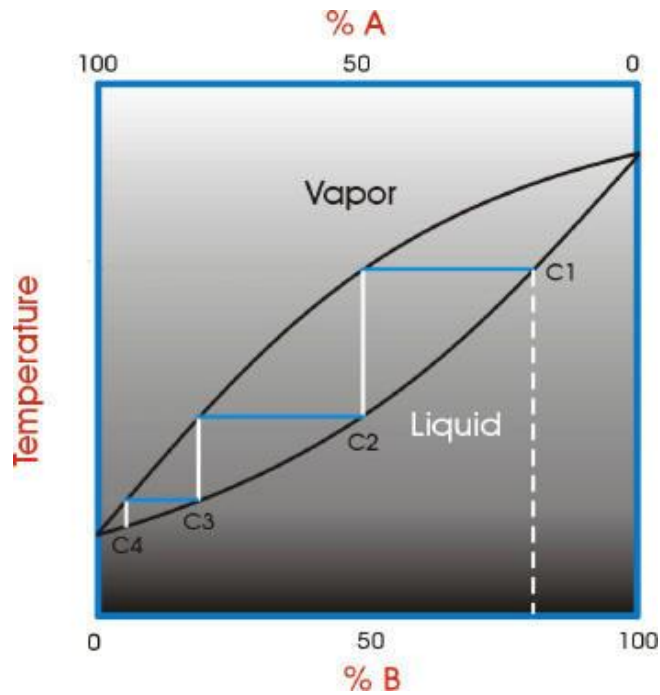


Figure 2.2 A typical VLE diagram

(Source: Smallwood, 2002)

A liquid mixture of A and B with certain temperature and a composition corresponds to the point C1, where its temperature can be read off horizontally to the y-axis, while its component composition can be read off by following the dashed line down to the x-axis at the top and bottom for composition of A and B, respectively. From the point C1, the distillation is happened by tracing the horizontal line to the left until it reaches the vapour curve. This process has improved the concentration of A, the lower boiling component. The material is then condensed by following the solid vertical line down to the liquid curve. If this was simple distillation, distillation could stop now. It can be seen that the purification effected by the simple distillation of such a mixture of volatile liquids is very imperfect.

In a fractional distillation, however, the distillation process continues. The condensed material is vapourised again by following the horizontal line across from the liquid curve at C2 to the vapour curve. There is another improvement in the concentration of the lower boiling component A. The vapour is condensed again by following the vertical line down to the liquid curve at point C3. These repeating vapourisation-condensation steps continue. The number of times that the process of vaporisation and condensation occurs depends on the efficiency of the distillation column. The more efficient the distillation column, the more times this happens and the purer the final product will be.

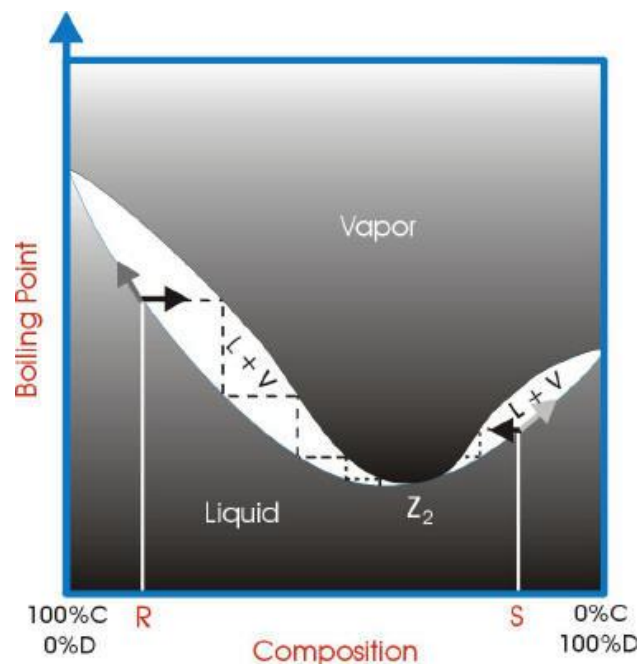


Figure 2.3 A VLE diagram showing azeotrope point, Z_2

(Source: Smallwood, 2002)

Figure 2.3 depicts a VLE diagram showing azeotropic point. For an azeotropic mixture, the vapour liquid curves are not ideal and have a point where the vapour curve meets the liquid curve. The component compositions, temperature and

pressure of liquid phase are equal to those of vapour phase. This point is called the azeotrope point, as shown as point Z_2 .

2.5 Azeotropic Distillation

In the thesis written by Hilmen (2000), he stated that further separation of azeotropic mixture by ordinary distillation is no longer possible since no enrichment of the vapour phase occurs at this point. Therefore, in most cases, azeotropic mixtures require special methods to facilitate their separation. Such methods utilize a mass separating agent other than energy that causes or enhances a selective mass transfer of the azeotrope-forming components.

In the definition of McGraw-Hill Concise Encyclopedia of Science and Technology (2005), azeotropic distillation is any of several processes by which liquid mixtures containing azeotropes may be separated into their pure components with the aid of an additional substance (called the entrainer, the solvent, or the mass separating agent) to facilitate the distillation. Therefore, it is often possible to ‘break’ the azeotrope by adding a carefully selected entrainer to the mixture, thereby achieve the desired separation.

Entrainers fall into at least four distinct categories that may be identified by the way in which they make the separation. These categories are: (1) liquid entrainers that do not induce liquid-phase separation, used in homogeneous azeotropic distillations, of which classical extractive distillation is a special case; (2) liquid

entrainers that do induce a liquid-phase separation, used in heterogeneous azeotropic distillations; (3) entrainers that react with one of the components; and (4) entrainers that dissociate ionically, that is, salts. Within each of these categories, not all entrainers will make the separation possible, that is, not all entrainers will break the azeotrope possible (McGraw-Hill Concise Encyclopedia of Science and Technology, 2005, pp 236-237).

In a journal reviewing about batch distillation written by Skouras et al. (2005), they stated that a process is termed extractive batch distillation when a heavy entrainer is added continuously in the top section of the batch column,. When the entrainer forms a homoazeotrope with at least one of the original components and is added batch-wise to the original mixture, the process is called homogeneous azeotropic or homoazeotropic batch distillation. When the entrainer forms a binary heteroazeotrope with at least one (and preferably with only one) of the original components or a ternary heteroazeotrope and is added batch-wise to the original mixture, the process is called heterogeneous azeotropic or heteroazeotropic batch distillation.

In order to determine whether a given entrainer is practicable, a schematic representation known as a residue curve map (RCM) for a mixture undergoing simple distillation is created. The path of liquid compositions starting from some initial point is the residue curve. The collection of all such curves for a given mixture is known as a residue curve map. These maps contain exactly the same information as the corresponding phase diagram for the mixture, but they represent it in such a way that it is more useful for understanding and designing distillation systems

(McGraw-Hill Concise Encyclopedia of Science and Technology, 2005, pp 236-237).

