# DESIGN AND OPERATION OF MULTIVESSEL BATCH DISTILLATION

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**DOCTOR OF PHILOSOPHY**

**UNIVERSITI MALAYSIA PAHANG**

**2012**

# DESIGN AND OPERATION OF MULTIVESSEL BATCH DISTILLATION

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Thesis submitted in fulfillment of the requirements
for the award of the degree of
Doctor of Philosophy

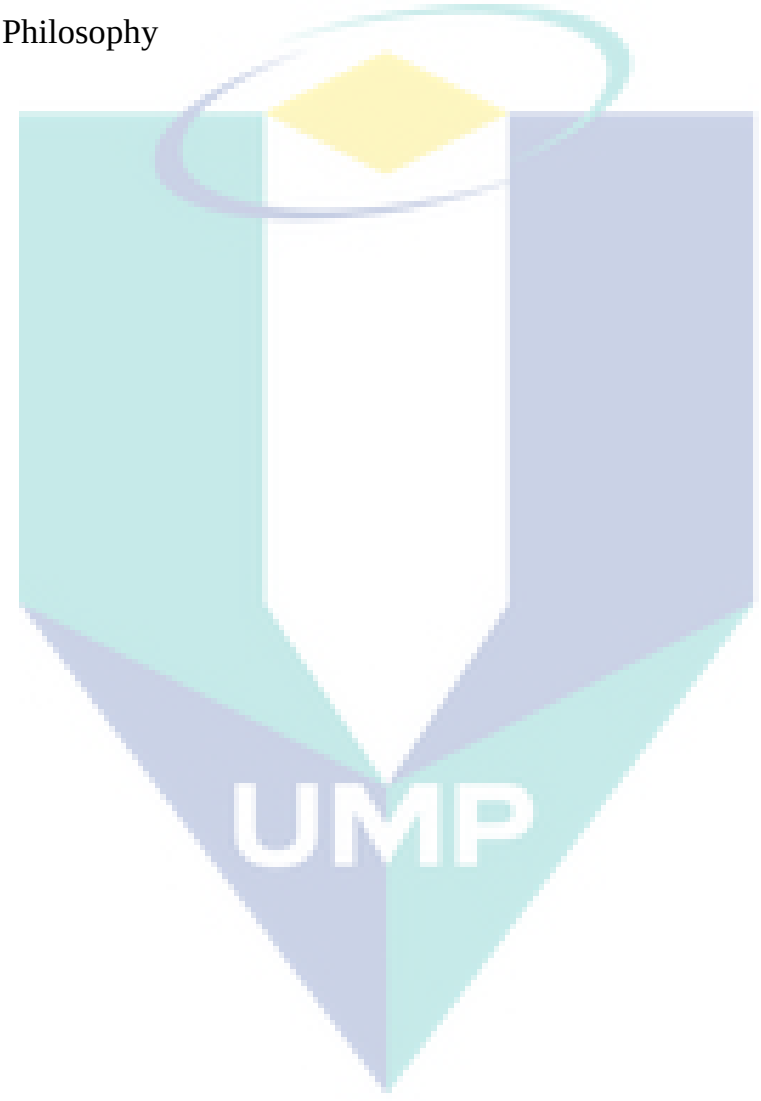
Faculty of Chemical and Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

2012

## STATEMENT OF AWARD FOR DEGREE

1. Doctor of Philosophy

Thesis submitted in fulfillment of the requirement for the award of the degree of Doctor of Philosophy



## 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 Doctor of Philosophy.

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I hereby declare that the work in 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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In the name of Allah, The Most Gracious and The Most Merciful

I humbly dedicate to…

my beloved wife,
Sita Fitriana

my lovely daughters:
Dzikrina Hisyam
Oryzati Hisyam
Raissatifa Hisyam

my mother in peace, my father

my best friends,

those who have colored my life on the right path

thank you…

UMP

## ACKNOWLEDGEMENT

First of all, Praise be to Allah, Lord of the worlds.

In this occasion, I would like to express my deepest gratitude to my supervisor, Dato' Prof. Dr. Rosli Mohd Yunus, for his invaluable advices, suggestion, and courage during my long journey in doing this research. More than just being a supervisor, he also unstoppably gives me broad views of sense of engineering, spirit of creativity, and the meaning of industry-linked research for commercialization.

I will also never forget all the supports of Prof. Dr. Badhrulhisham Abdul Aziz for his total supervision and monitoring on this research. His knowledge and experience on the topic of the research have obviously helped me in completing all the works. Not only in academic problems, he has also helped and guided me in passing the hard way of study. He was the only person I could talk to when the first time I came to UMP- formerly known as KUKTEM- as the first PhD student and the first foreigner. Thank you so much to Prof. Dr. Badhrulhisham Abdul Aziz.

I should also express my deep thanks to Dr. Chin Sim Yee for her kind patience, kindness, understanding, and all the discussions about this research.

In addition, I would like to express my special thanks to Prof. Shinji Hasebe of Kyoto University as the master and pioneer in this research field that has spent his valuable time for me. I really got valuable benefits from him for his kind helps, intensive discussion, deep knowledge, and brilliant ideas for completing this research. It was really grateful when I had opportunity to visit his laboratory and, at the same time, spent time to discuss with him about this research. I was really surprised when he was also available to spend his time to review and check my research report, although he is not an official supervisor. Once again, thank you so much to Prof. Shinji Hasebe.

I also cannot forget all the parties that have supported me, especially my wife, Sita, and all my children, Nina, Ory, and Ifa. They have sacrificed not only to

accompany me during the study, but also support at all time, by any means. I do understand that they have waited for my completion for a long time. I should pay for all the patience, togetherness, and their love. May Allah give rewards to them. My thanks should also be for all my friends that, honestly, cannot be mentioned one by one; more special to Bro Juwari Joesan for his kind help in developing Simulink; all my colleagues in the laboratory and faculty; and everybody that has interacted with me.

## ABSTRACT

Distillation processes in chemical industries have shown significant roles for separating valuable compounds from a mixture. In a specific separation process like separation of perfumes, essential oils, and pharmaceutical products, batch distillation is somehow preferable than continuous one. Batch distillation, however, in these separation processes is advantageous, particularly in the flexibility of fluctuating concentration of feed and products, and in changing production capacity based on market demand. In addition, the separation can be performed in only single batch distillation column for all the changing conditions.

Due to the dynamic behavior of the operation of batch distillation that leads to more difficult operation, such efforts have been done to attain the desired specifications and, at the same time, to increase the operability. The efforts include both the design and operation of batch distillation.

This research focuses on the investigation of separation behavior of multivessel batch distillation for multicomponent mixtures. The distillation unit used in this research consists of two columns with a vessel between the columns, a reflux vessel on the top of the column, and a reboiler (still pot). The experimental data are validated by simulation.

Several cases of separation processes are represented by case studies as follows: Top product recovery, removal of light and heavy impurities, and removal of middle boiling component. By using an ideal ternary mixture consisting of ethanol, 1-propanol, and n-butanol, the case studies were used to investigate the separation behavior. It is found that for the case of top product recovery, increasing the vessel holdup of the middle vessel did not make a significant effect to the increase of ethanol purity in top vessel. However, an increase of ethanol purity in top vessel was obtained by simultaneously increasing the middle vessel holdup and reducing the holdup of top vessel. In this case, reducing the top vessel holdup of 50% results in the maximum achievable ethanol purity in top vessel of 96%.

