Design of A Liquid – Liquid Extraction Column

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Date of examination: 06/10/2012
بسم الله الرحمن الرحيم

قال تعالى: ((فَتَعالَيْنِ اللَّهُ الْمَلِكُ الْحَقُّ وَلَا تَعالَيْنِ بِالْقُرْآنِ مِنْ قَبْلِ أَنْ يُقصَى إِلَيْكَ وَحِيْهُ وَقَلْ رَبِّ زَدْنِي عَلَمًا)).

صدق الله العظيم

سورة طه (114)
I would like to dedicate this work to the members of my family; my parents were instrumental in imparting me the appropriate education and most of all to the Almighty Allah who gave me strength and good health while conducting this research.
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I thank Allah for all his so many blessings and for making me a better person. Special thanks to the person who is there for me and he was more than supervisor, thank you from the bottom of my heart Dr. Gurashi Abdallah Gasmelseed for your unlimited encouragement and support. I want to tell my parents and all my family that no words can describe my gratitude and my love for them. Thank you and God bless you all. Many thanks to members and Staff at University of Gezira.
Abstract

Liquid-liquid extraction is one of the simplest and cost effective separation process which is well known and well investigated, however, the equilibrium data for mutual solubility and tie-lines are difficult to correlate and be applied. Many works introduced methods that predict liquid – liquid tie-line data, but the mutual solubility data is still plotted on binodal curve on equilateral triangle. In this work the extraction of acetic acid from a mixture of acetic acid and water was carried out by using isopropyl ether to design a liquid – liquid extraction column (sieve Tray). The equilibrium data of water, acetic acid and isopropyl ether ternary systems were determined at 25°C and 1 atm; and plotted on binodal curve on equilateral triangle. The construction of the tie-lines on the binodal curve to determine the number of theoretical stages was done graphically using the relevant correlations. This required experimental determination of the mutual solubility and tie-line data; the graphical method derived by Treybal is used to obtain the number of theoretical stages in the present work and proved to be accurate and easy to apply. The number of stages was determined using this method as well as all other design parameters of a sieve tray extraction column. Aspen plus simulation package was used to calculate the number of theoretical stages; the design is also made through hand calculations for a complete design of extraction column; the stages were compared with manual calculations, and it is found to be in agreement with the method investigated in this study.
تصميم برج استخلاص سائل - سائل
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ماجستير العلوم في الهندسة الكيميائية،أكتوبر 2012
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كلية الهندسة والتكنولوجيا
جامعة الجزيرة

ملخص الدراسة

عملية استخلاص السوائل بالمذيبات السائلة من عمليات الفصل المعروفة والمؤثرة اقتصادياً، وقد تم بحثها جيداً بالرغم من أن قابلية الذوبان المشتركة وخطوط الربط يصعب تطبيقها. كثير من العلماء بحثوا في هذا المجال حيث قدموا طرقًا للتنبؤ ببيانات خطوط الربط وقابلية الذوبان المشتركة التي ترسم علي منحنى عقد الانتزان الثنائي في مثلث متساوي الاضلاع. ففي هذه الدراسة تم استخلاص حامض الخليك من مزيج الماء وحمض الخليك باستخدام ايسوبروبيل إيثانول لتصميم برج لاستخلاص سائل من سائل (صينية المناخل)، حيث يتطلب تحديد بيانات الإنزال والتي تم تحديدها عند درجة حرارة 25 °C وضغط جوي 1 ATM تحديدها علي منحنى عقد الانتزان الثنائي علي المخطط الثلاثي القطاعات، وتم بناء خطوط الربط علي منحنى عقد الانتزان الثنائي لتحديد عدد المراحل النظرية والذي يجب أن يرسم بيانياً باستخدام خطوط الربط وهذا يتطلب إجراء تجارب لتحديد قابلية الذوبان المشتركة وخطوط الربط. الطريقة البيانية والتي ابتدعها العالم تريبال Telephone Treybal والذى عرض طريقة لتحديد عدد المراحل النظرية بدون استخدام بيانات خطوط الربط علي منحنى عقد الانتزان الثنائي تم استخدامها في هذا البحث وثبت أن هذه الطريقة صحيحة وسريعة ودقيقة وسهلة التطبيق ولا تتطلب القيام بتجارب لتحديد خطوط الربط. تم استخدام برنامج Aspen Plus لحساب عدد المراحل النظرية كما تم حساب عدد المراحل يدوياً ولقد تم التوصل إلى إجراء تصميم كاملاً لحساب سائل يدويًا وأليًا وتم مقارنتها والذي أدى إلى نتائج مطابقة مع الطريقة المثبتة في هذا البحث، وعليه فإن برنامج Aspen plus software يمكن استخدامه بوتوق في تصميم ابراج الاستخلاص.
**NOMENCLATURE**

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Chapter One
Introduction

1.1 Liquid – Liquid Extraction

One of the most frequently occurring problems in the field of chemical engineering is the separation of the components of a liquid solution; an industry has made this field an active area of research in the last decades. The separation of the components of a liquid mixture by treatment with a solvent in which one or more of the desired components is preferentially soluble is known as liquid – liquid extraction (LLE), also called solvent extraction (SX) which is a process that allows the separation of two or more components due to their unequal relative solubility’s in two different immiscible liquids, usually water and organic solvent. It is an extraction of a substance from one liquid phase into another liquid phase\(^1\).

In the operation, it is essential that the liquid - mixture feed and solvent are at least partially if not completely immiscible and, in essence, three stages are involved:
(a) Bringing the feed mixture and the solvent into intimate contact,
(b) Separation of the resulting two phases, and
(c) Removal and recovery of the solvent from each phase\(^2\).

The design of physical processes almost includes design of separation operations; the most common of these is distillation column.

Normally distillation is the most efficient method of separating a mixture into its constituents in the petroleum and chemical industries. It is the separation of key components in a mixture by the difference in their relative volatility, or boiling points. It is also known as fractional distillation or fractionation. In most cases, distillation is the most economical separating method for liquid mixtures. However, it can be energy intensive\(^3\).

The design of extraction equipment depends upon knowledge of the solubility of a solute between two solvents that are not completely miscible with each other. The simplest separation by extraction involves two components and a solvent –
ternary system. Equilibria in such cases are represented conveniently on triangular diagrams\textsuperscript{[4]}.

The graphical methods are still used to represent equilibrium data and perform extraction calculations for ternary systems.

Further, quaternary and higher multi–component mixtures are often encountered in liquid–liquid extraction processes, but there is no compact graphical way of representing their phase equilibria\textsuperscript{[5]}.

Designers are required to achieve the desired product quality at minimum cost and also to provide constant purity of product even though there may be variations in feed composition. A distillation unit should be considered together with its associated control\textsuperscript{[6]}.

Sieve plate or perforated plate extractor are often used for these operation and have found an increasingly wide range of application in industry\textsuperscript{[7]}.

In the classification of non-mechanically agitated contactors, the sieve tray extractor has an important role due to the relatively high throughputs, the moderate efficiency, and the simplicity of construction and operation, which is similar to the well-known sieve tray distillation column\textsuperscript{[8]}.

In industry complicated problems are often not solved by hand for two reasons: human errors and time constraints. There are many different simulation programs used in industry depending on the field, application, and desired simulation products (entire process unit, one piece of equipment, etc.). When used to its full capabilities, Aspen can be a very powerful tool for a chemical engineer in a variety fields including extraction processes\textsuperscript{[9]}. 
1.2 Objectives of research

1.2.1 To design a liquid – liquid extraction column.
1.2.2 To investigate the column performance and hydrodynamics at various conditions.
1.2.3 To compare the results calculated by software using Aspen plus against that obtained by manual calculations.
Chapter Two
Literature Review

2.1 Extractive distillation

is defined as distillation in the presence of a miscible, high boiling, relatively non-volatile component, the solvent, that forms no azeotrope with the other components in the mixture. The method is used for mixtures having a low value of relative volatility, nearing unity. Such mixtures cannot be separated by simple distillation, because the volatility of the two components in the mixture is nearly the same, causing them to evaporate at nearly the same temperature at a similar rate, making normal distillation impractical. The method of extractive distillation uses a separation solvent, which is generally non-volatile, has a high boiling point and is miscible with the mixture, but doesn't form an azeotropic mixture. The solvent interacts differently with the components of the mixture thereby causing their relative volatilities to change. This enables the new three-part mixture to be separated by normal distillation. The original component with the greatest volatility separates out as the top product. The bottom product consists of a mixture of the solvent and the other component, which can again be separated easily because the solvent does not form an azeotrope with it. The bottom product can be separated by any of the methods available. It is important to select a suitable separation solvent for this type of distillation. The solvent must alter the relative volatility by a wide enough margin for a successful result\textsuperscript{10}.