2.5.1 Heterogeneous Azeotropic Distillation (Separations Using a Light Entrainer)

According to Luybern and Chien (2010), they stated that one way to separate a mixture containing an azeotrope is to add a light entrainer into the system so that additional azeotropes(s) can be formed that helps in the separation. In order to make the separation feasible, there are two important characteristics of this additional azeotrope (or one of the additional azeotropes). Firstly, the azeotropic temperature of one additional azeotrope should be the minimum temperature of the whole ternary system. Second, this azeotrope should be heterogeneous so that natural liquid-liquid separation, without energy requirement, can be performed in a decanter at the top of the column.

Apart from that, Luybern and Chien (2010) further explained that depending on the number of additional azeotropes, there is a few different RCMs could be generated. One of the systems is with three additional azeotropes: one binary homogeneous azeotrope, one binary heterogeneous azeotrope, and one ternary heterogeneous azeotrope. An example is the separation of IPA and water with cyclohexane as the entrainer.

2.5.2 Extractive Distillation (Separations Using a Heavy Entrainer)

Based on the definition from the book written by Luybern and Chien (2010) (as cited in Doherty & Malone, 2001), 'extractive distillation' is a method of separating minimum boiling binary azeotropes by use of an entrainer that is the heaviest species in the mixture. It does not form any azeotropes with the original components, and is completely miscible with them in all proportions. Adding such heavy entrainer into the system alters the relative volatility of the original two components so that one original component can go overhead and the other component will go with the heavy entrainer to the column bottoms.

Vivek et. al (2009) stated that a heavy (high-boiling, low volatility) entrainer is used in an extractive distillation to separate a mixture exhibiting a minimum boiling azeotrope. The typical extractive distillation entrainer has a higher boiling point than the pure components, and it does not form any azeotropes with either of the pure components. A common industrial example of extractive distillation is the use of ethylene glycol to separate ethanol and water.

On the other hand, Luybern and Chien (2010) also mentioned that another example of the extractive distillation, which is the separation of IPA-water binary mixture using dimethyl sulfoxide (DMSO) as the heavy entrainer. In this case, IPA will go to the top of the column and water goes to the bottom of the column. Rectifying section, which is the upper section of the column (above the entrainer feed location) is used to separate IPA and the entrainer. The middle section of the column (between the entrainer feed stage and the fresh feed stage) is called extractive

section, and it is used to suppress water from going up the column. Also, the bottom section of the column (below the fresh feed location) is the stripping section. Its purpose is to keep IPA from going down the column. The bottoms product of the column is the mixture of water and entrainer.

2.5.3 Other Ways of Separating Azeotropes

There are alternatives for separating azeotropes in industry. A procedure for experimental evaluation of ionic liquids (ILs) as entrainers was conducted by Zhang, Han, Deng and Ji (2007). For selections of ILs which can be potentially used for the separation of the azeotropic mixture of water and IPA by extractive distillation, vapour-liquid equilibria were measured for ternary systems of water + IPA + IL at 100 kPa. Zhang et al. (2007) found that among the ILs studied, the analysis showed that the ILs with $[\text{OAc}]^-$ or $[\text{Cl}]^-$ as the anion, and $[\text{emim}]^+$ as the cation, have the most significant ability to enhance the relative volatility of IPA-water mixture.

Despite of the findings by Zhang et al., Luybern and Chien (2010) suggested that one method which can be categorised as extractive distillation is to use a dissolved salt (e.g. calcium chloride) as a separating agent to alter the relative volatility of the two components in the mixture. This behaviour, known as salt effect, is due to the preferential solvation of the ions (formed when the salts dissociates in solution) by the less volatile component of the solvent mixture. The bottom product of this saline extractive distillation column is the mixture of the salt and the less volatile component (e.g. water). The separation of this bottom stream may require an evaporative crystalliser and a spray dryer for recovery of the anhydrous salt.

2.6 Salt-Effect in Distillation

As mentioned in previous subsection, salt-effect distillation is a process of extractive distillation in which a salt that is soluble in the liquid phase of the system being separated is used in place of the normal liquid additive introduced to the extractive distillation column in order to affect the separation. In salt-effect distillation, the process is essentially the same as for a liquid agent, although the subsequent process used to recover the agent for recycling is different; that is, evaporation is used rather than distillation. The salt is added to the system by being dissolved in the re-entering reflux stream at the top of the column. Being non-volatile, it will reside in the liquid phase, flowing down the column and out in the bottom product stream (McGraw-Hill Concise Encyclopedia of Science and Technology, 2005, p.1942).

In the e-book entitled Perry's Chemical Engineers' Handbook (8th edition), four editors Doherty, Fidkowski, Malone and Taylor (2008) stated in the section 'Distillation' that there is another method of modifying the liquid-phase behaviour (and hence the relative volatility) of a mixture to effect a separation is by introducing a non-volatile, soluble, ionic salt. The process is analogous to extractive distillation with a high-boiling liquid. Since the salt is completely non-volatile, it still remains in the liquid phase and alters the relative volatility throughout the length of column. The ions of a salt are typically capable of causing much larger and more selective effects on liquid-phase behaviour than the molecules of a liquid solvent.

The major commercial use of salt-effect distillation is in the concentration of aqueous nitric acid, using the salt magnesium nitrate as the separating agent. Other commercial applications include acetone-methanol separation using calcium chloride and IPA-water separation using the same salt (McGraw-Hill Concise Encyclopedia of Science and Technology, 2005, p 1942).

Smallwood (2002) pointed out that an advantage of a salt over a liquid is that salt ions are able to cause larger alterations in the relative volatility than even the most polar of extraction solvents. Consequently, less separating material may be required with a corresponding reduction in energy. While it is easy to find liquid extraction solvents of the right volatility and polarity, there are fewer suitable salts with the right solubility except for solution in water.

2.6.1 How Salt Breaks Azeotropic Mixture

Kablukov (1891, 1903, 1904) reported the result of observations of the effect of salt on the partial pressures exerted over aqueous ethanol. He observed that salts soluble in water than in alcohol increases the vapour pressure of the alcohol and vice versa, variations in vapour pressure were proportional to salt concentration, and non-electrolytes seemed to have less effect on vapour pressure than electrolytes.

When a salt is dissolved in a mixed solvent consisting of two volatile, miscible liquid components, the salt may affect the activities of the two volatile components through the formation of liquid phase associations or complexes, as reviewed in Furter and Cook (1967) journal. If the dissolved salt associates

preferentially with the molecules of one component of the solvent compared with those of the other, the solubility relationship between the two volatile components is altered such that one component is 'salted out' in respect to the other. In such a case, the activities of the two volatile components of the liquid solution are altered relative to each other in a manner which results in a change of composition of the equilibrium vapour phase.

In addition, Vivek et al (2009) mentioned that an entrainer can facilitate separation of an azeotropic mixture by selectively altering the relative volatilities of the components in question, thereby effectively 'breaking' the azeotrope.

2.7 Scale Up Study

According to Zlokarnik (2006), he noted that chemical engineers have always wanted to find ways of simulating chemical processes in models in order to gain knowledge which will then assist them in designing new industrial plants. He further explained that chemical engineers faced with the same problem for another reason an industrial facility already exists but does not function properly, and suitable measurements have to be carried out in order to discover the cause of these difficulties as well as to provide a solution.

There are certain important questions that will always apply in scaling-up. Chemical engineers need to know which rules govern the adaptation of the process parameters in the model measurements to those of the full-scale plant. Also, they

need to verify the possibility of achieving complete similarity between the processes in the model and those in its full-scale counterpart. These questions touch on the theoretical fundamentals of models, these being based on dimensional analysis.