For the case of removal of light and heavy impurities, the concern was on the trade-off between product purity and product recovery. An agreement was found to have 91% purity of 1-propanol in the middle vessel with recovery of 74%. However, if it is desired to produce high purity product with suffering on the product recovery, reprocessing the top and bottom product can be carried out to recover the middle component in another batch.

In addition, for the case of removing a middle boiling component from a mixture, increasing the middle vessel holdup did not give significant effect to the ethanol purity in top vessel. This may, therefore, be uneconomical to carry out since it leads to more losses of ethanol in middle vessel.

An additional investigation on an introduction of slop vessels in the operation of multivessel batch distillation was also discussed in this thesis through simulation works. For the case studies discussed, it is found that slop vessel holdup and slop vessel position simultaneously give significant effects on the product purity of respective vessels.

# ABSTRAK

Proses penyulingan dalam industri kimia telah menunjukkan peranan yang penting untuk memisahkan komponen berharga dari suatu campuran. Di dalam proses yang spesifik seperti pemisahan bahan wangian, minyak pati dan produk farmaseutikal, penyulingan berkumpulan adalah lebih disukai berbanding penyulingan berterusan. Penyulingan berkumpulan dalam proses pemisahan tersebut mempunyai keunggulan, khususnya dalam fleksibiliti terhadap perubahan komposisi campuran dan perubahan spesifikasi produk. Di samping itu, proses pemisahan yang berbeza dapat dijalankan menggunakan sebuah turus penyulingan sahaja.

Oleh kerana perilaku dinamik daripada operasi pemisahan berkumpulan yang sukar, maka usaha untuk mendapatkan produk yang sesuai dengan spesifikasi yang ditentukan telah dilakukan, meliputi reka bentuk dan operasi penyulingan berkumpulan.

Penyelidikan ini difokuskan kepada kajian mengenai perilaku pemisahan campuran menggunakan penyulingan berkumpulan multivessel. Alat penyulingan di dalam penyelidikan ini terdiri daripada dua turus dan sebuah tangki yang diletakkan di antara dua turus tersebut.

Beberapa kes proses pemisahan di dalam penyelidikan ini adalah seperti berikut: pemulihan produk atas, penyingkiran pengotor (berat dan ringan), dan penyingkiran komponen dengan takat didih tengah. Dengan menggunakan campuran tiga komponen yang ideal meliputi ethanol, 1-propanol, dan n-butanol, kes-kes proses pemisahan digunakan untuk mengkaji perilaku pemisahan. Daripada kes pemulihan produk atas, didapat bahawa dengan meningkatkan isipadu tangki tengah, tidak ada kesan peningkatan kepekatan ethanol dalam tangki atas. Walau bagaimanapun, peningkatan kepekatan ethanol dalam tangki atas diperolehi dengan menambah isipadu tangki tengah dan secara bersama-sama mengurangi isipadu tangki atas. Sebagai contoh, mengurangi isipadu tangki atas sebesar 50% dapat meningkatkan kepekatan ethanol sehingga 96%.

Untuk kes penyingkiran pengotor dari tangki tengah, perhatian diberikan kepada trade-off antara kemurnian produk dan pemulihan produk. Sebagai contoh, kemurnian produk 1-propanol sebesar 91% didapati pada kadar pemulihan 1-propanol sebesar 74%. Walau bagaimanapun, pemrosesan semula produk atas dan produk bawah masih mengandungi pengotor ethanol dan n-butanol.

Sementara itu, pada kes penyingkiran komponen dengan takat didih tengah daripada suatu campuran, peningkatan isipadu tangki tengah tidak memberikan kesan yang ketara terhadap kepekatan ethanol pada tangki atas.

Di samping kajian yang dilakukan untuk mempelajari perilaku pemisahan dalam penyulingan berkumpulan multivessel, penyelidikan ini juga membuat kajian terhadap penggunaan slop vessel yang dijalankan dengan simulasi.

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# NOMENCLATURE

| | |
|---|---|
| $x_n$ | : component mole fraction of liquid in stage “n” |
| $y_n$ | : component mole fraction of vapour in stage “n” |
| $L$ | : liquid molal flow rate (mole/min) |
| $V$ | : vapour molal flow rate (mole/min) |
| $H$ | : holdup of top vessel (mole) |
| $M$ | : holdup of middle vessel (mole) |
| $B$ | : holdup of bottom vessel (mole) |
| $H_s$ | : stage holdup (mole) |
| $K_i$ | : equilibrium constant |
| $p^*$ | : saturated vapour pressure (bar) |

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# CHAPTER 1

# INTRODUCTION

## 1.1 BACKGROUND

The use of distillation in separation processes has widely been applied in chemical industries utilizing its ability to perform separation based on boiling point difference of components in a mixture. Fractionation of crude oil is the most obvious application of distillation. Other applications of this separation method can be shown in the production of ethanol, acetic acid, and solvents removal. Furthermore, distillation technique has also been employed in pharmaceutical industries such as concentrating antibiotics with methanol recovery, butanol recovery from broth extracts, *et cetera*. The ability of distillation to perform separation leads to the utilization of distillation column in a wide range of separation. That is the reason why distillation has attracted many researchers to carry out studies and investigations to obtain the desired products.

In modern era, such studies and improvements of the basic concept of distillation have been carried out by academicians and researchers, which have resulted in a lot of types of distillation columns. In term of operation, however, distillation column can be generally categorized into only two types: batch distillation and continuous distillation. Simple batch distillation has been known as the oldest separation technique, while the continuous distillation is a result of its improvement. However, those types of distillation are apparently different in operation as shown in Figure 1.1.

Batch distillation is the term applied to equipment, into which the raw liquid mixture is admitted and then boiled for certain duration of time. Volatile components are collected from the top product distillate. At the end of the distillation time, the liquid remaining in the still is withdrawn as the residue. In some cases the distillation is continued until the boiling point reaches some predetermined level, thus separating a volatile component from a less volatile residue. In other cases, two or more fractions

can be withdrawn at different times and these will be of decreasing volatility. During batch distillation, the concentrations change both in the liquid and in the vapor.

Despite batch distillation is considered as the origin of what is presently known as distillation separation technique, it has not been more popular than the continuous one since the continuous distillation is more feasible, effective, and less energy consuming for large scale industries. However, batch distillation has risen again in recent decades due to its advantages in the specific purposes of separation. Some of the advantages offered by batch distillation include:

1. Batch distillation is suitable for separation of seasonal campaign of products. Certain chemical products have fluctuated in market demand, and then batch distillation can be operated flexibly following the demand. This advantage is considered by pharmaceutical industries as the most flexible separation device.
2. Batch distillation can be operated to separate a mixture containing solid materials such as in the waste treatment and solvent recovery.
3. It is also advantageous to use batch distillation for purifying a mixture containing a large number of components by using only single column. This can be done because of the ability of batch column to deal with varying feed composition without changing the equipment. Shortly, more than one product can be produced from only single column of batch distillation.

Although the concept and the operation of batch distillation are much simpler than the continuous distillation, there are many problems encountered by designers and operators. Some of the problems include:

1. The conditions are not constant (unsteady state), which leads to the complexity in instrumentation and control.
2. The calculation is more difficult due to its dynamic processes.
3. The batch operation must be handled appropriately in every single step of process; therefore manpower cost could be higher.

