EFFECTS OF SALT CONCENTRATION ON VAPOR - LIQUID EQUILIBRIUM (VLE) OF AZEOTROPIC MIXTURE IN ULTRASONIC DISTILLATION SYSTEM

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NOR FAIZAH BINTI RAZALI

A thesis submitted as fulfillment of the award of the degree of Master of Engineering (Chemical)

Faculty of Chemical and Natural Resources Engineering
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NOVEMBER 2008

"I declare that this thesis entitled "**EFFECTS OF SALT CONCENTRATION ON VAPOR-LIQUID EQUILIBRIUM (VLE) OF AZEOTROPIC MIXTURE IN ULTRASONIC DISTILLATION SYSTEM**" is the result of my own work except cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any degree."

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Specially dedicated to

my beloved father, Razali bin Ismail, my beloved mother, Rujomah bte Jamil

and

those people who have guided and inspired me throughout my journey of education

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ABSTRACT

Methanol and methyl acetate form an azeotrope in their mixtures at 34.78 mole % of methanol. It is difficult and may be impossible to separate azeotropic mixture using conventional distillation column. This phenomenon gives big challenges in the chemical industry in order to solve it. In this study, potassium chloride was added in ultrasonic distillation system to study the combination effect of salt and ultrasonic wave on methanol-methyl acetate mixture. The salt was added with different range of concentration to select the optimum concentration that can eliminate the azeotropic point. Ultrasonic wave with frequency of 25 kHz and intensity of 200 $W/A.cm^2$ were used. The studies on the effect of different salt concentration at 0 wt%, 5 wt%, 10 wt% and 15 wt% to VLE of binary mixtures were done at that frequency and intensity to obtain the best salt concentration. The results obtained show that, as the salt concentration increased in the liquid phase, the equilibrium line shifts upwards and in the same time, the azeotropic point also move upward. The salt concentration used in this work give the results in the following order 15 wt% >10 wt% > 5 wt% > 0 wt% where the azeotropes point form at 70 mole %, 54 mole %, 48 mole % and 38 mole % of methanol accordingly. As the result, the best concentration of the potassium chloride for the methanol-methyl acetate separation in this project was at 15 wt% of concentration. These results show that the combination of ultrasonic and salt as a separating agent gave positive results and have a potential to be apply for industry in the future.

ABSTRAK

Campuran metanol dan metil acetat membentuk azeotrop pada titik 34.78 mol % dari metanol. Sebatian azeotrop ini merupakan campuran yang agak sukar dipisahkan dan mungkin tidak boleh dipisahkan oleh penyulingan biasa. Hal ini membuatkan industri kimia pada masa kini menghadapi cabaran yang agak besar untuk mengatasi masalah ini. Kajian ini memperkenalkan kaedah baru dalam percubaan untuk memisahkan campuran azeotrop ini, dimana garam digunakan sebagai agen pemisahan dalam sistem penyulingan ultrabunyi. Garam yang digunakan adalah kalium klorida, dimana garam ini di campurkan ke dalam sebatian metanol-metil acetat, dalam julat kepekatan yang berbeza agar nilai optimum kepekatan garam dapat diperoleh. Frekuensi gelombang ultrabunyi yang digunakan adalah 25 kHz, manakala keamatan ultrabunyi yang dibekalkan adalah pada 200 $W/A.cm^2$. Julat kepekatan garam yang digunakan adalah sebanyak 0 wt%, 5 wt%, 10 wt% dan 15 wt%. Keputusan yang diperoleh menunjukkan kesan yang agak baik apabila lengkungan keseimbangan metanol-metil acetat berganjak menjauhi garisan 45°C dan seterusnya melonjakkan nilai titik azeotrop. Hasil yang diperoleh adalah mengikut urutan 15 wt% >10 wt% > 5 wt% > 0 wt% dimana titik azeotrop yang diperoleh adalah pada 70 mol %, 54 mol %, 48 mol % dan 38 mol % dari metanol mengikut urutan. Kesimpulannya, kepekatan garam kalium klorida yang terbaik dalam projek ini adalah pada kepekatan 15 wt %. Keputusan ini menunjukkan kaedah yang diperkenalkan ini memberi hasil yang positif. Ini memungkinkan kaedah ini untuk diteruskan kajiannya, seterusnya diaplikasikan dalam industri pada masa hadapan.