2.7.1 Design

There is a logical sequence of steps that the designer must take when performing scaling up to the bench or pilot scale as well as to the commercial scale (Bisio & Kabel, 1985). It is simplified as followed:

i. Define the system:

System definition must contain all components, including those in small concentrations that might cause problems with internal build-up or with unexpected contamination of products.

ii. Establish separation criteria

This includes the purities required, what recoveries of mixture components are to be achieved etc. The establishment of the product purity specifications strongly influences the cost of the distillation separation operation.

iii. Obtain reliable physical property data

The uncertainty of a design result is strongly dependent upon the accuracy of the physical property data which are used in the computations. These data include the important solution thermodynamics parameters related to VLEs, phase densities, viscosities, surface tensions, and diffusion coefficients.

- iv. **Select a model for the determination of theoretical stages or transfer units**
Different models are applicable to different situations, and can range from rough graphical approaches to sophisticated and rigorous analytical approaches which require large computers for obtaining the solutions.
- v. **Obtain the stages/transfer units**
The computations involved giving information on liquid and vapour flow rates in the column as well as concentration, temperature, and pressure profile data.
- vi. **Size the distillation column**
This includes the hydraulic analysis to establish operating ranges, pressure drop, and mass transfer efficiency. This is the ultimate objective of the scale up process. The column dimensions so determined must represent an optimum combination of cost, reliability, and flexibility.

2.7.2 Operation

There are two operational scaling up steps which designers need to perform, which are the introduction of salt into distillation column and reflux ratio scheme.

2.7.2.1 Introduction of Salt into Distillation Column

Review from Furter and Cook (1967) stated that applying salt effect to a system of volatile components undergoing fractional distillation would involve

introducing the salt into the descending liquid phase near or at the top of the column, normally by dissolving in the entering reflux stream. Similar findings from Doherty et al. (2008) further support the statement by stating that for the separation of a binary mixture, the salt is fed at the top of the column by dissolving it in the hot reflux stream before the introduction into the column. Doherty et al. (2008) further explained that the salt must be adequately soluble in both components throughout the range of compositions encountered in the column.

2.7.2.2 Reflux Ratio Scheme

The progress of batch distillation can be controlled in several ways (Perry & Green, 1997):

- i. Constant reflux, varying overhead composition: Reflux is set at a predetermined value that is maintained for the run. Since pot liquid composition is changing, instantaneous composition of the distillate also changes.
- ii. Constant overhead composition, varying reflux: the amount of flow rate of liquid returned to the column is constantly increased to maintain a constant distillate composition.
- iii. Other control methods: a cycling procedure can be used to set the pattern for column operation. Another possibility is to optimise the reflux in order to achieve the desired separation in a minimum of time

or with a maximum thermodynamic efficiency. Complex operations may involve withdrawal of side streams, provision for inter-condensers, addition of feed to trays and periodic charge addition to the pot.

2.8 Industrial Applications

An industrial case study in which latex manufacturer is looking at the feasibility of recycling used solvent, IPA from coagulant solution comprising IPA, water, and calcium nitrate solution. A coagulant solution, mixture alcohol (solvent) and water, is generated as a water solvent in local latex dipping product manufacturing plant. Studies have shown the effectiveness of aqueous IPA, pure deionised (DI) water, and DI water plus 0.5% of a surfactant for removing loose particles from the surface of gloves.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter discusses the materials, equipments and methods used to conduct experiments. There were two stages of methodology in this research: laboratory scale experimental work and pilot plant experimental work.

3.2 Research Design

Optimum salt concentration needed to give ‘salting out’ effect to IPA-water binary system was determined through experiments. Reflux ratio, concentration of CaCl_2 salt, and number of stages are the designed parameters. The purity of the recovered IPA is more than 90 mol%.

3.3 Research Setting

This research was conducted through experimental work in the FKKSA laboratory. A bench scale experiment was conducted where the optimum salt concentration needed to give ‘salting out’ effect to IPA-water system was being studied first. This is followed by the pilot plant experiment where the optimum salt concentration was being studied. Next, the scale up study of the bench scale to pilot scale, which includes operation parameters such as start up strategy and salt addition method, is performed. Three participants were participated in this research, which were the researcher, supervisor, and laboratory assistant.

3.4 Materials

The materials used for experiments were (i) IPA with purity > 95 mol%, (ii) water and, (iii) calcium chloride salt (CaCl_2).

3.5 Equipments

Several equipments were used in the experimental work. A laboratory scale batch distillation unit and a sieve tray distillation column as pilot plant were used. Peristaltic pump is used for salt solution addition into pilot plant. A refractometer is also used for measuring the composition of the mixture.

3.6 Procedures

Two stages of methodology: laboratory scale experimental procedures and pilot plant experimental procedures are explained as followed. Calibration curve is prepared for reference.

3.6.1 Calibration Curve

In order to distinguish the concentration as well as the mole fraction of distillate, a calibration curve (Refractive Index versus Mole Fraction) was prepared for references. The collected data was tabulated in Table 3.1 and the calibration curve was plotted as in Figure 3.1. The calibration curve will be used for determining IPA composition of the distillate in the following experiments.

Table 3.1 Refractive Index Data for IPA-Water Mixture

IPA Mole Frac, z_1	IPA Molecular Weight (g/mole)	Weight (g)	Density (g/cm ³)	Volume (cm ³)	RI
0	60.1	0.000	0.785	0.00	1.3306
0.1	60.1	1.202	0.785	1.53	1.3525
0.2	60.1	2.404	0.785	3.06	1.3613
0.3	60.1	3.606	0.785	4.59	1.3658
0.4	60.1	4.808	0.785	6.12	1.3691
0.5	60.1	6.010	0.785	7.66	1.3716
0.6	60.1	7.212	0.785	9.19	1.3727
0.7	60.1	8.414	0.785	10.72	1.3730
0.8	60.1	9.616	0.785	12.25	1.3732
0.9	60.1	10.818	0.785	13.78	1.3731
1	60.1	12.020	0.785	15.31	1.3728