However, in many industries producing specialty chemicals, batch distillation has essential roles. Luyben (1989) has predicted that batch column will become more important in the future as the chemical plants shift to more small-volume and high value products. It can be explained by the fact that although continuous distillation has been receiving big attention for decades due to the expansion of petrochemical-based industry, the trend of chemical industry has led to the revival of the importance of batch distillation. In recent years, therefore, an attention to batch distillation has increased significantly, particularly for separation of fine and specialty chemicals.

Improvement of the operation of batch distillation is not only on the operating condition, but this includes also in the equipment design. Several researchers have proposed new designs of batch distillation, especially to deal with specific separation problems. Originated from simple batch distillation, novel designs have been invented. One of the most frequently investigated design is what is called as multi-effect batch distillation, which is batch distillation with a vessel located between the columns, single boiling pot, and single condenser (Hasebe *et al*, 1992).

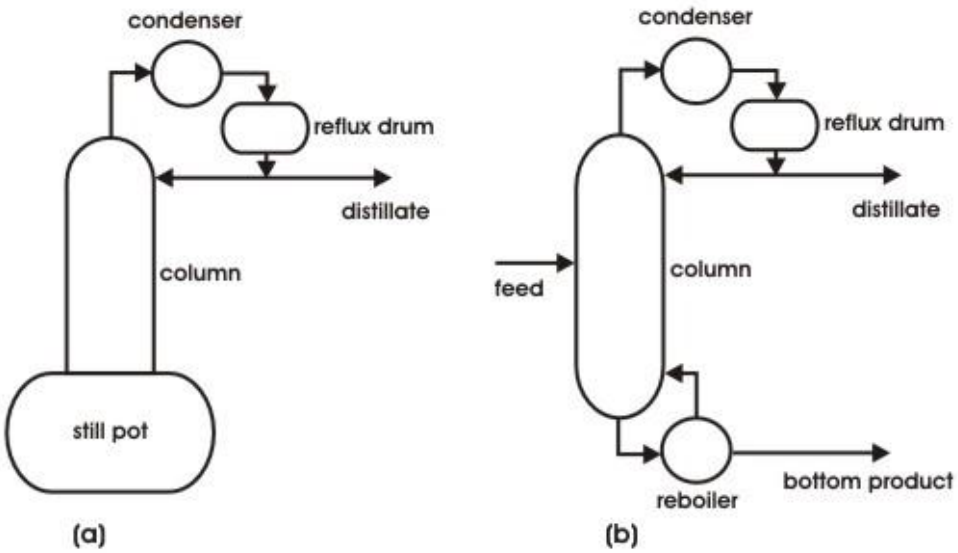


**Figure 1.1** Schematic picture of (a) batch column, and (b) continuous column

## 1.2 OPERATION OF BATCH DISTILLATION

According to the advantages of batch distillation compared to the continuous one for certain purposes of separation, some chemical industries have chosen batch distillation for their separation processes. Pharmaceuticals, fine and specialty chemicals, and food industries commonly utilize batch distillation. However, some issues on the batch distillation application have come up, especially from industrial experience. The issues include:

1. Batch distillation usually consumes more energy. It is driven by how long the operation is run. Because batch distillation is operated in an on-off mode due to its seasonal campaign, more energy is also consumed for start up.
2. Due to the changing (decreasing) product composition, reflux ratio becomes very important to keep optimal to meet the desired composition. But, on the other hand, increasing the reflux ratio will lead to the longer operating time, which means higher energy consumption.
3. In fact, batch distillation columns work in a very dynamic process where almost all the parameters change with time. As a consequence, therefore, a process control must be very reliable.

Dealing with specific purposes of separation using batch distillation, it is necessary to operate batch distillation in such a way so that the targeted product composition is achieved. Re-processing the products, either top or bottom products, can be chosen. That is called as operation planning of batch distillation. By using only single column, a series of separation tasks can be done. Another alternative, however, is also necessary to consider of using the so-called non conventional batch distillation column or complex batch distillation. By utilizing it, a simpler operation or less energy consumption may be obtained.

## 1.3 COMPLEX BATCH DISTILLATION

Recently, one has re-examined the operation of batch distillation as a whole. Several alternative column configurations have been suggested. They primarily differ in

the position of the major liquid holdup in the column. This leads to the so-called complex batch distillation, which mainly could be inverted, middle vessel and multi-vessel column configurations.

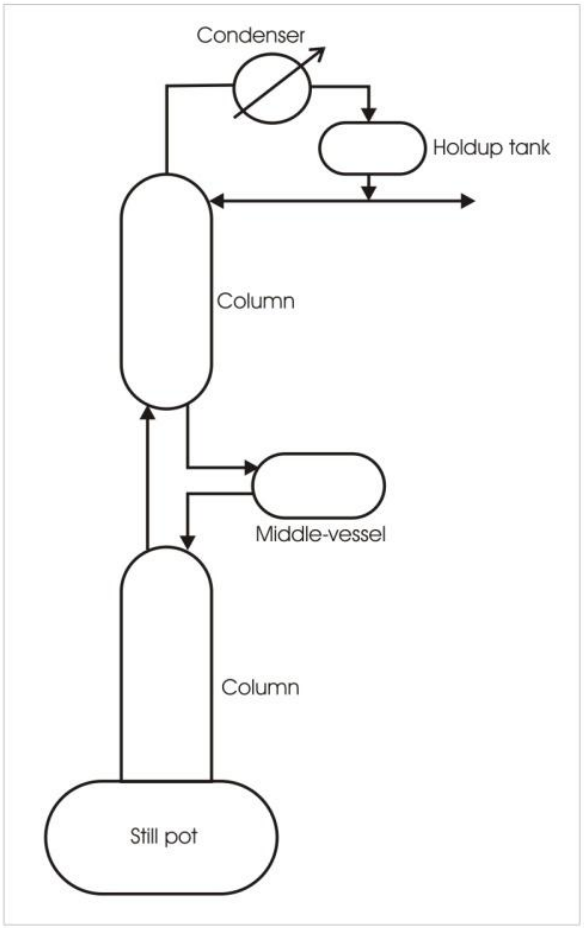


**Figure 1. 2** Multivessel batch distillation (one top vessel and one middle vessel)

This research emphasizes the operation of multivessel batch distillation under total reflux with varying holdup of the vessels. More specifically, the study is limited to the investigation of varying holdup with already filled vessels. The operation of this mode is simple, but it needs a good understanding of multi vessel column operation. The varying volume of vessel holdup may have significant effects on the separation performance, especially if the column is run under total reflux. From a simple logic, when steady state is reached after certain duration time of operation, every single substance will go to its own volatility according to its portion in the feed, respectively. At the end of the operation, most volatile substances will dominate the composition of

product in the reflux drum, and *vice versa*, heaviest substances will be most in the reboiler; while middle vessel is dominated by middle substances. If the volume of reflux drum holdup is set relatively lower than the amount of component A, for instance, in the feed, it can be predicted that component A will stay in that vessel dominantly after steady state condition is reached. During the operation, no product is withdrawn from the column. The final products are collected from the vessels, but however, only the reflux drum is commonly expected to produce the desired product for a batch.

## 1.4 PROBLEM STATEMENTS

The research has been inspired by an idea to ease the operation of a batch column without losing the objective to attain the desired product quality. Simplifying the complexity will lead to the absence of process control, or at least minimum requirement, as there is almost nothing to control anymore when total reflux policy is employed. In common distillation processes, reflux ratio is the most and the easiest parameter to control to reach specified products composition. Furthermore, in batch processes, process control has become one of most important issues since the process is dynamic, in which the component properties always change with time. This idea to reduce the role of process control in a dynamic condition might become attractive, both economically and in the operation.