2.2 Liquid–liquid extraction:

Liquid – Liquid extraction is a mass transfer operation in which a liquid solution (the feed) is contacted with an immiscible or nearly immiscible liquid (solvent) that exhibits preferential affinity or selectivity towards one or more of the components in the feed. Two streams result from this contact:
The extract, which is the solvent rich solution containing the desired extracted solute, and the raffinate, the residual feed solution containing little solute\cite{11}.

### 2.3 Extraction process:

Extraction is a process that separates components of a liquid mixture by contacting of a solution with another solvent that is immiscible with the original. The solvent is also soluble with a specific solute contained in the solution. Two phases are formed after the addition of the solvent, due to the differences in densities. The solvent is chosen so that the solute in the solution has more affinity toward the added solvent. Therefore mass transfer of the solute from the solution to the solvent occurs. Further separation of the extracted solute and the solvent will be necessary. However, these separation costs may be desirable in contrast to distillation and other separation processes for situations where extraction is applicable\cite{12}.

![Figure (2.1): A general extraction column](image)

A general extraction column has two input streams and two output streams. The input streams consist of a solution feed at the top containing the
solute to be extracted and a solvent feed at the bottom which extracts the solute from the solution. The solvent containing the extracted solute leaves the top of the column and it referred to as the extract stream. The solution exits from the bottom of the column containing only small amounts of solute and it is known as the raffinate. Further separation of the output streams may be required through other separation processes\textsuperscript{[11]}.

### 2.4 Column Hydrodynamics:

#### 2.4.1 Flooding in sieve extractor:

Occur when the flow rate of dispersed phase is prevented from flowing through the column and dragged out by the flow rate of continuous phase. Flooding can also arise if the flocculation zone expands to fill the stage. Correlation to predict the flooding velocities in sieve tray extractors seem important because they could permit us to fix the proper flow to good mass-transfer efficiency and also to estimate the column diameter. Flooding in tray tower of distillation column can occur or observed in either of following ways\textsuperscript{[16]}:

#### 2.4.1.1 Jet Flood:

In distillation operation froth of liquid- vapour mixture forms on each tray from which nearly clear vapour is separated and rises upward to meet the liquid on the next above tray. When froth of liquid-vapour mixture touches the next above tray it is called jet flooding. Actually the vapour flows through perforations of tray forms a free flowing jet after leaving the orifice. Liquid droplets are entrained in these free flowing jets. These free flowing jets combined from the froth. When this froth touches the next tray above, it is called jet flooding.

#### 2.4.1.2 Down comer Flooding:

In distillation column, liquid flows in downward direction by gravitational force but it flows against the pressure. When liquid flows from one tray to next below
tray, it flows from lower to higher pressure, hence, to compensate that, it elevates certain level inside the weir; it is called down comer flooding

2.4.2 Entrainment:

The use of the word entrainment is most often refers to the movement of one fluid by another.

2.4.3 Phase inversion:

Phase inversion is the phenomenon whereby the phases of a liquid – liquid dispersion interchange such that the dispersion phase spontaneously inverts to become the continuous phase and vice versa under conditions determined by the system properties, volume ratio and energy input. The phase inversion point is the holdup of the dispersed phase for a system at which the transition occurs i.e. when the dispersed phase becomes the continuous phase after an infinitesimal change is made to the system’s properties, phase ratio or energy input, Phase inversion can be regarded as a form of the instability of the system, the stability of the dispersion being the latest at the point of phase inversion. Phase inversion is thus a very important factor to consider in liquid – liquid extraction since it can be used effectively in the separation of two immiscible phases\(^ {12}\).

Phase inversion behaviour is affected by both the physical properties of the liquids that make up the system as well as the geometric factors of the vessel that the liquids are contained within. Fluid physical properties such as viscosity, density and interfacial tension are among those that affect the phase inversion process. Various geometrical factors such as the agitation speed, the number and type of impellers, the materials of construction and their wetting characteristics are found to influence phase inversion and the ambivalence range\(^ {13}\).

In a system of two immiscible liquids, usually water (or an aqueous solution) and an organic liquid (e.g. an oil), there are two general types of dispersions
which can be formed depending on the conditions of the system. Water-in-oil (W/O) dispersion is a dispersion formed when the aqueous phase is dispersed in the organic phase and an oil-in-water (O/W) dispersion is a dispersion which is formed when the organic phase is dispersed in the aqueous phase. This is illustrated in (Figure (2.2)) below. Thus, by definition, the phase inversion point is the holdup of the dispersed phase for a system at which the transition occurs i.e. when the dispersed phase becomes the continuous phase after an infinitesimal change is made to the system’s properties, phase ratio or energy input\textsuperscript{[14]}.

![Figure (2.2) Phase Inversion Process for an Oil-Water Dispersion System (Ar, 1999)](image)

In some operations, Spontaneous inversion can be extremely undesirable, especially for mixer-settlers, since the settling times are very different for oil-in-water systems and for water-in-oil systems. Knowing which phase will be the dispersed phase is important in these circumstances. For oil/water flows in
pipes, it is important to predict the phase Inversion point since it is in this vicinity that the extremes of the pressure gradients will often be found. Little is known about the detailed mechanism of the phase inversion phenomenon despite the fact that phase inversion has been studied for the past 40 years. In Recent years, there has been a revived interest in this area especially for liquid-liquid flow in pipes, because of the abrupt and significant changes that occur in the Frictional pressure drop and the rheological characteristics of the dispersion at or near the phase inversion point2 {see Figure (2.3)}. Nevertheless, much research is still urgently required in order to fully understand the phase inversion process and the mechanisms behind it15.

![Figure (2.3) Mixture viscosities as a function of input water volume fraction for low Viscosity oils (Ar, 1999)](image-url)
2.4.4 Dispersion of liquids:

When two immiscible liquids are mixed in extraction column, one of the liquids breaks up in the form of droplets suspended in the continuum of the other liquid. The liquid which is in the form of droplets is known as the dispersed phase, and the continuum liquid is known as the continuous phase. The mixing of immiscible liquids to form dispersed is important in several chemical processes. Dispersion increases the interfacial area available for the required interfacial transfer operation, and as a result it enhances the rate of these processes. Phase inversion is the transition from one phase dispersed to the other. Knowledge of the conditions under which the phase inversion occurs is important in the design of the liquid extraction columns. Factors which may influence the dispersion include the density difference which may affect system stability on the phase inversion, viscosities, interfacial tension, and temperature. The dependence of the dispersion on some of the factors on the phase inversion is either not established or the conclusions drawn are contradictory, resulting from different conditions at which the experiments were performed.[16]

2.5 Liquid – Liquid Extraction Column:

2.5.1 Columns:

There are more types of columns employed industrially, packed columns, pulse columns with plates or trays, mixers settlers, rotating disc contactor (RDC), Scheibel column and centrifugal contactors.

2.5.1.1 Packed columns:

packed columns are filled with some type of packing material, such as Raschin Rings, to create a tortuous path for the two solutions as they flow through the column (typically aqueous feed downward and solvent upward), ensuring that the two phases are in constant contact. Packed columns have no moving parts and are relatively simple to operate, but they are not very efficient.
Since columns do not have discrete stages, such as mixer-settlers or centrifugal contactors, the number of stages is determined by the height of a theoretical stage\textsuperscript{[17]}.

2.5.1.2 Pulse Column:

The most common type of column used, particularly in the nuclear industry, is the pulse column. In a pulse column, liquids are continuously fed to the column and flow counter-currently, as is done with a packed column, but mechanical energy is applied to pulse the liquids in the column up and down. This is normally done by injecting pressurized air into a pulse leg that pushes liquid into the column, then venting the pulse leg to fill the pulse leg with solution from the column; the pulse action lifts and lowers the solution in the column, usually only a few inches. The column is filled with perforated plates or other plates to promote droplet formation as the dispersed phase is pushed through the plates. This pulsing action reduces droplet size of the dispersed phase and improves mass transfer. There is a number of plate design used. Early pulse columns used sieve plates, which are flat plates with holes drilled into them. A more effective plate is the nozzle plate, must be configured according to the continuous phase in the column.

The French and Japanese pulse columns employ a ‘disk and donut’ configuration, where the plates are solid (no openings) but the alternating plates enable effective contacting of the phases. The separation interface is controlled during column operation using bubble probes in the disengaging section. The probes are interfaced to a controller that drains heavy phase from the bottom of the column.