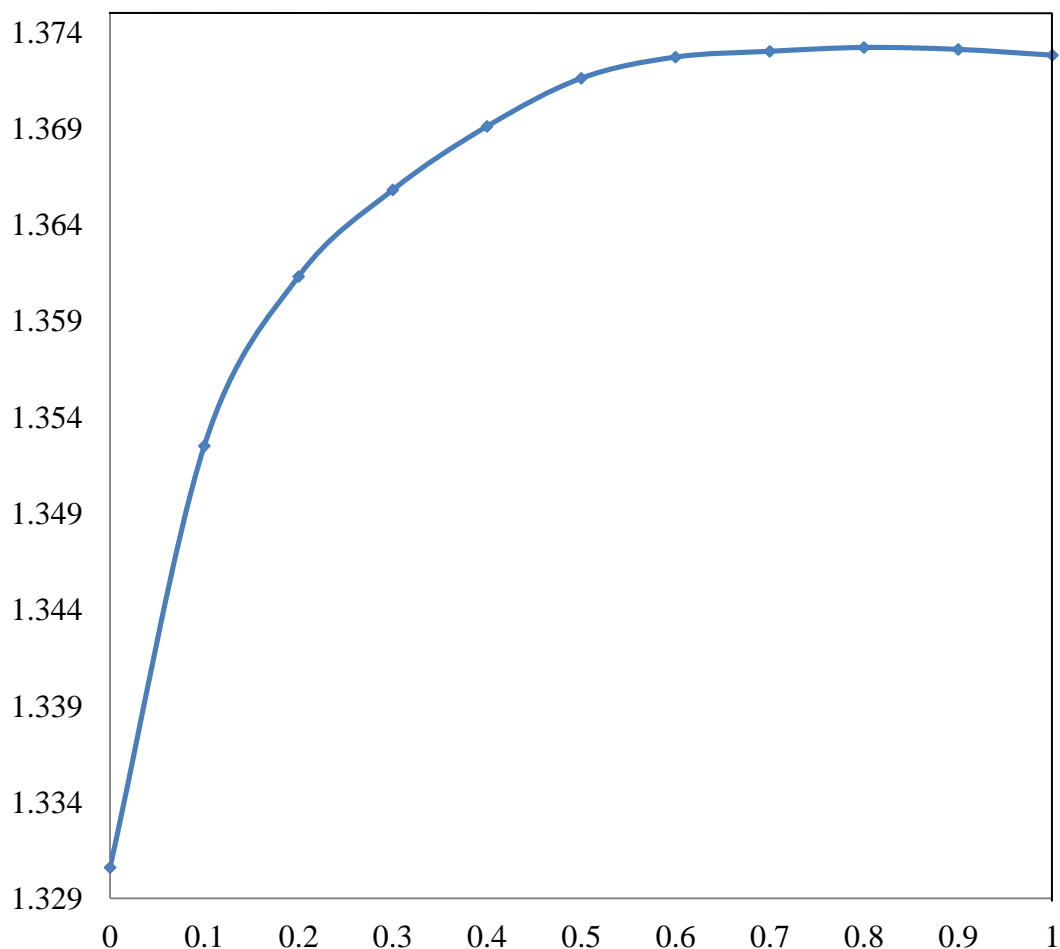


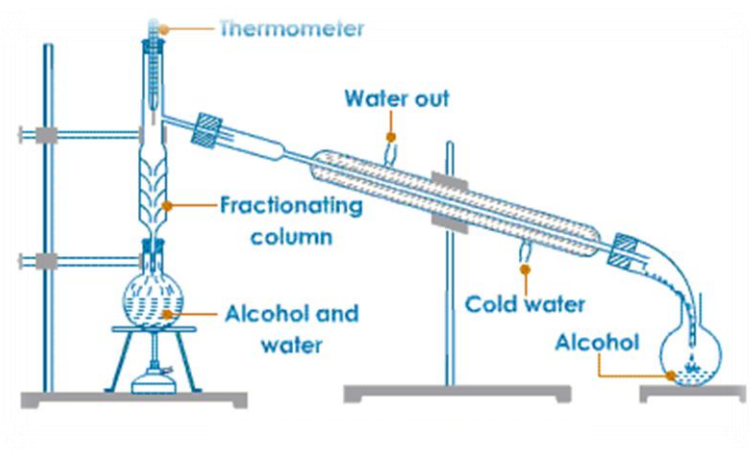
Figure 3.1 Calibration Curve for IPA-Water Mixture

3.6.2 Laboratory Scale Experimental Procedures

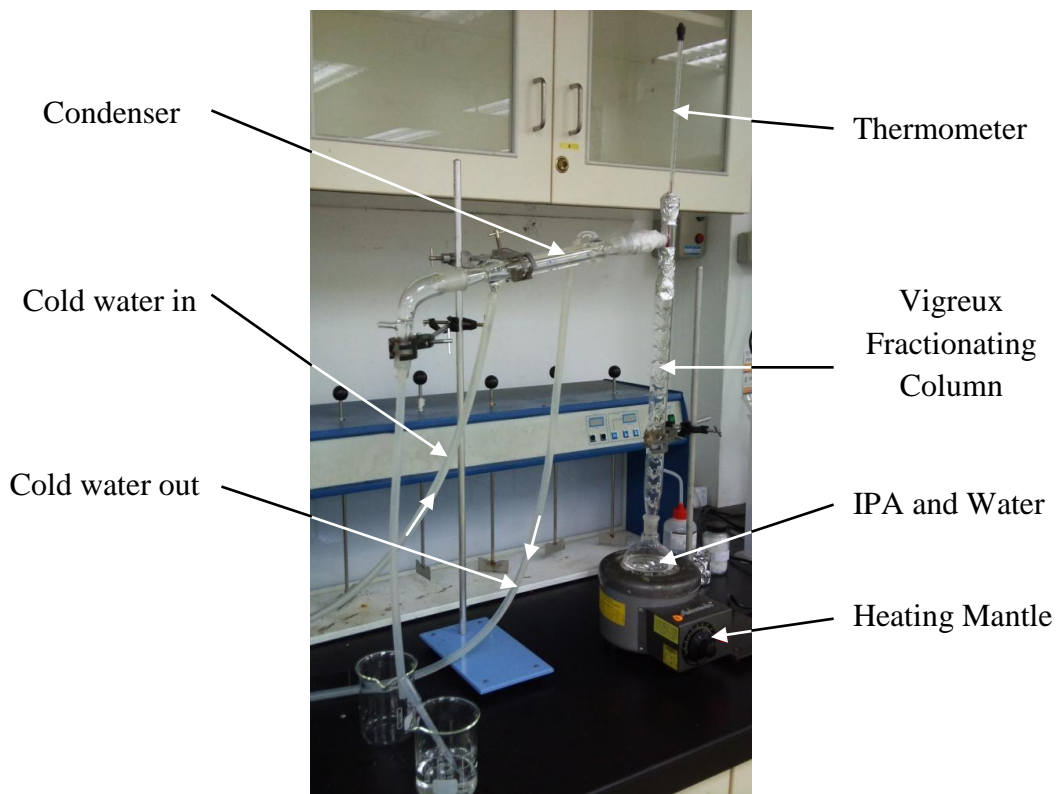
Figure 3.2 depicts a bench scale batch distillation column. The operating procedures can be summarised as followed.

i. Preliminary procedures

Before the experiment started, some preliminary procedures were taken. Firstly, IPA-water solution was prepared by mixing IPA with DI water at a composition 50 mol% to agree with a true application of a waste IPA



(a)



(b)

Figure 3.2 (a) An illustration of a laboratory scale batch distillation unit
 (b) A laboratory scale batch distillation unit used by researcher

(Source: Science E-portfolio, n.d.)

stream in the industry. Next, thermometer was checked for the position (the bulb of the thermometer must be below the arm of the distillation head) and ensured that the bottom of the distillation pot touching the heating surface of the heating mantel. After that, a piece of condenser tubing was securely attached to each condenser outlets. The other end of the “water in” tubing was securely connected to the cooling water source (or hood), while the other end of the “water out” tubing was placed in the sink (or back of the hood). The cooling water flow rate was adjusted to moderate flow rate to avoid building up pressure in the condenser wall.