Operating batch distillation column in a total reflux mode leads to a steady state condition of the process, in which the component properties do not change anymore. Since reflux ratio has been determined constant in this mode, thus the only parameter that needs to be considered is the operating time. In multivessel batch distillation operation, the product purity in the product vessel will only depend upon two factors: operating time and product vessel holdup. Once the vessel holdup has been specified, then the maximum possible product purity will only depend upon the operating time. Nevertheless, the key point in this work is on the introduction of the variable holdup product vessel.

Based on the operation issues as mentioned above, thus understanding the separation behavior of multivessel batch distillation under total reflux policy with

variable holdup product vessel is necessary. Composition profile of the components in each product vessel should be known to determine how long the operation is run. Furthermore, a strategy of separation can be determined by knowing the composition profile in the product vessels.

## 1.5 RESEARCH OBJECTIVES

The main objective of this research is to study separation behavior of multivessel batch distillation under total reflux policy. Deriving the main objective, several practical goals were determined as follows:

1. To design and fabricate a multivessel batch distillation. This work will result in a said new design product vessel, by which the vessel holdup is changeable.
2. To study the operation of multivessel batch distillation for separating non-azeotropic mixture under total reflux policy. The study includes:
    a. Performing simulation works for the separation of ternary non-azeotropic alcohol mixture utilizing Simulink environment in Matlab platform.
    b. Performing experimental works for the separation of ternary non-azeotropic alcohol mixture using a multivessel batch distillation to verify the simulation.
3. To investigate the introduction of slop vessels along the columns for the separation of ternary non-azeotropic alcohol mixture in a multivessel batch distillation under total reflux policy with varied vessel holdup.

## 1.6 SCOPE OF THE RESEARCH

The scope of this research includes experimental and simulation works. The experiments cover the separation of ternary alcohol mixture using multivessel batch distillation with two columns and one middle vessel without any additional slop vessels (*i.e.* batch distillation with a middle vessel), while the simulation covers the separation of similar mixture to the experiments with several column configurations. The configuration includes introducing slop vessels that act as slop cuts in regular batch distillation.

The simulation works start from mathematical models development based on material balance equations of the multivessel column. Such assumptions of the model are applied as mentioned later in Chapter 3 of this research thesis. The mathematical models are translated to programming language in m-file form of Matlab. In this step, Simulink of Matlab platform is used for the simulation due to the fact that it is advantageous for dynamic system simulation.

The simulation covers the separation behavior studies by investigating case studies representing several actual cases:

1. Recovery of light component in small concentration as top product (0.16, 0.42, 0.42)
2. Removal of light and heavy impurities (0.21, 0.66, 0.13), while the middle component was intended as the product
3. Removal of middle boiling component in small concentration (0.55, 0.10, 0.35)

The above case studies are also verified by experimental works. The experiments are carried out to validate the simulation results based on the case studies.

However, an additional set of simulation is also performed for the introduction of slop vessels along the column to examine the effects of introducing slop vessels to the separation behavior. The simulation works are able to provide information of the concentration profile in the product vessels as a function of the operating time.

In addition, the design and fabrication of the experimental rig were also covered in the research since there was no existing apparatus available in the laboratory.

## 1.7 RESEARCH NOVELTY AND CONTRIBUTION

In fact, there have been only a few numbers of researches in the area of multivessel batch distillation since Hasebe *et al*. (1992) pioneered the study. Most of the studies only covered the simulation parts, while experimental works are very rare. In this country, to date, yet the study on multivessel batch distillation has not been found.

Therefore, this research would become the first one in the country. In addition, physically the equipment that has been fabricated from this research would be the pioneer at universities and research centers and even in industries in Malaysia. However, the benefits of the research could be spread out over the world.

In international, in spite of several researchers with research on complex batch distillation column, the idea to design the variable-holdup vessel has not been yet found. This new design of the vessel will allow the batch distillation column much easier to operate. It will add one more element of flexibility to the batch column, *i.e.* capacity.

It is obvious now that this research will give real contribution either to academics and industries. Academically, this research is expected to inspire other researchers to further investigate the new design to obtain more improved design. First, this research reveals a new design on the product vessel of multivessel batch distillation, by which the product holdup (*i.e.* product capacity) can be easily adjusted. Secondly, slop vessels along the column are introduced to increase the maximum product purity achievable. The simulation of this study gives quite general figure of separation using multivessel batch distillation under total reflux policy by including the introduction of slop vessels.

## 1.8 BENEFITS OF THE RESEARCH

The design and fabrication of the multivessel batch distillation would give real benefits to the university, particularly Universiti Malaysia Pahang, for teaching and research purposes in the future. This benefit will allow further research on the separation processes, especially the separation of food products, natural products, pharmaceuticals, which are more suitable to be processed in batch mode. More importantly, these products have been determined as superior research focus in the country.

Technically, operating a multivessel batch distillation under total reflux leads to simpler operation and lower cost. Firstly, the separation unit with total reflux operation

can be operated very much simply. Products can be collected from the vessels without worrying that the composition decreases with time. The product composition in the product vessels tends to go to a steady state condition after a certain period of operation. The worst case if the operating time is too long is that the operation will consume higher energy. Secondly, the total cost is relatively small since there is no critical need of process control system in the unit, *i.e*. sequential process control system. There is almost nothing to control. Once the separation behavior of a certain mixture has been identified, then by setting the operating time the separation can be done very easily. Both the capital cost and the operating cost are relatively lower compared to the conventional batch column.

Introducing a new design of product vessel, by which the product volume can be adjusted as the separation strategy, and the slop vessels for compensating undesired components obviously allows the multivessel batch distillation to be more flexible in operation. This would be much more beneficial for the separation processes in dealing with changing feed composition and fluctuating product purity. Therefore, there will be more options of the strategy of separation.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 INTRODUCTION

Batch distillation has received extensive attention over the past few years, partly due to the fact that it is frequently used in the production of small quantities of chemical and biochemical products of high value. Its main advantage in this context is its versatility or flexibility.

This chapter presents a review on the batch distillation researches that have been carried out within the last few years. This includes conventional or regular batch distillation and the complex design and operation of batch distillation column. More specifically, some parts of the review will emphasize the operation of batch distillation with intermediate vessels, such as middle vessel batch distillation and multivessel batch distillation.

## 2.2 REGULAR BATCH DISTILLATION

Batch processes are, sometimes, preferable to the continuous processes as discussed in the previous parts. For instance, batch process can be developed faster, which is important for new products to establish a market share rapidly. Batch processes allow flexibility in scheduling demands, which is also important for products with uncertain demands, and also for the production of multiple products using the same equipment.

Batch distillation is an alternative of choice for processing specialty and fine chemicals as well as in the production of flavors, fragrances, pharmaceuticals, dyes, and some other products, which are high value products to be produced in small volume (Salomone, 1997). Compared to continuous distillation, batch distillation is more suitable for small production due to its flexibility.

As in the case of simple distillation, a fixed quantity of liquid is originally charged to the batch still. During distillation process, the vapor passes upward through the column. The whole column is an enriching section, so that regular batch distillation is also called as enriching column. The vapor is condensed into liquid at the top of the column. Part of the liquid is returned back to the column as reflux, and the remainder withdrawn as distillate.