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LIST OF SYMBOLS AND ABBREVIATION

VLE	-	Vapor-Liquid Equilibrium
m	-	Meter
°C	-	Degree Celsius
ml	-	Mill liter
g	-	Gram
KCl	-	Potassium Chloride
%	-	Percentage

CHAPTER I

INRODUCTION

1.1 Introduction

Distillation is the most widely applied separation technology and will continue as important process in the future because there is simply no industrially viable alternative around. Eventhough this technique confronted challenges from other technologies, it still improves from time to time and moves to a higher level of sophistication. Nevertheless, there are still many technical barriers faced by distillation. Not all liquid mixture can be separated by ordinary fractional distillation. The separation becomes difficult and expensive when the components of the system have low relative volatilities ($1.00 < \alpha < 1.05$). This is because a large numbers of trays are required and, usually, a high reflux ratio as well. A different problem occurs if the system forms azeotropes, where the azeotropic composition limits the separation (Cheresources.com website, 2008).

Azeotrope means literally that the vapor boiling from a liquid has the same composition as the liquid. The azeotropic mixture depends upon the degree of non-ideality of a mixture and the difference in boiling points between the two pure components (Kim et al., 1997).

It has been known that the separation of components by simple distillation is impossible if the mixture exhibits an azeotropic at a specified temperature and pressure. For a mixture that has an azeotrope, the separation process relies on the addition of specially chosen chemicals to eliminate the azeotropes entirely. Salts are one of the mass-separating agents that can be introduced in distillation system in order to solve this problem (Banat et al., 1997).

A salt dissolved in a mixture of volatile components may affect the activities of the components through the formation of complexes (Yao et al., 1999). The salt will dissociates into ions in the liquid mixture and alters the relative volatilities to make the separation becomes possible (Banat et al., 1997).

Normally, even little salt may bring an appreciable effect on the relative volatility where this is called the effect of preferential salvation. The use of a salt instead of another separating agent in distillation gives several advantages including lower energy consumption. It also gives high purity of the overhead products because salts are non-volatile and hence do not evaporate or condense during distillation process (Banat et al., 1997). Salts also has lower toxicity lever comparing with other liquid separation agents such as benzene.

The previous research has verified the feasibility of using ultrasonic wave to enhance the separation of binary mixtures in distillation column and overcoming the challenges of azeotropic separation. In this project, salt was used to be tested in ultrasonic distillation system. The salt was introduced in the flask with different concentration. Once the optimum salt concentration was achieved, further analysis can be done by calculate its relative volatility.

This research was about to see the effect of salts in ultrasonic distillation system in order to enhance the separation process. The selected binary mixture was methanol and methyl acetate, while the salt used was potassium chloride (KCl).

1.2 Problem statement

Distillation is the common separating method to separate components in liquid mixtures. However, this process may be complicated by the formation of azeotropes due to non idealities in the mixture. These azeotropes can make a given separation impossible by conventional distillation processes. Industries always looking forward to obtain the solution for this problem.

Salt is one of the separating agents in distillation process for separating close-boiling or azeotropes systems that cannot easily be purified using ordinary distillation. Salt has been proved for eliminating the azeotropes entirely. In this work, vapor-liquid equilibrium (VLE) studies were conducted to determine the optimum concentration of selected salt in ultrasonic distillation system.

1.3 Objective

The main objective of this research is to study the separation of azeotropic mixture by using salt on VLE in ultrasonic distillation system.

1.4 Scope of Research

The scope of this research was to identify the appropriate salt concentration to be applied in ultrasonic distillation system.

CHAPTER II

LITERATURE REVIEW

2.1 Distillation

Distillation is a method of separating mixtures where a liquid or vapor mixture of two or more substances is separated into its component fractions of desired purity, by the application and removal of heat. The separation of mixture is based on differences in their volatilities in a boiling liquid mixture (Fair, 2000). Distillation is the most common separation technique and it consumes enormous amounts of energy, both in terms of cooling and heating requirements. It can contribute to more than 50% of plant operating costs. The best way to reduce operating costs of existing unit is to improve their efficiency and operation via process optimization and control (Distillation website, 2008).