ii. Experimental procedure

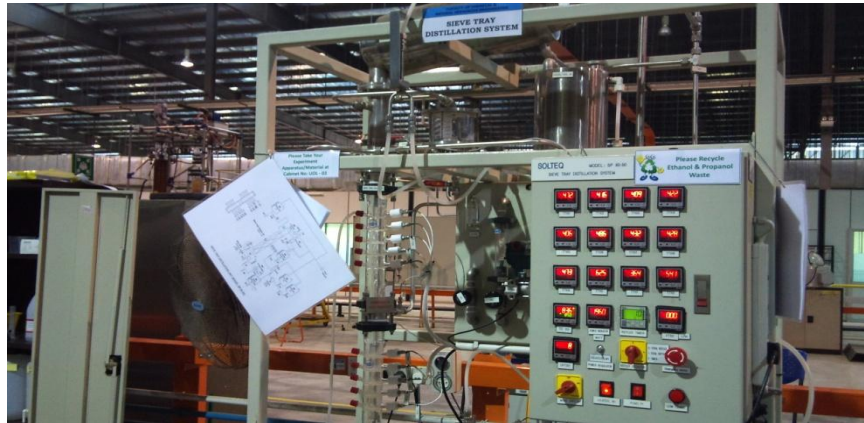
The IPA-water mixture was charged to the bottom of the column. The heating mantle was switched on, and water for cooling the condenser was opened to flow. Heat was added to the reboiler and vapour boil-up moved up the column. When distillate was collected at receiver, distillate composition was recorded by using a refractometer and system temperature was read off from thermometer every two minutes. When the distillate was collected at a slower rate or production of distillate started to cease, the experiment was ended.

iii. Trial

The experiment is repeated for other salt concentration as well as other initial IPA composition z_1 of the IPA-water mixture. The results are to be used for analysis.

3.6.3 Pilot Plant Experimental Procedures

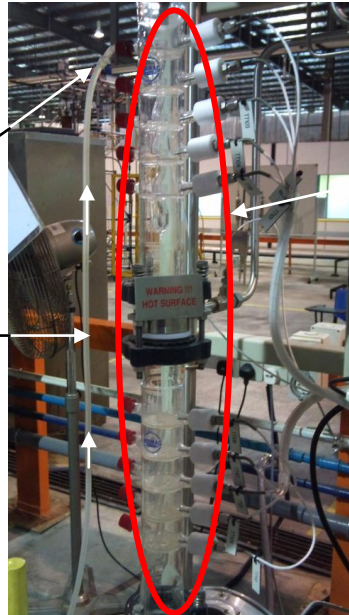
Figure 3.3 depicts a bench scale batch distillation column. The operating procedures can be summarised as followed.



(a)

Salt addition to the top of distillation column

Salt solution is added using peristaltic pump.



Distillation column (Oldershaw type)

(b)

Figure 3.3 (a) Sieve tray distillation pilot plant. (b) Distillation column (Oldershaw type)

i. Preliminary procedures

Before the experiment started, some preliminary procedures were taken. All vent lines were in the open position and all drain valves were closed. Next, the overhead condenser cooling water supply was turned on. The next step was finding composition of the initial charge by refractometer analysis. The IPA-water mixture level charged to the boiler was checked so that it was not lower than the specified level.

ii. Start up strategy and procedures

Start up was performed by turning on the control panel and heat duty was set just to provide the necessary heat needed to heat up the IPA-water- CaCl_2 mixture to a temperature right above the azeotropic point. Total reflux was used by switching the switch to 'zero' position. The column was let to settle down to uniform operation under total reflux. When the distillate was started to form and was collected, entrainer (CaCl_2 salt solution) was added continuously into the column at a flowrate of 15 mL/min using a peristaltic pump. At each five minutes, following steps were carried out: (i) thermometer reading was recorded, and (ii) sample was taken from the top product-sampling valve and sample composition was recorded using refractometer.

iii. Shut down

After the distillation ends, the distillation column unit was turned off its power supply. The distillation unit was washed with clean water to clean

the internal walls free from any salt solution residue. The condenser cooling water supply was shut when experiment was ended.

3.7 Data Collection and Analysis

Experiment data from both laboratory scale and pilot plant experiment was recorded manually when the experiments were running. Using a refractometer and calibration curve as reference, the composition of the sampled solution was analysed. Upon getting the optimum salt concentration for separating of IPA and water mixture, scaling up study was carried out.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Laboratory Experiment Result Discussion

Sada, Morisue, and Miyahara (1975) have outlined some basic theories on the VLE data for both binary and ternary systems. They proposed that mixed solvent composition in both binary and ternary system is expressed by mole fraction of IPA

$$z_1 = n_1 / (n_1 + n_2) \quad (4.1)$$

and it was held within ± 0.05 in this experimental work. In a ternary system consisting of a salt and two volatile components, there are five individual species can be found in the liquid phase: molecules of each of two volatile components, both ions of the salt and undissociated salt. The dissolved salt, however, dissociates completely to individual ions in very dilute solution. Hence, assumption is needed to be made such that one mole of salt dissociates to v_+ moles of cations and v_- moles of anions in the mixed solvent. Mole fraction of the component j is

$$x_j = n_j / (n_1 + n_2 + vn_3) \quad (j=1, 2, \text{ or } 3) \quad (4.2)$$

where

$$v = v_+ + v_- \quad (4.3)$$

On the other hand, salt will not be vapourised and exist in vapour phase due to its low volatility. In contrast, some amount of the other two volatile components (IPA and water) will gain sufficient energy upon heating and manage to escape from the liquid phase, vapourised and enter vapour phase. The vapour phase mole fraction of the solvent i is

$$y_i = n'_i / (n'_1 + n'_2) \quad (i=1 \text{ or } 2) \quad (4.4)$$

where the prime refers to the vapour phase. The vapour, upon condensation, form distillate and drip to receiving flask. Therefore, the vapour composition can be analysed by checking the distillate composition using a refractometer.

4.1.1 Result for batch distillation with no salt addition

VLE data for binary system of IPA and water (no salt addition) were shown in Table 4.1. Batch distillation of IPA-water mixture with no salt addition was carried out. Two sets of IPA-water mixtures with different initial IPA mole fractions were separated using conventional batch distillation. The result was tabulated in Table 4.1.

Table 4.1 Data for IPA-water batch distillation with no salt addition

Time (min)	$z_1 = 0.51$			$z_1 = 0.60$		
	Temperature (°C)	Refractory Index	y_1	Temperature (°C)	Refractory Index	y_1
-	26	1.3717	0.51	26	1.3727	0.60
2	78	1.3724	0.56	78	1.3731	0.71
4	78	1.3726	0.58	78	1.3731	0.71
6	78	1.3727	0.60	78	1.3731	0.71
8	78	1.3728	0.60	78	1.3731	0.71
10	78	1.3729	0.63	78	1.3731	0.71
12	78	1.3730	0.70	78	1.3731	0.71
14	78	1.3730	0.70	78	1.3731	0.71
16	78	1.3730	0.70	78	1.3731	0.71

By using two IPA –water mixture with different initial IPA composition ($z_1 = 0.51$ and 0.60), the maximum purities of IPA can be obtained in each of the two experiments via conventional batch distillation were approximately 70 mol%. ($z_1 = 0.70$). The boiling temperature of IPA-water binary system was recorded as 78 °C. These two parameters were compared with values described in previous papers.