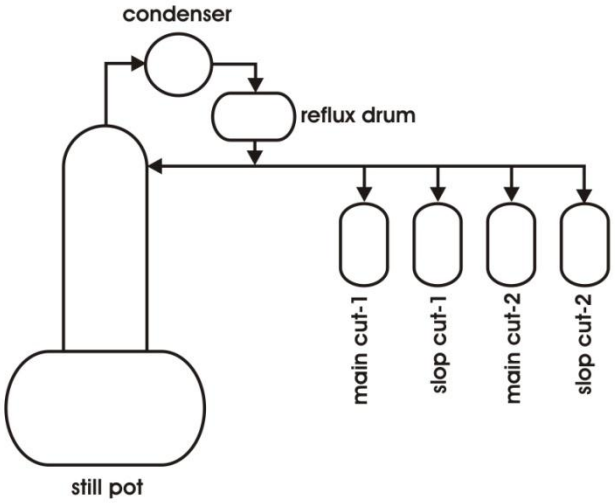


**Figure 2. 1** Batch distillation column with main cuts and slop cuts

As schematically shown in Figure 2.1, the top products are collected in the accumulators. Main cuts are used to accumulate the products that satisfy the specification, while slop cuts are used to accommodate the off specs products that can be re-processed again in the next batch operation. For multicomponent separation in a batch distillation, main cut-1 is used to accumulate the lightest product (most volatile), main cut-2 for the second most volatile, and so on. Between the two pure products obtained from the operation, there is an intermediate cut that works to accumulate the transition between product-1 and product-2. It is called as slop cut. This cut contains a mixture of the two products, and as a consequence, it cannot be considered as product cut. The purity of the components in the slop cut does not meet the specification for the two products, respectively.

In a batch process, the main steps are operated in a discreet mode. In contrast with a continuous process, a batch process does not deliver its product continuously but in discrete manner. This means that mass, temperature, concentration, and other

properties vary with time, thus the batch distillation process is one of dynamic processes in chemical industry.

Analysis of dynamic system in batch distillation plays an important role due to the fact that batch distillation works in dynamic mode in nature. Elgue *et al.* (2004) studied the start up operation of batch distillation column. He emphasized the start up operation since this is the most dynamic in batch processes. As shown in Figure (), Elgue *et al.* (2004) presented the analysis of the temperature record that offers dynamic information about both hydrodynamic and thermodynamic variables.

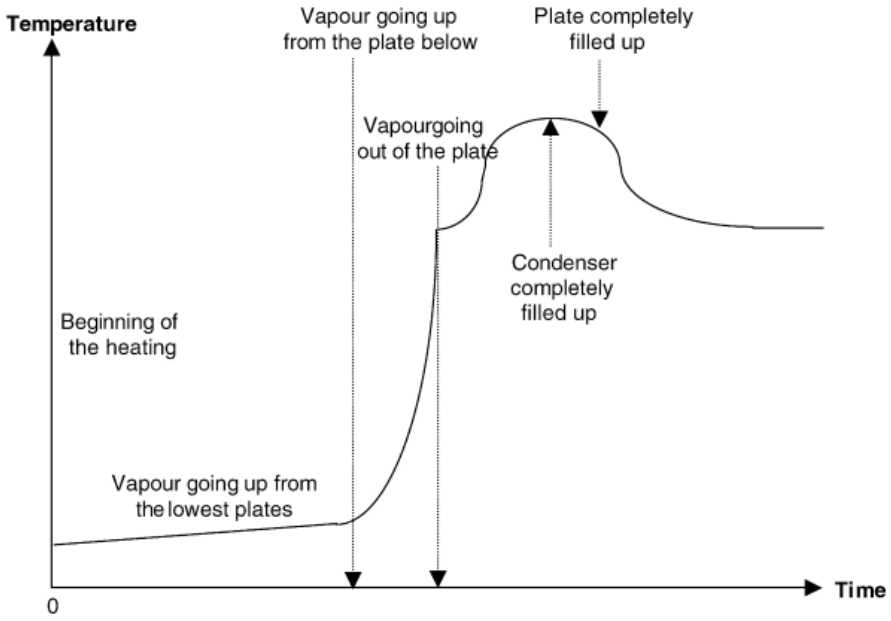


**Figure 2. 2** Analysis of tray dynamics studied by Elgue *et al.* (2004)

The study of the dynamics of a simple batch distillation usually assumes the equilibrium between the liquid and vapor phases. In contrast to this approach, Silva *et al.* (2003) investigated the dynamics behavior of non-equilibrium simple batch distillation. He studied the dynamics behavior through simulation works based on a mathematical model and singular points for ideal solutions. He used a ternary mixture consisting of propane, n-butane, and n-pentane for representing the most volatile, intermediate boiling component, and the least volatile, respectively.

### 2.2.1 Operation policy

Determining the best way of running a batch distillation column is important in order to improve the efficiency of energy consumption and the product quality, to reduce production time and to reduce waste cuts. Since the number of trays has already been determined, then the only effort to attain the target is setting up the operation mode or operation policy. The most common parameter in the operation policy of a batch column is reflux ratio.

Batch distillation is usually operated with production cycles consisting of charging, heat-up, equilibration, product draw-off, slop cut (off-cut removal), product draw-off, cool-down, dumping and cleanup. The size of the off-cuts (if any) depends on the sharpness of separation between the products. The sharpness of separation depends on holdup, relative volatility, reflux and number of plates or height of packing. Intermediate cuts are often recycled (Miladi and Mujtaba, 2004). Furthermore, the authors explained in more detail the operation of regular batch distillation as described in the following steps:

1. The reboiler is charged with the material to be processed and heat is applied to it to bring the material to its boiling point temperature.
2. Depending on the reboiler duty, a part of the material is vaporized and the vapor travels upward both through the plate holes and downcomers and almost instantly reaches the condenser.
3. At this time, the coolant valve is opened and the condensed liquid is stored into a reflux drum. The reflux valve is opened when the liquid fills the condenser holdup tank. At this point some products may also be collected simultaneously.
4. The liquid begins to flow into the top plate and collects on the plate because of the retention made by the vapor flow. When the liquid level passes the weir height (thus filling the holdup), the liquid begins to fall to the plate below and the same phenomenon is repeated until reaching the reboiler.

5. If no product was withdrawn in step 3, the column is now run under total reflux operation until the unit is taken to a steady state or to a state when the distillate composition reaches the desired product purity.

In term of batch distillation operation, there are various modes of operation of the different batch distillation columns as explained by Miladi and Mujtaba (2004). The conventional operating policies for regular batch distillation columns are:

1. Constant reflux ratio (variable distillate composition). The reflux rate is made constant with a consequence that the distillate purity decreases by the time.
2. Constant distillate composition (variable reflux ratio). In contrary with the constant reflux ratio policy, the constant distillate composition will lead to the varied reflux ratio to keep the distillate purity constant.

However, there is another operation type that is actually a trade-off of the above two types. This is called as optimal reflux or variable reflux. An optimal reflux policy is chosen so that the objective functions are satisfied (minimum time, maximum product, maximum profit, *et cetera*), subject to any constraints at the end of the process.

For multicomponent separation in regular batch distillation, the profile of products composition is typical as shown in Figure 2.2. The lightest component evaporates first, followed by the second lightest, and so on. When the first component composition starts to decrease, at the same time the second component composition begins to increase. There is a certain time where the product composition does not meet the specification; it is called as slop cut. At this time, the product mainly consists of a mixture of the first and second components.