A pulsed packed column consists of a vertical cylindrical vessel fitted with packing. It is important that the packing be wetted preferentially by the continuous phase, thus ensuring that the drops of dispersed phase will not be
severely coalesced within the packed volume. Light and heavy liquids, either one of which is dispersed in the form of droplets, pass counter-currently through the column. At the top or bottom of the column, the dispersed phase coalesces at an interface layer. For perforated (sieve) plate columns, the column is fitted with horizontal plates which occupy the entire column cross-section without any down comers. The unique features of pulsed perforated plate columns are their low axial mixing and high extraction efficiency which are due to uniform distribution of energy over a cross-section of the column, and hence, uniform distribution of droplets in the column\textsuperscript{18}.

2.5.3 Sieve Trays:

Have tray deck areas uniformly perforated with round holes. Tray designs with perforations as small as 6mm or as large as 25mm are common with 13mm/19mm being the most frequently used. Vapour flow through the tray deck to contact the liquid is controlled by the number and size of the perforations. For efficient operation, the hole velocity must be sufficient to balance the head of liquid on the tray deck and thus prevent liquid from passing through the perforations to the tray below. On the other hand high hole velocities may cause severe liquid entrainment to the tray above. Consequently Sieve Trays have a narrow operating range, no more than 2:1. It is particularly suitable for corrosive systems where absence of mechanical moving parts is advantageous. Either the heavy liquid or the light liquid may be dispersed. If the light phase is dispersed, the light liquid flows through the perforations of each plate and is dispersed into drops which rise through the continuous phase. The continuous phase flows horizontally across each plate and passes to the plate below through the down comer\textsuperscript{19}.
2.6 The design of a sieve tray extraction column:

Requires basically the specification of two dimensions; the diameter and the height of the column. The designers know the number of theoretical stages needed for the separation, choose the separation between two plates, and determine the overall efficiency.

In recent years, liquid - liquid extraction has gained increased attention as a commercial separation method in the process industry. In the classification of non- mechanically agitated contactors, the sieve tray extractor has an important role due to the relatively high throughputs, the moderate efficiency, and the simplicity of construction and operation, which is similar to the well-known sieve tray distillation column.

The operation of a liquid-liquid extraction column, where the light liquid is the dispersed phase, is shown in figure (2.1). The heavy liquid flows downward through such an extractor horizontally across each tray and through the downcomers from tray to tray. The light liquid issues from the formations in each tray in the form of jets or drops, rises through the heavy liquid in the form of drops (drop rise), enters into a layer of light liquid which accumulates immediately under each tray.

2.7 Determination of a sieve Tower Diameter (D_T):

The diameter of the column must be large enough to permit two phases to flow counter-currently through the column without flooding.

\[ D_T = \sqrt{\frac{4 \times A_T}{\pi}} \]  \hspace{1cm} (2.1)

Where:

\( A_T \) = total plate area

2.8 Determination of the velocity through perforations (V_o):

Hole size in a sieve plate is one of the important factors of the velocity and efficiency of the plate.
\[ V_0 = 2.69 \left( \frac{d_j}{d_o} \right)^2 \left[ \frac{\sigma}{d_j(0.5137 \rho_D + 0.4719 \rho_c)} \right]^{0.5} \] .......................................................... (2.2)

Where
\( d_j \) = jet diameter
\( d_o \) = orifice diameter
\( \sigma \) = interfacial tension
\( \sigma_D \) = density of dispersed phase
\( \sigma_c \) = density of continuous phase

2.9 Determination of the Tower Height (H_T):

The tower height can be related to the number of trays in the column. The following formula assumes that a spacing of tower feet between trays will be sufficient including additional five to ten feet at both ends of the tower. This includes a fifteen percent excess allowance of space (Douglas, 1988).

\[ H_T = (N_a - 1)C_t + \frac{N_aC_t}{10} + 0.1H_T \] .......................................................... (2.3)

Thus

\[ H_T = \frac{1}{0.9} (N_a - 1)C_t + \frac{N_aC_t}{9} \] .......................................................... (2.4)

Where
\( N_a \) = the number of actual stages
\( C_t \) = tray spacing

2.10 Determination of the theoretical Trays (N_T):

In order to develop a method for the design of distillation units to give the desired fractionation, it is necessary, in the first instance, to develop an analytical approach which enables the necessary number of trays to be calculated. First the heat and material flows over the trays, the condenser, and the reboiler must be established. Thermodynamic data are required to establish how much mass transfer is needed to establish equilibrium between the streams leaving each tray. The required diameter of the column will be dictated by the necessity to accommodate the desired flow rates, to operate within the available
drop in pressure, while at the same time effecting the desired degree of mixing of the streams on each tray.

Four streams are involved in the transfer of heat and material across a plate, as shown in Figure (2.4) in which plate \( n \) receives liquid \( L_{n+1} \) from plate \( n + 1 \) above, and vapour \( V_{n-1} \) from plate \( n - 1 \) below. Plate \( n \) supplies liquid \( L_n \) to plate \( n - 1 \), and vapour \( V_n \) to plate \( n + 1 \).

The action of the plate is to bring about mixing so that the vapour \( V_n \), of composition \( y_n \), approaches equilibrium with the liquid \( L_n \), of composition \( x_n \).

The streams \( L_{n+1} \) and \( V_{n-1} \) cannot be in equilibrium and, during the interchange process on the plate, some of the more volatile component is vaporised from the liquid \( L_{n+1} \), decreasing its concentration to \( x_n \), and some of the less volatile component is condensed from \( V_{n-1} \), increasing the vapour concentration to \( y_n \).

The heat required to vaporise the more volatile component from the liquid is supplied by partial condensation of the vapour \( V_{n-1} \). Thus the resulting effect is that the more volatile component is passed from the liquid running down the column to the vapour rising up, whilst the less volatile component is transferred in the opposite direction.

![Figure (2.4) Material balance over a plate](image)
2.11 Heat balance over a plate

A heat balance across plate \( n \) may be written as:

\[
L_{n+1}H_{n+1}^L + V_{n-1}H_{n-1}^V = V_n H_n^V + L_n H_n^L + \text{losses} + \text{heat of mixing} \quad \text{......... (2.5)}
\]

Where

- \( H_n^L \) is the enthalpy per mole of the liquid on plate \( n \), and
- \( H_n^V \) is the enthalpy per mole of the vapour rising from plate \( n \).

This equation is difficult to handle for the majority of mixtures, and some simplifying assumptions are usually made. Thus, with good lagging, the heat losses will be small and may be neglected, and for an ideal system the heat of mixing is zero. For such mixtures, the molar heat of vaporisation may be taken as constant and independent of the composition. Thus, one mole of vapour \( V_{n-1} \) on condensing releases sufficient heat to liberate one mole of vapour \( V_n \). It follows that \( V_n = V_{n-1} \), so that the molar vapour flow is constant up the column unless material enters or is withdrawn from the section. The temperature change from one plate to the next will be small, and \( H_n^L \) may be taken as equal to \( H_{n+1}^L \).

Applying these simplifications to equation (2.5), it is seen that \( L_n = L_{n+1} \), so that the moles of liquid reflux are also constant in this section of the column. Thus \( V_n \) and \( L_n \) are constant over the rectifying section, and \( V_m \) and \( L_m \) are constant over the stripping section.

For these conditions there are two basic methods for determining the number of plates required. The first is due to SOREL\textsuperscript{[20]} and later modified by LEWIS\textsuperscript{[21]}, and the second is due to MCCABE and THIELE\textsuperscript{[22]}. The Lewis method is used here for binary systems, this method is also the basis of modern computerised methods. The McCabe–Thiele method is particularly important since it introduces the idea of the operating line which is an important common concept in multistage operations. The best assessment of these methods and their various applications is given by UNDERWOOD\textsuperscript{[23]}. 
2.12 Determination of the Column efficiency ($E_o$):

There is no entirely satisfactory method available for finding the tray efficiency. Various correlations are available for finding tray efficiency, but they are used only if the reliable actual data on the same or similar system are not available. Overall efficiency of tray tower is given by the ratio of theoretical stages to real stages by following equation:

$$E_o = \frac{N_T}{N_a}$$  \hspace{1cm} (2.6)

Where

$N_T$ = the number of theoretical stages \\
$N_a$ = the number of actual stages \\

2.13 Determination of the number of actual stages ($N_a$):

This is determined by taking the quotient of the number of theoretical trays to the tray efficiency. Typical values for tray efficiency range from 0.5 to 0.7 (Douglas, 1988). These values depend on the type of trays being used. As well as the internal liquid and vapour flow rates.

$$N_a = \frac{N_T}{E_o}$$  \hspace{1cm} (2.7)