Sada et al. (1975) had presented a vapour-liquid equilibrium curve which was calculated from the total pressure data by using numerical method developed by Mixon et al. Figure 4.1 depicts a VLE diagram for IPA-water system modified from Sada et al (1975). The actual IPA composition in vapour phase was plotted against in liquid phase. A line with $z_1 = y_1$ was drawn to represent equilibrium state in IPA compositions between liquid phase and vapour phase. The intersection point of the curve and line represents the azeotropic point of IPA-water binary system, a certain indistinguishable compositions with vapour and liquid IPA compositions of 70 mol% ($z_1 = 0.70$) at pressure 101.32 kPa and temperature 75 °C.

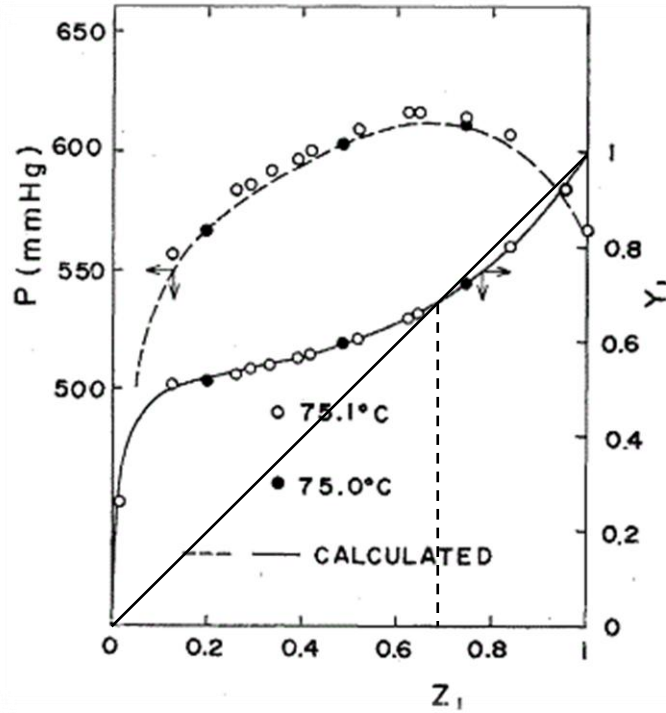


Figure 4.1 Modified Vapour-liquid equilibrium diagram for isopropanol-water system at 75 °C and 101.32 kPa

(Source: Sada et al., 1975)

On the other hand, these data were also supported by Arce, Arce Jr, Martinez-Ageitos, Rodil, & Soto (2003) where they had shown that the azeotropic point of IPA-water system falls at temperature approximately 80.3 °C and a composition of $z_1 = y_1 = 0.70$ mol%, as shown in Figure 4.2. The upper curve represents the respective IPA composition in the vapour while the lower curve represents that of in the liquid. Both the curves meet at a point wherein it is the azeotropic point of that IPA-water binary system. Consequently, the experimental result in this research shows a good agreement with the data described in previous papers done by Sada et al (1975) and Arce et al (2003), and demonstrates that apparatus used in this work is probably reliable.

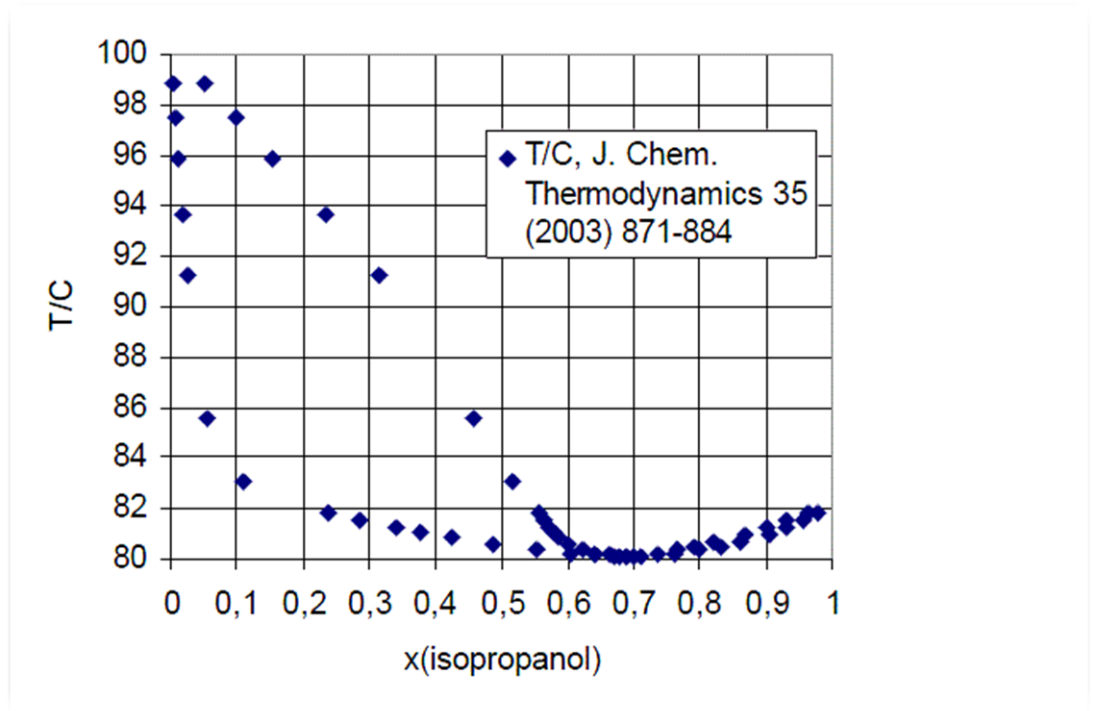


Figure 4.2 T-x-y diagram for isopropanol-water system
(Source: Arce et al., 2003)

4.1.2 Result for batch distillation with salt addition

Data for ternary systems of IPA, water and calcium chloride salt were shown in Table 4.2 and Table 4.3. Different salt concentration was used during experiment, where the salt concentrations used are 5 wt%, 10 wt% and 15 wt% of the isopropanol weight for that specific value of z_i used. Different IPA distillate compositions were obtained.

Table 4.2 Batch distillation data for IPA-water ($z_1 = 0.50$) with salt addition

Time (min)	$x_3 = 0.0130$			$x_3 = 0.0250$			$x_3 = 0.0362$		
	RI	y_1	T ($^{\circ}\text{C}$)	RI	y_1	T ($^{\circ}\text{C}$)	RI	y_1	T ($^{\circ}\text{C}$)
-	1.3714	0.450	29	1.3715	0.470	29	1.3717	0.510	29
2	1.3730	0.700	78	1.3727	0.600	78	1.3727	0.600	78
4	1.3732	0.800	79	1.3731	0.760	78	1.3730	0.700	78
6	1.3730	0.700	79	1.3732	0.800	78	1.3731	0.760	78
8	1.3728	0.610	79	1.3730	0.910	78	1.3732	0.800	78
10	1.3726	0.580	79	1.3729	0.910	78	1.3730	0.910	78
12	1.3725	0.570	79	1.3732	0.800	78	1.3732	0.800	78
14	1.3714	0.490	98	1.3730	0.700	78	1.3713	0.485	98
16	1.3369	0.024	98	1.3426	0.050	98	1.3426	0.052	98
18	1.3330	0.012	98	1.3333	0.013	98	1.3397	0.040	98
20	1.3323	0.008	98	1.3321	0.006	98	1.3324	0.010	98
22	1.3323	0.008	99	1.3321	0.006	98	1.3319	0.005	99

In Table 4.2 are shown the maximum recoverable IPA purity from initial composition of 0.50 ($z_1 = 0.50$) with different salt concentrations of 1.30 mol%, 2.50 mol% and 3.62 mol% ($x_3 = 0.0130$, 0.0250, and 0.0362 respectively). The IPA purity increases as the salt concentration increases until the maximum recoverable IPA purity of 91.0 mol% ($y_1 = 0.91$). With an initial IPA composition of 50.0 mol% ($z_1 = 0.50$), the laboratory scale work has shown that the optimum salt concentration needed to give ‘salting out’ effect to the IPA-water binary system is 2.50 mol% ($x_3 = 0.0250$), which corresponds to about 10% of the IPA weight of the system.