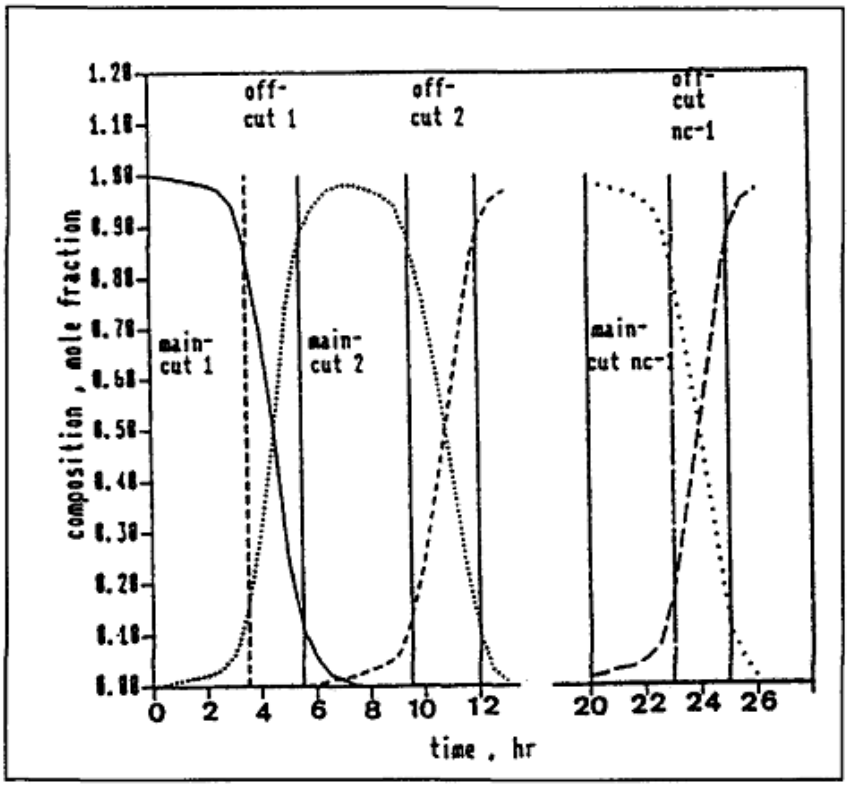


**Figure 2. 3** Typical instant distillate composition profile of regular batch distillation (Miladi and Mujtaba, 2004)

In addition, Bonsfills and Puigjaner (2004) studied the simulation of batch distillation and verified the results by experimental data. They developed a mathematical model based on mass balances and vapor-liquid equilibrium equations to simulate several case studies including methanol-water, cyclohexane-toluene-chlorobenzene, toluene-n-butanol, and toluene-n-butanol-n-octanol mixtures, respectively, with various values of reflux ratio. The authors considered that the model developed based on mass balances and vapor-liquid equilibrium equations is a rapid and reliable model that has been extensively validated with satisfactory results. It can be applied to multicomponent mixtures and also binary azeotropic mixtures.

Furthermore, a study of operating parameters of batch distillation was also carried out by Bonny (1999) that presented the problem of the optimization of only single multicomponent batch distillation by taking into account a new operating parameter corresponding to the start of the collection of product cuts and by the use of variable reflux ratio. He also introduced the policy of switching product tanks.

### *2.2.1a Constant reflux with changing distillate composition*

With the constant reflux ratio policy, the reflux ratio is maintained constant throughout the batch operation. It is the simplest policy to implement, but less efficient than the other variable reflux ratio policies. If a batch distillation is to be run at constant reflux, then it follows that the distillate product composition must change with time. Normally, the constant reflux policy is operated with an initial period under total reflux.

### *2.2.1b Constant distillate composition with variable reflux*

With the constant distillate composition policy, the product is usually withdrawn at the maximum possible rate consistent with there being sufficient reflux to maintain the product composition at the desired value. As the batch progresses, the composition of the product tends to deteriorate and the reflux ratio is increased gradually by reducing the product withdrawal rate, until the desired composition can no longer be maintained. At this point, the product is diverted to another receiver and an intermediate cut (off-cut) is withdrawn usually at a constant high reflux ratio. This procedure is repeated until all the product fractions are removed. If the next batch contains the same mixture components, the off-cuts are usually returned to the still together with fresh charge. According to Skouras and Skogestad (2004), the constant distillate composition policy is found to be by far the most time consuming. It is clearly understood that the constant distillate composition policy requires variable reflux ratio, which does not affect the distillate composition instantly.

### *2.2.1c Optimal reflux ratio*

This policy denotes that the reflux flow rate is optimized as a function of time during the operation. An optimal reflux is chosen when distillate purity has been reached as desired. Sharif *et al.* (1998) summarized the time profile of reflux ratio under this mode could take the shapes of linear, polynomial or exponential or even combination of them with at least one of the following objectives:

1. To maximize distillate. Maximizing the amount of product of a specified purity produced over a prescribed period of time.
2. To minimize time. Minimizing the amount of time required to produce products of a specified quantity and quality.
3. To maximize profit. Maximizing an economic profit function subject to product purity and quantity constraints.

Various methods of optimization are applied to solve the above problems of optimal reflux ratio mode. Almost all researchers have agreed on the superiority of optimal reflux ratio policy. However, there are yet some debates on the amount how much the benefits could be obtained from this policy.

Among the typical operation policies mentioned above, Sorensen and Skogestad (1994) proposed an unusual operating mode, which is called as cyclic policy. In their study, they compared the cyclic operating policy with the conventional operating policies in terms of minimum operating time. The term of cyclic means the external reflux and distillate flows are cycled. This operation is characterized by repeating three periods of operation: filling up, total reflux, and dumping. Later, Sorensen (1999) presented a more comprehensive review on the cyclic operating policy, both in theory and practice. She once again stressed the advantages of this policy compared to conventional schemes:

1. It achieves the maximum attainable separation in the column.
2. There is a minimal need for control.
3. It is less sensitive to disturbances and, therefore, safer to operate.

### 2.2.2 Model formulation of regular batch distillation

Basically, regular batch distillation is simply an evaporator with column section. The still (reboiler) is charged with a liquid feed which is then boiled. Vapor from the reboiler enters the bottom of a rectification column with certain number of theoretical stages and a total condenser which yields a saturated liquid product along with the reflux (Doherty and Malone, 2001).

Many researchers have developed mathematical model of batch distillation. Al-Tuwain and Luyben (1991) studied a short-cut method based on mass balances, constant relative volatility and mathematical correlations to design batch distillation columns. Diwekar and Madhavan (1991) also presented a rapid method to design batch distillation columns, based on the equations of Fenske-Underwood-Gilliland (FUG) and constant relative volatility. Mujtaba and Macchietto (1993) have mainly focused on searching for the optimal operation strategies in batch distillation. Surprisingly, based on their studies on optimization problems, their works concluded that optimal reflux ratio is mostly close to the constant reflux ratio. Even, Mujtaba and Macchietto (1995) investigated simultaneous optimization of design and operation of multicomponent batch distillation for single and multiple separation tasks.

In more recent years, Barolo and Botteon (1997) worked with a short-cut model with constant relativity for a column at infinite reflux ratio in order to obtain theoretically pure components of a binary mixture. In addition, Sharif *et al.* (1998) proposed solving the model using the Fenske-Underwood-Gilliland (FUG) method assuming equimolar overflow, constant relative volatility, and negligible column holdup. Moreover, the authors also presented an algorithm for the simultaneous design and operational optimization of batch distillation column.