Where

$N_T$ = the number of theoretical stages \\
$E_o$ = tray efficiency
Chapter Three

Materials and Methods

The following need to be carefully evaluated when optimizing the design and operation of the extraction process:

- Solvent Selection
- Operating Conditions
- Mode of Operation
- Extractor Type
- Design Criteria

3.1 Selection of extraction conditions

Depending on the nature of the extraction process, the temperature, pH and residence time could have an effect on the yield. The pH becomes significant in metal and bio-extractions. In bio-extractions (e.g., penicillin) and some agrochemicals (e.g. Orthene), pH is maintained to improve distribution coefficient and minimize degradation of product. In metal extraction, kinetic considerations govern the pH. In dissociation-based extraction of organic molecules, pH can play a significant role (e.g., cresols separation). Sometimes, the solvent itself may participate in undesirable reactions under certain pH conditions (e.g., ethyl acetate may undergo hydrolysis in presence of mineral acids to acetic acid and ethanol). Residence time is an important parameter in reactive extraction processes (e.g., metals separations, formaldehyde extraction from aqueous streams) and in processes involving short-life components (e.g., antibiotics & vitamins)

3.2 Selection of mode of operation

Extractors can be operated in crosscurrent, co-current or counter-current mode. The following section compares these configurations.
3.2.1 Cross-Current Operation

Cross-current operation is mostly used in batch operation. Batch extractors have traditionally been used in low capacity multi-product plants such as are typical in the pharmaceutical and agrochemical industries. For washing and neutralization operations that require very few stages, crosscurrent operation is particularly practical and economical and offers a great deal of flexibility. Single stage extraction is used when the extraction is fairly simple and can be achieved without a high amount of solvent. If more than one stage is required, multiple solvent-washes are given.

Though operation in cross-current mode offers more flexibility, it is not very desirable due to the high solvent requirements and low extraction yields. The following illustration gives quick method to calculate solvent requirements for cross-current mode of extraction.

3.2.1.1 A single-stage extraction can be represented as:

![Diagram of single-stage extraction](image)

Figure (3.1) Single-Stage Extraction

A feed with a composition located at F in figure (3.1) being contacted with S kg of pure solvent, located at point S. M is the location of the mixture that results when F kg feed are contacted with the solvent, and represents the equilibrium tie-lines.

Composing total and component solute mass balances as previously stipulated, we obtain

\[ F + S = E + R = M \]  

(3 – 1)
Point M₁ can be located on line FS.

Where

\[ F = \text{Feed quantity / rate, mass / mass/time} \]
\[ R = \text{Raffinate quantity / rate, mass} \]
\[ S = \text{Solvent quantity / rate, mass} \]
\[ E = \text{Extract quantity / rate, mass} \]
\[ M = \text{the mixture} \]

\( Xₕ, X_r, Y_s, \) and \( Y_e \) are the weight fractions of solute in the feed, raffinate, solvent and extract, respectively.

The component mass balance can be represented as:

\[ Fx_F + Sy_S = Rx_R + Ey_E = Mx_M \] \( (3-2) \)

If the solvent is pure S \((y_s = 0)\), thus

\[ Fx_F + Sy_S = Mx_m \] \( (3-3) \)

it is desired to calculate the equilibrium composition that results, as well as the amount of raffinate(R) and extract (E) produced. We first locate the composition \( x_m \) of the mixing point M by eliminating total mass M from the left side of the two balances. Thus \(^{[18]} \).

\[ x_m = \frac{Fx_F + Sy_S}{F + S} \] \( (3-4) \)

### 3.2.2 Multi-stage extraction:

3.2.2.1 Multi-stage cross-current operation:

![Multi-Stage Cross Current Operation](image)

Figure (3.2): Multi-Stage Cross Current Operation
This kind of extraction is an extraction of the single step extraction because more single step units are combined as given in figure (3.2)

For the multi step extraction with cross flow the raffinate of each step is contacted in the following step with pure solvent. The extracts are withdrawn from each step and given to the solvent regeneration.

The concentration of compound (C) in raffinate and extract decreases from step to step. If the point of feed (F) and solvent(S) are known the first mixing point (M₁) can be determined in the same way as for the single steps extraction.

This mixing point separates in raffinate R₁ and extract E₁. For the following steps the raffinate is the feed which is contacted with solvent L. The total extract results from the extract of the single steps:

\[ E = \sum_{i=1}^{n} E_i \]  

(3 - 5)

The last raffinate concentration (Rₙ) can be also achieved in single step extraction. The corresponding mixing point M₁ can be constructed as crossing point of FS and R₁E₁.

By the law of balance it is obvious that the amount of solvent for the single step extraction is much higher than for the multi step extraction with cross flow.

3.2.3 Multi stage extraction with counter-current flow:

The feed and the solvent flow in counter current way through the apparatus. This is a continuous process where feed and solvent enter the apparatus at opposite ends. While raffinate is contacted with pure solvent the extract is contacted with the feed.

![Figure (3.3) multi stage extraction with counter current flow](image-url)
Basic for the construction are the mass balance. It is obvious that the difference of the mass flow D in a section between two steps is constant. The result is that the balance lines cross in one point, the pole point P total balance:

\[ F - E_1 = R_n - S = \Delta \] ................................................................. (3 - 6)

Balance for one step (e.g. m):

\[ R_{m-1} - E_m = R_n - S = \Delta \] ................................................................. (3 - 7)

With D as a hypothetical amount of the pole point P result the amount F and the single raffinates as mixing point of P with the corresponding extracts.

If S is given and \( R_n \) is wanted with the knowledge of the hypothetical pole point amount results:

\[ R_n = P + L \quad \text{or} \quad R_n = L \cdot \frac{P}{PR_n} \] ................................................................. (3 - 8)

Where \( P = \) pole point

If \( R_n \) is given so S can be determined by the law of balance:

\[ L = R_n \cdot \frac{R_{nP}}{PL} \] .................................................................................. (3 - 9)

The position of the pole point P results as crossing point of the lines F and \( R_n \)S.

In most cases not all four points F ,E_1 ,R_n and L are given normally.

The raffinate concentration \( R_n \) and either the extract concentration E_1 or the amount of solvent is given. With the help of the mixing point the missing point can be determined.

Combining this raffinate \( R_1 \) with the pole point last in the extract \( E_2 \) of the next step. The line \( PR_1E_2 \) is a balance line. This construction is repeated until the desired raffinate concentration is reached. By the numbers of raffinate points the number of theoretical steps is determined^{[21]}.

3.3 choosing a solvent system:

One important aspect when choosing a solvent system for extraction is to pick two immiscible solvents. Some common liquid - liquid extraction solvent pairs are water - ether, water - dichloromethane, and water - hexane. Notice that each combination includes water. Most extractions involve water because it is highly
polar and immiscible with most organic solvents. In addition, the compound that attempting to extract must be soluble in the organic solvent, but insoluble in the water layer. An organic compound like benzene is simple to extract from water, because its solubility in water is very low. However, solvents like ethanol and methanol will not separate using liquid - liquid extraction techniques, because they are soluble in both organic solvents and water.

There are also practical concerns when choosing extraction solvents. As mentioned previously.

**3.4 Solvent Selection:**

For the selection of a suitable solvent, one has to consider not only the extraction selectivity, but also the ease of handling and regeneration, the solubility in the raffinate and the product cost. Solvents differ in their extraction capabilities depending on their own and the solute’s chemical structure. Sandler presents a table showing Organic-Group interactions from which one can identify the desired functional group(s) in the solvent for any given solute\(^{[20]}\).

Once the functional group is identified, possible solvents can be screened in the laboratory. The distribution coefficient and selectivity are most important parameters that govern solvent selection. The distribution coefficient (m) or partition coefficient for a component (A) is defined as the ratio of concentration of A in extract phase to that in raffinate phase. Selectivity can be defined as the ability of the solvent to pick up the desired component in the feed as compared to other components. The desired properties of solvents are a high distribution coefficient, good selectivity towards solute and little or no miscibility with feed solution. Also, the solvent should be easily recoverable for recycle. Designing an extractor is usually a fine balance between capital and operating costs. Usually, a good solvent also exhibits some miscibility with feed solution. Consequently, while extracting larger quantities of solute, the solvent could also extract significant amount of feed solution.
Other factors affecting solvent selection are boiling point, density, interfacial tension, viscosity, corrosiveness, flammability, toxicity, and stability, compatibility with product, availability and cost.

The extraction process can become very expensive if the solvent needed to be used is costly.

For an existing process, replacing the solvent is usually a last resort because this would call for going back to laboratory screening of the solvent and process optimization. However, changes in environmental regulations and economic considerations often induce the need to improve the process in terms of solute recovery.