Table 4.3 Batch distillation data for IPA-water ($z_1 = 0.60$) with salt addition

Time (min)	$x_3 = 0.0157$			$x_3 = 0.0305$			$x_3 = 0.0444$		
	RI	y_1	T ($^{\circ}\text{C}$)	RI	y_1	T ($^{\circ}\text{C}$)	RI	y_1	T ($^{\circ}\text{C}$)
-	1.3723	0.550	30	1.3723	0.550	31	1.3727	0.600	30
2	1.3729	0.640	78	1.3727	0.600	77	1.3730	0.700	78
4	1.3730	0.700	78	1.3732	0.800	78	1.3732	0.800	78
6	1.3731	0.910	79	1.3731	0.910	78	1.3730	0.910	78
8	1.3731	0.910	79	1.3731	0.910	79	1.3730	0.910	79
10	1.3731	0.910	79	1.3731	0.910	79	1.3730	0.910	79
12	1.3730	0.700	79	1.3732	0.800	79	1.3730	0.910	79
14	1.3713	0.485	98	1.3728	0.620	98	1.3732	0.800	79
16	1.3390	0.035	98	1.3637	0.250	98	1.3691	0.400	98
18	1.3335	0.015	98	1.3398	0.040	98	1.3324	0.010	98
20	1.3325	0.010	98	1.3324	0.010	98	1.3318	0.005	99
22	1.3318	0.005	98	1.3318	0.005	98	1.3318	0.005	99

In Table 4.3 are shown the maximum recoverable IPA purity from initial composition of 60 mol% ($z_1 = 0.60$) with different salt concentrations of 1.57 mol%, 3.05 mol% and 4.44 mol% ($x_3 = 0.0157$, 0.0305, and 0.0444 respectively). Similar to previous result in Table 4.2, the purity of IPA recovered increases as the salt concentration increases until the maximum value of 91 mol%. With an initial IPA composition of 60 mol% ($z_1 = 0.60$), the laboratory scale experiment has shown that the optimum salt concentration needed to give ‘salting out’ effect to the IPA-water binary system is 1.57 mol%, which corresponds to about 5% of the IPA weight of the system.

From these data, it can be seen that as the initial IPA composition increases, the salt concentration needed to be used to give the ‘salting out; effect to the IPA-water system is decreasing. This trend can be explained by the previous work performed by Sada et al (1975). By referring Table 4.4, for three different initial IPA

Table 4.4 VLE data for IPA-water-CaCl₂ system at 75.1 °C

z_1	x_3	y_1	P [mmHg]	$\ln \gamma_1'$	$\ln \gamma_2$	$\frac{\Delta\mu}{RT}$
0.126	0	0.504	557.2	1.370	0.076	0
0.126	0.010	0.541	578.8	1.501	0.020	0.147
0.126	0.030	0.616	599.0	1.706	-0.047	0.455
0.125*	0.050	0.695	605.0	1.879	-0.224	0.809
0.125*	0.069	0.770	599.0	2.026	-0.474	1.190
0.125*	0.099	0.858	570.3	2.155	-0.932	1.782
0.124*	0.127**	0.910	530.1	2.229	-1.399	2.299
0.260	0	0.530	584.3	0.741	0.237	0
0.260	0.010	0.569	596.0	0.855	0.188	0.158
0.259*	0.030	0.651	607.4	1.051	-0.037	0.502
0.259*	0.050	0.729	606.0	1.204	-0.175	0.869
0.259*	0.070	0.802	592.8	1.372	-0.466	1.279
0.259*	0.100	0.881	554.8	1.428	-0.965	1.882
0.258*	0.115**	0.910	529.1	1.459	-1.257	2.196
0.333	0	0.550	592.1	0.543	0.309	0
0.333	0.010	0.594	602.0	0.657	0.243	0.180
0.332*	0.030	0.677	607.5	0.841	0.064	0.538
0.331*	0.050	0.754	602.1	0.989	-0.177	0.919
0.330*	0.070	0.823	584.8	1.098	-0.490	1.330
0.330*	0.100	0.894	543.7	1.180	-1.001	1.933
0.330*	0.109**	0.910	529.7	1.196	-1.168	2.116

* immiscible region, ** saturated value

(Source: Sada et al., 1975)

composition system, the salt mole fraction and hence the salt concentration needed to recover the IPA for purity more than 90 mol% is lower. As values of z_1 increase from 0.124 to 0.258 and 0.330, x_3 show a decreasing trend from value of 0.127 to 0.115 and 0.109, respectively, to obtain a y_1 value of 0.910. By plotting the data from experiment as well as the data obtained from journal, it can be used for prediction of optimum salt concentration for other initial IPA compositions, as shown in Figure 4.3.