General model developed is based on mass balance and equilibrium vapor-liquid equations. Figure 2.4 depicts a schematic representation of the batch distillation. It is applicable to both multi-component and binary mixtures. The model is based on the following assumptions:

1. A total condenser is employed.
2. The molar flow rate is constant.
3. The molar holdups in the liquid phase for each theoretical stage are constant.
4. The molar holdup in the vapor phase is neglected.
5. Vapor and liquid on any particular theoretical stage are in equilibrium.
6. Adiabatic column and theoretical trays

Based on Figure 2.4, a set of mathematical equations of regular batch distillation was summarized by Mujtaba (1994).

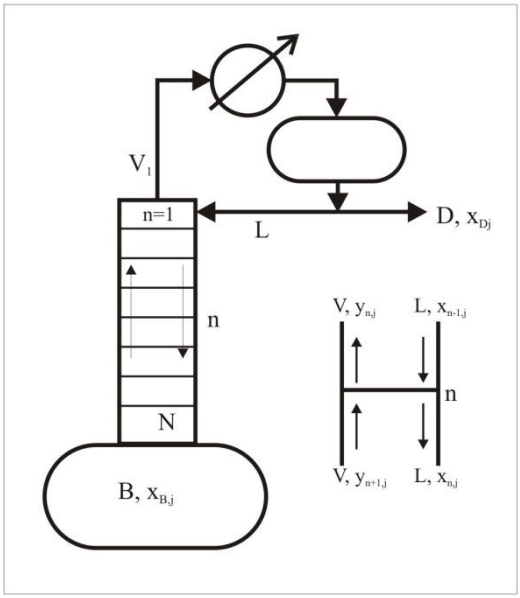


**Figure 2. 4.** Schematic representation of batch distillation column

### 2.2.3 Multiple tasks batch distillation

The use of regular batch distillation for product recovery from a multicomponent mixture was also deeply studied by Zamar *et al.* (1998). The authors presented a method for the preliminary design of separation networks of batch distillation. Analytical partition functions are derived based on the assumption that the distribution of components in an actual batch distillation can be approximated by the distribution of a batch column at a total reflux. They, therefore, stated that this method permits computation of mass balances of multiple separation networks (allowing recycle of intermediate cuts) by solving a linear system of equations. Figure 2.5 shows an example of a network of separation tasks developed by Zamar *et al.* (1998).

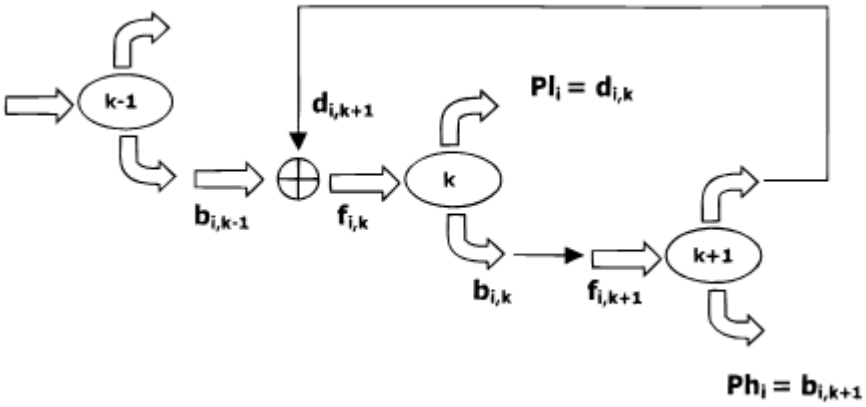


**Figure 2. 5** A network of separation tasks (Zamar *et al.* (1998)

The authors claimed that the form proposed for the mass balances with task balances and connecting equations is helpful in reducing the programming efforts for the setup of new cases to explore.

## 2.3 COMPLEX BATCH DISTILLATION

More complex design of batch distillation had actually been initiated by Robinson and Gilliland back in 1950 (Barolo *et al*, 1996), but however, a higher attention on the complex column appeared when Hasebe *et al.* (1992) re-invented the complex batch distillation column as shown in Figure 2.4. Since that time, other researchers started to investigate both design and operation of complex batch distillation.

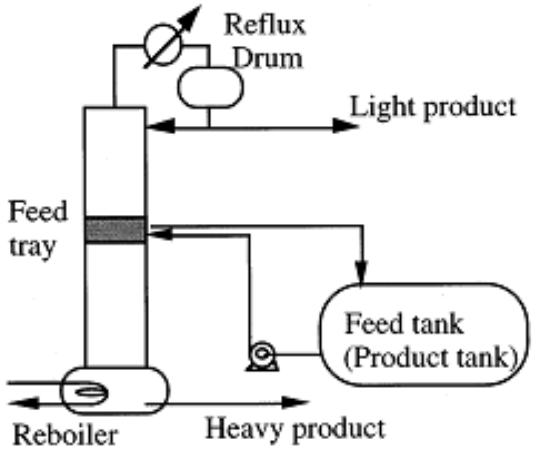


**Figure 2. 6** Complex batch distillation column proposed by Hasebe *et al.* (1992)

Mujtaba and Macchietto (1992) discussed the use of a complex column to improve the operation of reactive batch distillation. They found that this column configuration improved conversion and product yield significantly when the reaction products had two extreme boiling points (highest and lowest in the reaction mixture). The use of an inverted column for cases where the reaction product had a higher boiling point than the reactants was suggested but no examples were given. Mujtaba and Macchietto (1994) discussed the use of inverted and complex columns for an example with reactive batch distillation. For this example, the inverted column gave a lower conversion than the regular column. This was explained in terms of the difference in relative volatility between the heavy components compared to between the light ones. They performed comparative studies between conventional and complex columns under optimal operation. A theoretical analysis of the dynamic behavior of a complex column has been presented by Davidyan (1994) and by Meski and Morari (1995) using a simplified column model with no plate holdup and an infinite number of stages in both column sections. Safrit *et al.* (1996) examined the potential of a middle vessel column to separate azeotropic mixtures. In order to separate azeotropic mixtures in general, a sequence of batch columns must normally be used.

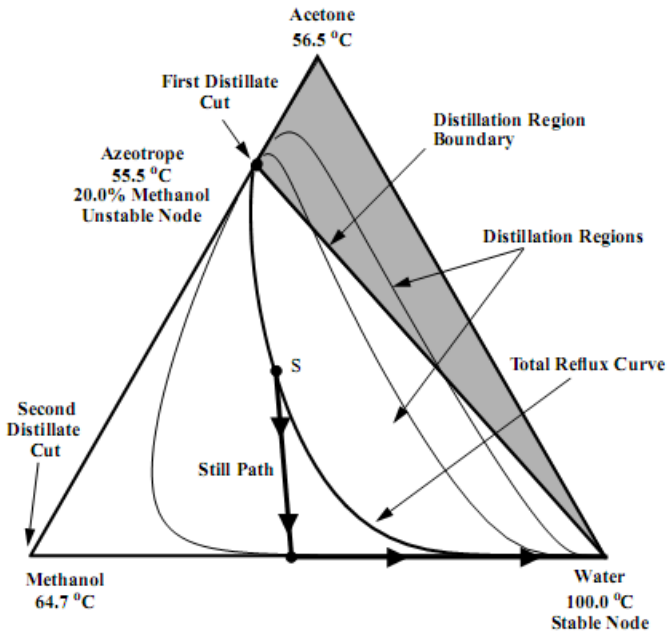


**Figure 2. 7** Product sequence of methanol-water-aceton mixture separation studied by Safrit (1996)

Many complex designs have been invented by researchers. The use of multiple heat-integrated complex columns was suggested by Hasebe *et al.* (1995) as an alternative to a train of continuous columns for the separation of multicomponent systems. This kind of "multivessel" batch column has been further studied by Skogestad.(1995), who considered the potential of this configuration with respect to a conventional batch column and proposed a strategy for product composition control.