Also the availability of specialized and proprietary solvents that score over conventional solvents in terms of performance and economics for several extraction processes can provide additional incentives for a solvent change.

### 3.5 Design parameters:

The following are a partial list of the needed physical properties in liquid – liquid extraction separations. It is by no means complete; other properties will be needed for some of the calculations, and specially those needed to size the diameter of the column. It is whoverever complete as it relates to the described theory:

#### 3.5.1 Temperature:

Plays important role in extraction than in other separation processes. It is only dependent upon the temperatures of the streams fed into the column. There is not a heating requirement for the process and $\Delta H$ of mixing is generally insignificant.

For this reasons, extraction can be regarded as an isothermal process.

#### 3.5.2 Pressure:

Plays only a small role in extraction. When combined with the temperature considerations it is only necessary that the mixture remain in the two phase liquid region. The fact that extraction processes can be run at isothermal and
isobaric conditions is quite beneficial to the phase stability of the system. Phase stability from a thermodynamic standpoint is temperature and pressure dependent and since these are not changing the stability of the phases will not change.

3.5.3 Activity coefficients:
Are the most important physical property in the extraction process. The reason for this is that these are used to determine the miscibility of the solute in both of the solvents involved. While there are many different equations available to determine a particular activity some are better than others for our purposes. When working with liquid–liquid systems the NRTL and the UNIFAC models are the most accurate in predicting the activities of the liquids involved. Although better than such predictive models such as Van Laar or Margules they still fall short of perfection.

Once a predictive model has been plotted on a diagram it will most likely be necessary to fix the exact equilibrium line experimentally for the most accurate data. The activity coefficient also determine the partition factor which will determine whether or noy a good separation is possible.

3.5.4 Viscosity:
Is a property that cannot be overlooked, its presence appears in two different areas, flooding and choice of equipment. Viscosity is also valuable in the determination of what type of system to use for extraction.
3.6 Limitations of tray operations:

We must consider the under what extremes can be used as separation process:

3.6.1 Suitable solvent:

- Solvent partially soluble with the carrier.
- Feed components immiscible with the solvent.
- Solute is soluble in the carrier and at the same time completely or partially soluble in the solvent.
- Different densities than the feed components for a phase separation to facilitate and maintain the capacity of the extractor high.
- Extremely high selectivity for the solute for the solvent to dissolve the maximum amount of solute and the minimum amount of the carrier.
- Low viscosity increases the capacity of the extraction column and does not allow for the settling rate of dispersion to be slow.
- Chemically stable and inert toward other components of the system
- Low cost, nontoxic and non flammable

3.6.2 Equipment:

- Interfacial tension and viscosity
- High interfacial tension and viscosity leads to more power being supplied to maintain rapid mass transfer throughout the extraction process.
- Low interfacial tension and viscosity leads to the formation of an emulsion.
Chapter Four

Results and Discussion

4.1 Design of A sieve – tray extraction tower:

Case study:

- The Feed is 7000 kg/hr of an acetic acid (C) – Water (A) contains 35% acid.
- The solvent used to perform the counter currently extraction is pure isopropyl ether (S) with a flow rate equal to 21000 kg/hr.
- The exiting raffinate stream contains 2% acetic acid.
- operating is 25°C and 1 atm.

4.1.1 The number of theoretical stages Calculation:

Equilibrium data of the ternary system: Water (A) – Acetic acid (C) – isopropyl ether (S) at 25°C and 1 atm.

Table (4.1): equilibrium data of ternary system

<table>
<thead>
<tr>
<th></th>
<th>Water – rich phase</th>
<th></th>
<th>Solvent – rich phase</th>
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</thead>
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<td>$X_{AA}$</td>
<td>$X_{CA}$</td>
<td>$X_{SA}$</td>
<td>$X_{AS}$</td>
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<td>0.464</td>
<td>0.165</td>
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</tr>
</tbody>
</table>
4.1.1.1 Procedure and Calculations:

A total material balance around the entire plant is:
\[ F + S = E_1 + R_N = M \] ................................................................. (4 - 1)

\[ F = \frac{\text{mass}}{\text{time}} \] contains substances Water (A) and Acetic acid (C) = 7000 kg/hr

\[ S = \frac{\text{mass}}{\text{time}} \] of Solvent Isopropyl ether (S) = 21000 kg/hr

\[ M = F + S \] .................................................................................... (4 - 2)

\[ M = 7000 + 21000 = 28000 \text{ kg/hr} \]

Point M can be located on line FS

\[ E_1 + R_N = M \] .................................................................................... (4 - 3)

A material balance for acetic acid (C):

\[ Fx_{C,F} + Sy_{C,S} = E_1y_{C,E1} + R_Nx_{C,RN} = Mx_{C,M} \] ................................................................. (4 - 4)

\[ Fx_{C,F} + Sy_{C,S} = Mx_{C,M} \] .................................................................................... (4 - 5)

\[ X_{C,F} = \text{Weight fraction of acetic acid (C) in Feed} = 0.35 \]

\[ Y_{C,S} = \text{Weight fraction of acetic acid (C) in Solvent} = 0 \]

The solvent is pure isopropyl ether (S).

**4.1.1.2 Determination of the Mixture composition (X_{C,M}):**

From equation (4.5)

\[ Fx_{C,F} + Sy_{C,S} = Mx_{C,M} \]

\[ (7000*0.35) + (21000*0.0) = 28000 * X_{C,M} \]
$X_{C,M} = 0.0875$

**Figure (4.2) The location of the mixing point composition ($X_{C,M}$)**

### 4.1.1.3 Determination of the Extract (E) Composition ($y_{C,E}$):

The extract (E) composition ($y_{C,E}$) is determined by drawing a straight line from ($x_{C,RN} = 0.02$) through ($x_{C,M} = 0.0875$) until the line intersects the extract line at the final extract composition {from figure (4.3)}
4.1.1.4 Determination of the difference point $\Delta_R$:

The difference point is then found at the intersection of two lines:

- One line connects the feed ($x_{C,F} = 0.35$) and extract composition ($y_{C,E1} = 0.1$)
- The other line connects the raffinate ($x_{C,RN} = 0.02$) and solvent composition ($y_{C,S} = 0$).
4.1.1.5 The graphical Construction:

After location of points F, S, M, E₁, R₇ and Δᵣ, a few lines are drawn from point Δᵣ to intersect the two branches of the solubility curves. A tie-line from E₁ provides R₁ since extract and raffinate from the first stage are in equilibrium. A line from Δᵣ through R₁ when extended provides E₂.

A tie-line from E₂ provides R₂, a tie-line from E₃ provides R₃, a tie-line from E₄ provides R₄, a tie-line from E₅ provides R₅, a tie-line from E₆ provides R₆ and a tie-line from E₇ provides R₇ = 0.02

The lowest possible value of \( x_{c,N} \) is given by the water-rich end of the tie-line which passes through S.

A few lines are drawn from point Δᵣ to intersect the two branches of the solubility curves {from figure (4.5)}.  

Figure (4.4) The location of the difference point Δᵣ
The operating concentration $x_{c,\text{op}}$ and $y_{c,\text{op}}$ corresponding to these are given in Table (4.2).

Table (4.2) Operating Curve Data

<table>
<thead>
<tr>
<th>$x_{c,\text{op}}$</th>
<th>$y_{c,\text{op}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$x_{c,\text{RN}} = 0.02$</td>
<td>$y_{c,S} = 0$</td>
</tr>
<tr>
<td>0.055</td>
<td>0.01</td>
</tr>
<tr>
<td>0.140</td>
<td>0.03</td>
</tr>
<tr>
<td>0.210</td>
<td>0.05</td>
</tr>
<tr>
<td>0.275</td>
<td>0.07</td>
</tr>
<tr>
<td>0.330</td>
<td>0.09</td>
</tr>
<tr>
<td>$x_{c,F} = 0.35$</td>
<td>$y_{c,E1} = 0.10$</td>
</tr>
</tbody>
</table>
The concentration \( x = \frac{x_{CA}}{x_{AA}} \) and \( y = \frac{x_{CS}}{x_{SS}} \) corresponding to these are given in Table (4.3).

\[
x = \frac{x_{CA}}{x_{AA}}
\]

\[
x = \frac{0.007}{0.981} = 0.007
\]

In same way, the other points were determined.

\[
y = \frac{x_{CS}}{x_{SS}}
\]

\[
y = \frac{0.002}{0.993} = 0.002
\]

In same way, the other points were determined.