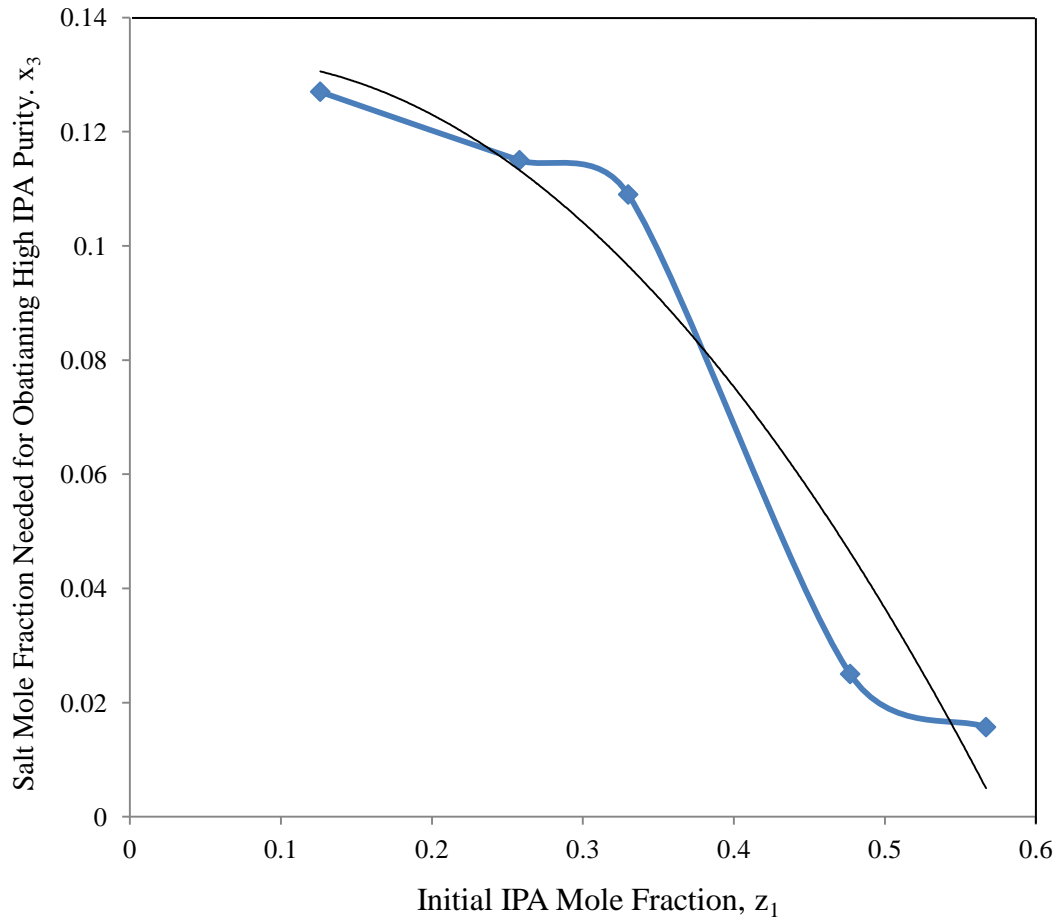


Figure 4.3 Graph of x_3 versus z_1

4.2 Pilot Plant Experiment Result

In Table 4.5 are shown pilot plant experimental result. The heat duty was set just to provide the necessary heat needed to heat up the IPA-water- CaCl_2 mixture to a temperature right above the azeotropic point. At time $T=55$ and 60 minutes, the refractory index shows a constant value. Within the specified parameters, this phenomenon indicates that the maximum recoverable IPA purity reaches a refractory index value of 1.3728, which equals to a IPA composition of 62 mol%.

Table 4.5 Pilot plant experiment result

Time, T (min)	Refractory Index	Mole Fraction	Temperature (°C)
-	1.3527	0.100	31
5	1.3569	0.138	78
10	1.3592	0.168	78
15	1.3667	0.325	78
20	1.3687	0.385	78
25	1.3701	0.435	79
30	1.3715	0.500	79
35	1.3718	0.505	79
40	1.3722	0.550	79
45	1.3725	0.580	79
50	1.3728	0.620	79
55	1.3728	0.620	79
60	1.3728	0.620	79

The highest recoverable composition of IPA is only about 0.620. It is clearly shown that experiment run by pilot plant has failed to recover the IPA for purity more than 90 mol%. The explanation of this phenomenon is discussed on the next subdivision.

4.3 Comparison between laboratory scale and pilot plant experiments

There are differences between the laboratory scale and pilot plant scale experiments from the perspective of operating procedures and outcome.

4.3.1 Operating procedures

4.3.1.1 Salt addition method

In laboratory scale experiment, calcium chloride salt was just added simply by dissolving it in the still. However, salt addition in pilot plant was done by feeding it at the top of the column. Addition of salt by feeding at the top of distillation column can maximize the contact between the descending flow of salt solution and the IPA-water binary mixture in each sieve tray. In contrast, this salt addition method is hard to be done in the laboratory scale as there is no additional inlet mouth on the Vigreux fractionating column. Hence, the salt solution is rather simply added in the still rather than feeding it at the top.

4.3.1.2 Reflux ratio

In laboratory scale experiment, reflux ratio cannot be manipulated as the apparatus used are rather simple and straightforward. IPA-water mixture was boiled continuously in the distillation flask. On the other hand, total reflux ratio was used pilot plant experiments, where the column is operated with the entire condensed overhead stream returned to column as reflux (Perry & Green, 2008). All the liquid reaching the reboiler was again vapourised and returned to the column as vapour. This repeating process enhanced the heat and mass transfer processes, thereby improving the separation process and recovering a higher purity IPA. The changes of instantaneous composition of the distillate in both the experiments are the explanation of the reflux ratio scheme applied.

4.3.2 Experimental Outcomes

Results from lab scale experiment showed that addition of calcium chloride salt to the azeotropic IPA-water binary system has successfully recovered the IPA for purity of 91 mol %. This result has supported by the previous work by Sada et al. (1975). However, the separation of IPA-water binary system with salt addition was not achieved with a maximum recoverable IPA purity of 62 mol %, which further explained that the system has not reach the azeotropic point.

Bisio and Kabel (1985) have suggested that there are uncertainties in the distillation process even though the methodology for scale up and design of such equipment is straightforward and well established. They had pinpointed that errors and inaccuracies can arise in the equilibrium data from problems in the laboratory as well as failure in determining thermodynamic consistency of the results. Nevertheless, error also can be arisen in the extension of binary data to multi-component systems, which is a common obstacle that pure component properties do not relate as predicted to properties of the same components in solution with other materials. These errors can cause large changes when close-boiling mixtures (IPA and water) are being considered.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The optimum salt concentration needed to break the azeotropic point of IPA-water mixture as well as the scaling up study of salt effect in batch distillation from laboratory scale to pilot plant scale were examined during the period of study. The optimum salt concentration needed is generally decreasing as z_1 is higher. For IPA-water system with value of $z_1 = 0.50$, 2.5 mol% ($x_3 = 0.025$) of calcium chloride salt was needed for distilling IPA-water mixture to recover 91 mol% of IPA. Similarly, 1.57 mol% ($x_3=0.0157$) of calcium chloride salt was required to recover 91 mol% of IPA from a IPA-water system with $z_1 = 0.60$.

Differences in experiments conducted at laboratory scale and pilot plant scale were accessed. In laboratory scale experiment, salt solution was added directly to the still before distillation and no reflux ratio can be manipulated due to simpler apparatus. However, salt solution was added near to the top of column and total

reflux ratio was applied to provide better mass and heat transfer in salt effect distillation.

Using laboratory scale apparatus, a high purity of 91 mol% of IPA was recovered shows that IPA-water azeotropic point is successfully broken down by calcium chloride salt addition. The salt solution added to distillation column of pilot plant, however, did not help in recovering high purity of IPA, which can be explained by errors in the extension of binary data to multi-component systems.

5.2 Recommendations

5.2.1 Increase Number of Parameters to be studied

Besides studying on the optimum salt concentration, parameters such reflux ratio, number of stages in distillation column, and are possible alternatives that can be added on to enhance and widen the study.

5.2.2 Change the Type of Distillation Column Used

Other than sieve tray distillation unit, optional distillation units such as bubble cap distillation column unit and batch distillation column unit with reflux ratio can be chosen to perform the pilot plant study. Reflux ratio is one of the major factors that can provide effective separation in distillation. Hence, batch distillation

column unit with a reflux ratio controller can be used to study the effect of reflux ratio on separating and recovering high purity IPA from IPA-water binary system.

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APPENDIX A

A SCHEMATIC DIAGRAM OF SIEVE TRAY DISTILLATION COLUMN