Distillation exists either in batch or continuous mode. In batch distillation, the composition of the source material, the vapors of the distilling compounds and the distillate change during the distillation. In batch distillation, a still is charged (supplied) with a batch of feed mixture, which is then separated into its component fractions which are collected sequentially from most volatile to less volatile, with the bottoms (remaining least or non-volatile fraction) removed at the end. The still can then be recharged and the process repeated. In continuous distillation, the source materials, vapors, and distillate are kept at a constant composition by carefully adding the source material and removing fractions from both vapor and liquid in the system. This results in a better control of the separation process (Wikipedia website, 2008).

2.2 Vapour-Liquid-Equilibrium (VLE)

2.2.1 Introduction

VLE measurements are tedious and time-consuming because measurement conditions are often controlled and recorded manually. Cost reduction can be achieved by affordable automation, which permits a more efficient operation of the apparatus and, in some cases, an increase in accuracy. One problem associated with automation is that researchers working with experimental thermodynamics seldom seem to have the expertise needed in laboratory automation (Ussi-Kyyny, 2004).

However, when automation expertise has been successfully created in the laboratory, the goal should be to implement data acquisition programs and automation software to increase the measurement output of the experimental devices. It is thereby possible to decrease the cost of one individual measurement point substantially. Suitable methods for determination of VLE vary. In some cases several methods can be applied, but in the most difficult cases measurements are almost impossible. The selection of methods and apparatuses depend on the physical properties of the system studied such as vapor pressure, component stability, material compatibility, measurement accuracy and safety. The properties determined specifically for binary vapor liquid equilibrium systems are temperature, pressure and the compositions of the constituent phases (Ussi-Kyyny, 2004).

The determination of composition is the most complex task.The devices needed are often expensive and there is no universal analytical device that is suitable for all components. Gas chromatography is used most often for the determination of the composition of phases. Other methods for composition determination, although seldom applied in VLE measurements, include mass spectrometry, various spectroscopic methods, and density and refractive measurement (Ussi-Kyyny, 2004).

2.2.2 Vapour-Liquid-Equilibrium (VLE) Curves

Constant pressure VLE data is obtained from boiling point diagrams. Figure 2.1 shows the plot that often presented for VLE data of binary mixtures. The VLE plot expresses the bubble-point and the dew-point of a binary mixture at constant pressure. The curved line is called the equilibrium line and describes the compositions of the liquid and vapor in equilibrium at some fixed pressure. This particular VLE plot shows a binary mixture that has a uniform vapor-liquid equilibrium that is relatively easy to separate (Distillation website, 2008).

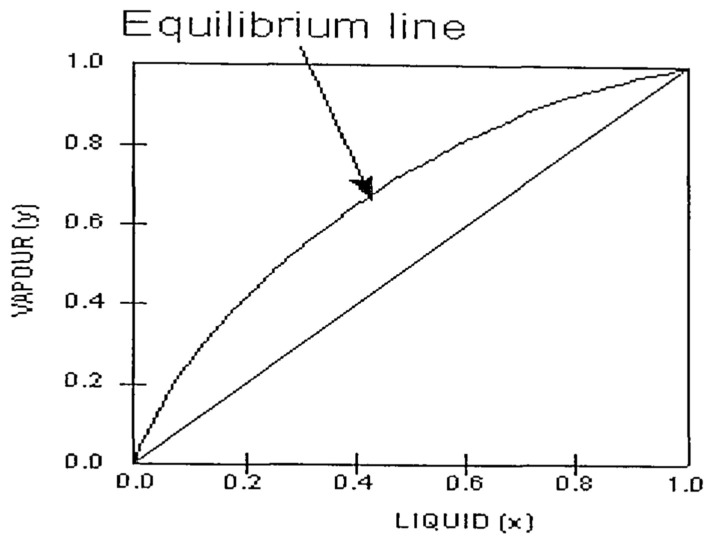

Figure 2.1: VLE graph of binary mixtures

2.3 Azeotropic Formation in Binary Mixtures

The most intriguing VLE curves are generated by azeotropic systems. An azeotrope is a liquid mixture which when vaporised, produces the same composition as the liquid. Figure 2.2 shows two different azeotropic systems, one with a minimum boiling point and one with a maximum boiling point. In both plots, the equilibrium curves cross the diagonal lines, and this are azeotropic points where the azeotropes occur. In other words azeotropic systems give rise to VLE plots where the equilibrium curves crosses the diagonals (Distillation website, 2008).