<table>
<thead>
<tr>
<th>( x = \frac{x_{CA}}{x_{AA}} )</th>
<th>( y = \frac{x_{CS}}{x_{SS}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.007</td>
<td>0.002</td>
</tr>
<tr>
<td>0.0144</td>
<td>0.004</td>
</tr>
<tr>
<td>0.0304</td>
<td>0.008</td>
</tr>
<tr>
<td>0.0698</td>
<td>0.0196</td>
</tr>
<tr>
<td>0.158</td>
<td>0.0514</td>
</tr>
<tr>
<td>0.359</td>
<td>0.135</td>
</tr>
<tr>
<td>0.623</td>
<td>0.302</td>
</tr>
<tr>
<td>0.982</td>
<td>0.535</td>
</tr>
<tr>
<td>1.25</td>
<td>0.743</td>
</tr>
</tbody>
</table>

These data are calculated from \{table (4.1)\}, the concentration \( x_{c,op} \) and \( y_{c,op} \) are plotted on X and Y coordinates as shown in figure (4.6) to generate an operating curve.
The graphical approach is determined using the McCabe – Thiele Method for binary mixtures. Tie-line data provide the equilibrium curve \( X \) vs. \( Y \) and the theoretical stages are stepped off in the resulting McCabe – Thiele diagram.

**4.1.1.6 Calculation of the number of stages:**

Following the tie line from point E to the other side of the equilibrium curve will give the composition of an intermediate raffinate stage. Another operating line is drawn from the operating point, through this intermediate point, and ends at point E; this is a stage of the system. This procedure should be repeated until stages have been constructed to R, the raffinate composition. Figure (4.6) shows this procedure for a general case. A total of 7 Equilibrium stages are required for special separation.

![Equilibrium curve and Operating curve](image)

Figure (4.6): Construction of the equilibrium, operating curves, and step off of the number of theoretical stages
Table (4.4): Extract and Raffinate Concentration Profiles:

<table>
<thead>
<tr>
<th>Stage</th>
<th>$X_{c,Ri}$</th>
<th>$Y_{c,Ei}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>0.275</td>
<td>0.0750</td>
</tr>
<tr>
<td>2.</td>
<td>0.205</td>
<td>0.0500</td>
</tr>
<tr>
<td>3.</td>
<td>0.155</td>
<td>0.0350</td>
</tr>
<tr>
<td>4.</td>
<td>0.110</td>
<td>0.0225</td>
</tr>
<tr>
<td>5.</td>
<td>0.075</td>
<td>0.0125</td>
</tr>
<tr>
<td>6.</td>
<td>0.050</td>
<td>0.0067</td>
</tr>
<tr>
<td>7.</td>
<td>0.020</td>
<td>0.000</td>
</tr>
</tbody>
</table>

4.1.1.7 Determination of the extract ($E_i$) and raffinate ($R_i$) Flow rates:

From equation (4.1) & (4.2):

$$E_1 = M - R_N$$ .............................................................. (4 - 6)

Equation (4.3) and (4.4) gives:

$$F x_{c,F} + S y_{c,S} = (M - R_N) y_{c,E1} + R_N x_{c,RN}$$ .............................................................. (4 - 7)

$$R_N = \frac{F x_{c,F} + S y_{c,S} - M y_{c,E1}}{x_{c,RN} - y_{c,E1}}$$ .............................................................. (4 - 8)

$$R_N = \frac{(7000 \times 0.35) + (21000 \times 0) - (28000 \times 0.1)}{(0.02 - 0.1)} = 4375 \text{ kg/hr}$$

$$R_N = 4375 \text{ kg/hr}$$

$$E_1 = 28000 - 4375 = 23625 \text{ kg/hr}$$
4.1.1.8 Stage wise determination of the number of theoretical stage:

Stage (1):

\[ R_1 = ? \quad \text{x}_{c,R1} = 0.275 \]
\[ E_2 = ? \quad \text{y}_{c,E2} = 0.075 \]

\[ F = 7000 \text{ kg/hr} \quad \text{E}_1 = 23625 \]
\[ \text{x}_{c,F} = 0.35 \quad \text{y}_{c,E1} = 0.1 \]

A total material balance:
\[ F + E_2 = R_1 + E_1 \]

Acetic acid balance:
\[ F\text{x}_{c,F} + E_2\text{y}_{c,E2} = R_1\text{x}_{c,R1} + E_1\text{y}_{c,E1} \]

From equation (4.9):
\[ R_1 = F + E_2 - E_1 \]

Equation (4.10) and equation (4.11) gives:
\[ E_2 = \frac{F(x_{c,F} - x_{c,R1}) + E_1(x_{c,R1} - y_{c,E1})}{(x_{c,R1} - y_{c,E2})} \]

\[ E_2 = \frac{7000(0.35 - 0.275) + 23625(0.275 - 0.1)}{(0.275 - 0.075)} \]
\[ E_2 = 23295 \text{ kg/hr} \]
\[ R_1 = 7000 + 23295 - 23625 = 6670 \text{ kg/hr} \]
\[ R_1 = 6670 \text{ Kg/hr} \]
Stage (2):

\[ x_{c,R2} = 0.205 \]
\[ y_{c,E3} = 0.05 \]

\[ R_2 = ? \]
\[ E_3 = ? \]

\[ R_1 = 6670 \text{ kg/hr} \]
\[ y_{c,E2} = 0.075 \]

A total material balance:
\[ R_1 + E_3 = R_2 + E_2 \] \hspace{1cm} (4 - 13)

Acetic acid balance:
\[ R_1 x_{c,R1} + E_3 y_{c,E3} = R_2 x_{c,R2} + E_2 y_{c,E2} \] \hspace{1cm} (4 - 14)

From equation (4.13):
\[ R_2 = R_1 + E_3 - E_2 \] \hspace{1cm} (4 - 15)

Equation (4.14) and equation (4.15) gives:
\[ E_3 = \frac{R_1 (x_{c,R1} - x_{c,R2}) + E_2 (x_{c,R2} - y_{c,E2})}{(x_{c,R2} - y_{c,E3})} \] \hspace{1cm} (4 - 16)

\[ E_3 = \frac{6670(0.275 - 0.205) + 23295(0.205 - 0.075)}{(0.205 - 0.05)} = 22555 \text{ kg/hr} \]

\[ E_3 = 22555 \text{ kg/hr} \]
\[ R_2 = 6670 + 22555 - 23295 = 5930 \text{ kg/hr} \]
\[ R_2 = 5930 \text{ kg/hr} \]
Stage (3):

\[ x_{c,R3} = 0.155 \quad Y_{c,E4} = 0.035 \]
\[ R_3 = ? \quad E_4 = ? \]

\[ R_2 = 5930 \text{ kg/hr} \quad E_3 = 22555 \text{ kg/hr} \]
\[ x_{c,R2} = 0.205 \quad y_{c,E3} = 0.0500 \]

A total material balance:

\[ R_2 + E_4 = R_3 + E_3 \]

(4 - 17)

Acetic acid balance:

\[ R_2 x_{c,R2} + E_4 y_{c,E4} = R_3 x_{c,R3} + E_3 y_{c,E3} \]

(4 - 18)

From equation (4.17):

\[ R_3 = R_2 + E_4 - E_3 \]

(4 - 19)

Equation (4.17) and equation (4.18) gives:

\[ E_4 = \frac{R_2(x_{c,R2} - x_{c,R3}) + E_3(x_{c,R3} - y_{c,E3})}{(x_{c,R3} - y_{c,E4})} \]

(4 - 20)

\[ E_4 = \frac{5930(0.205-0.155)+22555(0.155-0.05)}{(0.155-0.035)} = 22205 \text{ kg/hr} \]

\[ E_4 = 22205 \text{ kg/hr} \]

\[ R_3 = 5930 + 22205 - 22555 = 5580 \text{ kg/hr} \]

\[ R_3 = 5580 \text{ kg/hr} \]
Stage (4):

\[ x_{c,R4} = 0.11 \quad y_{c,E5} = 0.0225 \]

\[ R_4 = ? \quad E_5 = ? \]

\[ R_3 = 5580 \text{ kg/hr} \quad E_4 = 22205 \text{ kg/hr} \]

\[ x_{c,R3} = 0.155 \quad y_{c,E4} = 0.035 \]

A total material balance:

\[ R_3 + E_5 = R_4 + E_4 \]

Acetic acid balance:

\[ R_3 x_{c,R3} + E_5 y_{c,E5} = R_4 x_{c,R4} + E_4 y_{c,E4} \]

From equation (4.21):

\[ R_4 = R_3 + E_5 - E_4 \]

Equation (4.22) and equation (4.23) gives:

\[ E_5 = \frac{R_3(x_{c,R3} - x_{c,R4}) + E_4(x_{c,R4} - y_{c,E4})}{(x_{c,R4} - y_{c,E4})} \]

\[ E_5 = \frac{5580(0.155 - 0.11) + 22205(0.11 - 0.035)}{0.11 - 0.0225} = 21900 \text{ kg/hr} \]

\[ E_5 = 21900 \text{ kg/hr} \]

\[ R_4 = 5580 + 21900 - 22205 = 5275 \text{ kg/hr} \]

\[ R_4 = 5275 \text{ kg/hr} \]
Stage (5):

\[
\begin{align*}
    x_{c,R5} &= 0.075 & y_{c,E6} &= 0.0125 \\
    R_5 &= ? & E_6 &= ? \\
    E_5 &= 21900 \text{ kg/hr} & R_4 &= 5275 \text{ kg/hr} \\
    y_{c,E5} &= 0.0225 & x_{c,R4} &= 0.11
\end{align*}
\]

A total material balance:

\[R_4 + E_6 = R_5 + E_5\] ................................................................. (4 - 25)

Acetic acid balance:

\[R_4 x_{c,R4} + E_6 y_{c,E6} = R_5 x_{c,R5} + E_5 y_{c,E5}\] ................................................................. (4 - 26)

From equation (4.25):

\[R_5 = R_4 + E_6 - E_5\] ...................................................................................... (4 - 27)

Equation (4.26) and equation (4.27) gives:

\[E_6 = \frac{R_4(x_{c,R4} - x_{c,R5}) + E_5(x_{c,R5} - y_{c,E5})}{(x_{c,R5} - y_{c,E5})} \] ................................................................. (4 - 28)

\[E_6 = \frac{5275(0.11 - 0.075) + 21900(0.075 - 0.0225)}{(0.075 - 0.0125)} = 21350 \text{ kg/hr}\]

\[E_6 = 21350 \text{ kg/hr}\]

\[R_5 = R_4 + E_6 - E_5\]

\[R_5 = 5275 + 21350 - 21900 = 4725 \text{ kg/hr}\]

\[R_5 = 4725 \text{ kg/hr}\]
Stage (6):

\[ x_{c,R6} = 0.050 \quad \quad y_{c,E7} = 0.0067 \]

\[ E_7 =? \quad \quad R_6 =? \]

\[ E_6 = 21350 \text{ kg/hr} \quad \quad R_5 = 4725 \text{ kg/hr} \]

\[ y_{c,E6} = 0.0125 \quad \quad x_{c,R5} = 0.075 \]

A total material balance:

\[ R_5 + E_7 = R_6 + E_6 \] \hspace{1cm} (4 - 29)

Acetic acid balance:

\[ R_5 x_{c,R5} + E_7 y_{c,E7} = R_6 x_{c,R6} + E_6 y_{c,E6} \] \hspace{1cm} (4 - 30)

From equation (4.29):

\[ R_6 = R_5 + E_7 - E_6 \] \hspace{1cm} (4 - 31)

Equation (4.30) and equation (4.31) gives:

\[ E_7 = \frac{R_5 (x_{c,R5} - x_{c,R6}) + E_6 (x_{c,R6} - y_{c,E6})}{(x_{c,R6} - y_{c,E7})} \] \hspace{1cm} (4 - 32)

\[ E_7 = \frac{4725(0.075 - 0.05) + 21350(0.05 - 0.0125)}{(0.05 - 0.0067)} = 21200 \text{ kg/hr} \]

\[ E_7 = 21200 \text{ kg/hr} \]

\[ R_6 = R_5 + E_7 - E_6 \]

\[ R_6 = 4725 + 21200 - 21350 = 4575 \text{ kg/hr} \]

\[ R_6 = 4575 \text{ kg/hr} \]
Table (4.5): Flow Rate Profiles

<table>
<thead>
<tr>
<th>Stage</th>
<th>$E_i$ (kg/hr)</th>
<th>$R_i$ (kg/hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>23625</td>
<td>6670</td>
</tr>
<tr>
<td>2.</td>
<td>23295</td>
<td>5930</td>
</tr>
<tr>
<td>3.</td>
<td>22555</td>
<td>5580</td>
</tr>
<tr>
<td>4.</td>
<td>22205</td>
<td>5275</td>
</tr>
<tr>
<td>5.</td>
<td>21900</td>
<td>4725</td>
</tr>
<tr>
<td>6.</td>
<td>21350</td>
<td>4575</td>
</tr>
<tr>
<td>7.</td>
<td>21200</td>
<td>4375</td>
</tr>
</tbody>
</table>
Figure (4.7): Diagram of Counter-Current Extraction and Raffinate flow rate
4.1.2 Determination of a sieve tower Diameter (D_T):

Table (4.6): The Design Condition Data of sieve tray extractor

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow rate of water solution(continuous) (kg/hr)</td>
<td>C</td>
<td>7000</td>
</tr>
<tr>
<td>Density of water solution(continuous) (kg/m$^3$)</td>
<td>$\rho_c$</td>
<td>1009</td>
</tr>
<tr>
<td>Density of Isopropyl ether solution (dispersed) (kg/m$^3$)</td>
<td>$\rho_o$</td>
<td>730</td>
</tr>
<tr>
<td>Flow rate of Isopropyl ether solution (dispersed) (kg/hr)</td>
<td>D</td>
<td>21000</td>
</tr>
<tr>
<td>Viscosity of water solution (continuous) (kg/m.s)</td>
<td>$\mu_c$</td>
<td>3.1*10^{-3}</td>
</tr>
<tr>
<td>Volumetric rate of water solution (m$^3$/s)</td>
<td>$q_c$</td>
<td>2.22*10^{-3}</td>
</tr>
<tr>
<td>Volumetric rate of Isopropyl ether solution (dispersed) (m$^3$/s)</td>
<td>$q_o$</td>
<td>7.61*10^{-3}</td>
</tr>
<tr>
<td>Viscosity of Isopropyl ether solution (dispersed) (kg/m.s)</td>
<td>$\mu_o$</td>
<td>0.90*10^{-3}</td>
</tr>
<tr>
<td>Interfacial tension (N/m)</td>
<td>$\sigma$</td>
<td>0.013</td>
</tr>
<tr>
<td>Conversion factor (kg.m/N. s$^2$)</td>
<td>$g_c$</td>
<td>1</td>
</tr>
<tr>
<td>Acceleration of gravity (m/s$^2$)</td>
<td>g</td>
<td>9.807</td>
</tr>
</tbody>
</table>

4.1.2.1 Columns perforations:

4.1.2.1.1 The Orifice diameter to jet diameter ratio ($\frac{d_o}{d_j}$):

\[
d_o = \text{Orifice diameter}^{[17]} = 6\text{mm} = 0.006\text{m}
\]
\[
p = \text{Triangular pitch}^{[17]} \text{ on 15mm centres} = 0.015\text{m}
\]
\[
\Delta \rho = \rho_c - \rho D = 1009 - 730 = 279\text{kg/m}^3
\]
\[
= 279 \text{kg/m}^3 \rho \Delta
\]
\[
\frac{d_o}{d_j} = 0.485 \left[ \frac{d_o}{\left( \frac{\sigma g c}{\Delta \rho g} \right)^{0.5}} \right]^2 + 1 \quad \text{for} \quad \frac{d_o}{\left( \frac{\sigma g c}{\Delta \rho g} \right)^{0.5}} < 0.785 \quad \ldots \ldots \ldots \ldots \quad (4 - 33)
\]
\[
\frac{d_o}{d_j} = 1.51 \frac{d_o}{\left( \frac{\sigma g c}{\Delta \rho g} \right)^{0.5}} + 0.12 \quad \text{for} \quad \frac{d_o}{\left( \frac{\sigma g c}{\Delta \rho g} \right)^{0.5}} > 0.785 \quad \ldots \ldots \ldots \ldots \quad (4 - 34)
\]
\[
\frac{d_o}{\left( \frac{\sigma g c}{\Delta \rho g} \right)^{0.5}} = \frac{0.006}{\left( \frac{0.013 + 1}{279 \times 9.807} \right)^{0.5}} = 2.753
\]
The velocity through perforations (Orifice) $V_o$:

$$V_o = 2.69 \left( \frac{d_j}{d_o} \right)^2 \left[ \frac{\sigma}{d_j(0.5137 \rho_D + 0.4719 \rho_c)} \right]^{0.5} \tag{4 - 35}$$

$$V_o = 2.69 \left( \frac{1.403 \times 10^{-3}}{0.006} \right)^2 \left[ \frac{0.013}{1.403 \times 10^{-3}(0.5137 \times 730 + 0.4719 \times 1009)} \right]^{0.5}$$

$$V_o = 0.01535 \text{ m/s}$$

If the resulting velocity calculates to be less than 0.1 m/s, $V_o$ should be set at 0.1 m/s $V_o = 0.1$ m/s

4.1.2.1.3 Perforation area, $A_{per}$:

$$A_{per} = \frac{q_d}{V_o} \tag{4 - 36}$$

$$A_{per} = \frac{0.00761}{0.1} = 0.0761 \text{ m}^2$$

$$A_{per} = 0.0761 \text{ m}^2$$

4.1.2.1.4 Number of perforations, $N_o$:

$$N_o = \frac{A_{per}}{\left( \frac{\pi d_o}{4} \right)^2} \tag{4 - 37}$$

$$N_o = \frac{0.0761}{\left( \frac{\pi (0.006)}{4} \right)^2} = 2691.49$$

$$N_o = 2691.5$$

4.1.2.1.5 Plate area for perforations, $A_p$:

$$A_p = \frac{A_{per}}{0.907 \left( \frac{d_o}{p} \right)^2} \tag{4 - 38}$$
\[ A_P = \frac{0.0761}{0.907 \left( \frac{0.06}{0.015} \right)^2} = 0.5244 \, m^2 \]

\[ A_P = 0.5244 \, m^2 \]

### 4.1.2.2 Downspouts:

Set: the continuous-phase velocity \( V_d \) = the terminal velocity of dispersed-phase drop \( V_t \)

\[ V_d = V_t \]

\[ d_P = 0.7 \, mm = 0.0007 \, m \]

\[ V_t = \frac{0.8364(\Delta \rho)_{0.5742} (d_p)_{0.7037} (g)_{0.5742}}{(\rho_c)_{0.4446} (\sigma_g)_{0.01873} (\mu_c)_{0.11087}} \]

\[ V_t = \frac{0.8364(279)^{0.5742} \times (0.0007)^{0.7037} \times (9.807)^{0.5742}}{\left(1009\right)^{0.4446} \times (0.013 \times 1)^{0.01873} \times (0.0031)^{0.11087}} = 0.045 \, m/s \]

\[ V_t = V_d = 0.045 \, m/s \]

### 4.1.2.2.1 Downspout area, \( A_d \):

\[ A_d = \frac{q_c}{V_d} \]

\[ A_d = \frac{0.0022}{0.045} = 0.0489 \, m^2 \]

\[ A_d = 0.0489 \, m^2 \]

### 4.1.2.2.2 Total plate area, \( A_T \):

\[ A_T = \frac{A_p + 2A_d}{0.8} \]

\[ A_T = \frac{0.5244 + (2 \times 0.0489)}{0.8} = 0.77775 \, m^2 \]

\[ A_T = 0.77775 \, m^2 \]

### 4.1.2.2.3 Tower Diameter, \( D_T \):

\[ D_T = \sqrt{\frac{4 \times A_T}{\pi}} \]

\[ D_T = \sqrt{\frac{4 \times 0.77775}{\pi}} \]

\[ D_T = \sqrt{\frac{3.111}{\pi}} \]

\[ D_T \approx 1.122 \, m \]
\[
D_T = \sqrt{\frac{4 \times 0.77775}{\pi}} = 1.00 \text{ m}
\]

\[
D_T = 1.00 \text{ m}
\]

### 4.1.3 Tray Efficiency:

\[
E_o = \frac{N_T}{N_a}
\]

\[\text{........................................................... (4 – 43)}\]

\[E_o = 0.70\]

### 4.1.4 Number of actual stages, \(N_a\):

\(N_T = \) the number of theoretical stages = 7

\[
N_a = \frac{N_T}{E_o}
\]

\[\text{........................................................... (4 – 43A)}\]

\[
N_a = \frac{N_T}{E_o} = \frac{7}{0.7} = 10
\]

\[N_a = 10\]

### 4.1.5 Tower Height:

\[
H_T = (N_a - 1)C_t + \frac{N_a C_t}{10} + 0.1 H_T \text{........................................................... (4 - 44)}
\]

\[0.9 H_T = (N_a - 1)C_t + \frac{N_a C_t}{10}\]

Set: \(C_t = \) the tray spacing = 0.45 m

\[
H_T = \frac{(9 \times 0.45) + 0.45}{0.9} = 5 \text{ m}
\]

\[H_T = 5 \text{ m}\]
Figure (4.8): Single Sieve Plate
### Table (4.7): Recommended Design parameters:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material of Construction</td>
<td>Stainless steel</td>
</tr>
<tr>
<td>Type of column</td>
<td>Sieve plate</td>
</tr>
<tr>
<td>Hole diameter ( d_o )</td>
<td>0.006 m</td>
</tr>
<tr>
<td>Hole pitch ( p )</td>
<td>0.015 m</td>
</tr>
<tr>
<td>Jet diameter ( d_j )</td>
<td>1.403 * 10^{-3} m</td>
</tr>
<tr>
<td>Perforation area ( A_{per} )</td>
<td>0.0761 m²</td>
</tr>
<tr>
<td>Number of perforations ( N_o )</td>
<td>2691.5</td>
</tr>
<tr>
<td>Plate area for perforations ( A_p )</td>
<td>0.5244 m²</td>
</tr>
<tr>
<td>Downspout area ( A_d )</td>
<td>0.0489 m²</td>
</tr>
<tr>
<td>Total plate area ( A_T )</td>
<td>0.77775 m²</td>
</tr>
<tr>
<td>Tower Diameter ( D_T )</td>
<td>1 m</td>
</tr>
<tr>
<td>Tray Spacing ( C_t )</td>
<td>0.45 m</td>
</tr>
<tr>
<td>Stage efficiency ( E_o )</td>
<td>0.70</td>
</tr>
<tr>
<td>Number of theoretical stages ( N_T )</td>
<td>7</td>
</tr>
<tr>
<td>Number of actual Stages ( N_a )</td>
<td>10</td>
</tr>
<tr>
<td>Tower Height ( H_T )</td>
<td>5 m</td>
</tr>
</tbody>
</table>

### Table (4.8): Comparison between Hand Calculations and ASPEN PLUS SOFTWARE

<table>
<thead>
<tr>
<th>Data</th>
<th>Calculation</th>
<th>Aspen Simulation</th>
<th>Error %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of theoretical stages</td>
<td>7</td>
<td>7</td>
<td>0</td>
</tr>
<tr>
<td>Tower Diameter</td>
<td>1</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Stage Efficiency</td>
<td>0.7</td>
<td>0.7</td>
<td>0</td>
</tr>
<tr>
<td>Number of theoretical Stages</td>
<td>7</td>
<td>7</td>
<td>0</td>
</tr>
<tr>
<td>Number of Actual Stages</td>
<td>10</td>
<td>10</td>
<td>0</td>
</tr>
</tbody>
</table>
Chapter five

Conclusion and Recommendations

5.1 Conclusion

In this study, it is shown manual calculations, and ASPEN SOFTWARE in the design of a Liquid – Liquid extraction column (sieve) showed very good agreement, this means that ASPEN can be confidently used for design of separation columns.

First, it is present the extraction of acetic acid from water using isopropyl ether. The graphical method, derived by TREYBAL is used to obtain the number of equilibrium stages.

The data for water, Acetic acid and isopropyl ether were obtained at 25 °C and pressure 1 atm.

The liquid – liquid equilibrium was drawn in the form of ternary graph (TREYBAL METHOD) for the feed containing 35% Acetic acid. The distribution coefficient data were obtained using models available in ASPEN SOFTWARE, thought, it could appreciate the effect of solvent on design of a liquid – liquid extraction column. However, this study definitely gives a firsthand knowledge about the problem and provides a good insight into the complex of the parameters governing the design.

5.2 Recommendations

1. It is recommended that Aspen Plus can be confidently used for the design of extraction columns.

2. The optimum mass-transfer efficiency in sieve tray extractors is obtained at a high velocity of the dispersed phase, but if this velocity is increased more than 85%, the extractor may flood.
References

Appendixes

Steps of running ASPEN PLUS V7.2 - aspenONE windows:

A. Data input windows:
B. Data output windows:

![Image of data output windows]

<table>
<thead>
<tr>
<th>Material</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate</td>
<td>%</td>
<td>10.00</td>
</tr>
<tr>
<td>Water</td>
<td>%</td>
<td>80.00</td>
</tr>
<tr>
<td>Air</td>
<td>%</td>
<td>10.00</td>
</tr>
</tbody>
</table>

![Image of material data]

![Image of simulation output]

![Image of simulation output with details]

![Image of simulation output with details]

![Image of simulation output with details]