In addition, Barolo *et al.* (1996) studied the dynamics and the operation of a pilot plant complex batch column that were analyzed by experiments. The main characteristics of the complex column were shown by comparison with the behavior of a conventional rectifying batch column. In another article, Barolo *et al.* (1996) also presented some issues in the design and operation of batch distillation with a middle vessel. He and the co-authors discussed the issues either in the operation, design, and control.

### 2.3.1 Theoretical foundation of multivessel batch distillation (MVBD)

A multivessel batch distillation (MVBD) column has very similar configuration to that of a conventional batch distillation but with one or more intermediate vessels (product vessels) as shown in Figure 2.6(b). Hasebe *et al* (1997) illustrated more general multivessel batch distillation, which is called as multi-effect batch distillation (MEBAD), with several columns and vessels between the consecutive columns. As shown in Figure 2.8, heat is only charged into the vessel 1, which is actually a reboiler/still pot. A condenser is installed at the top of the least column. Between two columns, there is a vessel.

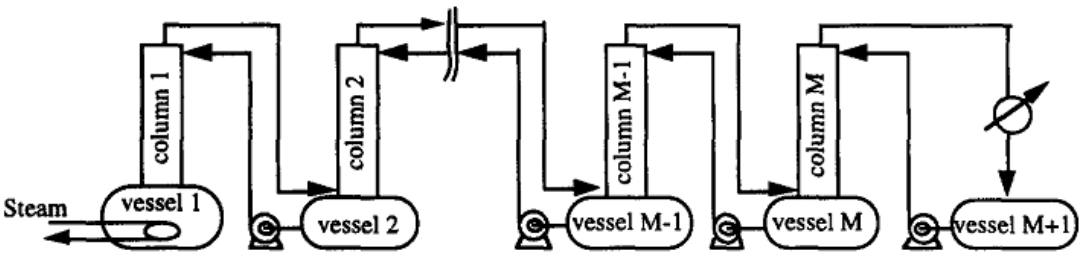


**Figure 2. 8** Multi-effect batch distillation (MEBAD) as illustrated by Hasebe *et al* (1997)

Engeline and Skogestad (2005) studied the multi-effect batch distillation by applying it to an industrial case. They simulated three cases: a non-integrated base case, a multi-effect indirect split arrangement, and a multi-effect prefractionator arrangement.

The simplest configuration of multivessel batch distillation might be batch distillation column with a middle vessel or middle vessel batch distillation. This column consists of only two columns and a vessel located between the columns. To some cases, another vessel is installed above the top column after the condenser as previously illustrated in Figure 2.6. This configuration is also called as middle vessel batch distillation.

In a case where the separation process in a middle vessel batch distillation is operated under total reflux, the operation is run in a discreet manner. The reboiler is filled with feed. Energy input to the plant is performed through the evaporator/reboiler. Three desired product fractions are collected in three product vessels, which are mounted along the column. Before steady state is attained, no product is withdrawn from the system. The liquid stream entering each product vessel is returned to the distillation column. This corresponds to operation with total reflux (*i.e.* cyclic operation). Thus, the first important advantage of MVBD is the operation with the highest possible separation performance.

The second is that no reflux strategy is required as it is also the case with cyclic operation of conventional batch distillation studied by Sørensen (1999). If the separation efficiency is high enough, the mode of total reflux operation does not give rise to the off-cut characteristic of batch distillation with sequential product withdrawal. Both of these facts lead to significant advantages in terms of the batch time and product yield, and improve the economics of the process.

As studied by Gruetzmann *et al.* (2006), a distinguishing feature of a cyclic operation is that the entire process always consists of the following two stages. During the startup phase, the individual product fractions are accumulated in the product vessels. The product composition changes with time. In this phase, all of the product

vessels are emptied and the products are delivered to the product tanks. At steady state, all product compositions must correspond to the set point. These set points result from the quality specification of the products. Figure 2.5 illustrates the general separation behavior of a middle vessel batch distillation, which is a type of multivessel batch distillation. Typically, the process consists of start up, steady state, and process control.

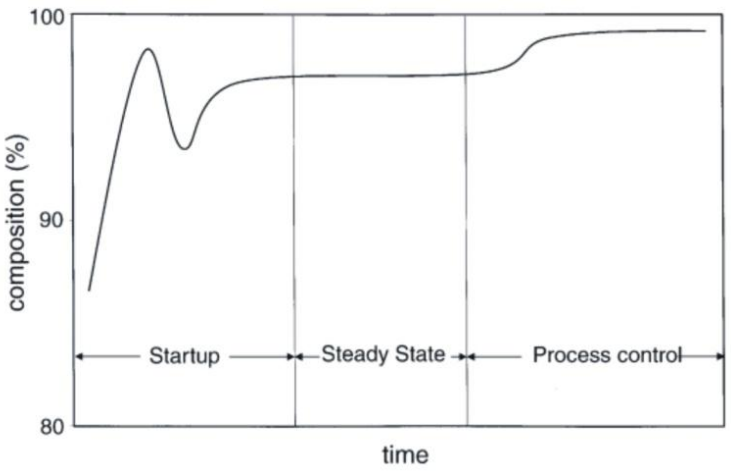


**Figure 2. 9** General process behavior of a middle vessel batch distillation with total reflux (Gruetzmann, 2006)

Regarding the middle vessel batch distillation, Davidyan (1994) demonstrated the application of batch distillation columns with a middle vessel for separating binary and ternary mixtures. Based on this work, he presented an analytical analysis of the dynamic behavior of the middle vessel batch distillation column for separating multicomponent mixtures. The authors pointed out several advantages and new opportunities for the middle vessel configuration as compared to conventional/regular batch distillation column.

In his paper, Davidyan (1994) mentioned that a binary mixture can be separated into pure components under finite boiling rate and reflux ratio and, in some cases, faster than using conventional types of batch distillation columns. For ternary separation, one can always choose parameters, like reflux ratio and boiling rate, such that the composition in the middle vessel tends to either one of the three components (*e.g.* the intermediate-boiling component for ideal mixtures). A multi-component

mixture can be separated into heavy, intermediate and light fractions simultaneously, so one can remove light and heavy impurities from the mixture. In term of holdup effects and start up model, Abdul Aziz (1994) concluded that the effect of holdup in complex column is quite similar to the conventional column.

### 2.3.2 Operation of complex columns

If a multivessel vessel batch distillation column is operated under total reflux, the charge in each vessel will be purified as the distillation proceeds. However, the purity in each vessel will depend on the number of plates in each section of the column, vapor boil up, the amount of initial charge in each vessel, and the duration of operation time (Mujtaba, 2004).

Meski and Morari (1995) analyzed the behavior of the middle vessel configuration for binary and ternary mixtures at total reflux and minimum reflux conditions. They confirm that the middle vessel batch distillation columns always perform better than the conventional batch distillation design. They also conclude that it is optimal to operate the column under constant reflux ratio and boiling rate in the case of binary separation, keeping the composition in the middle vessel constant at the initial feed composition with gradual decreasing holdup of the middle vessel.

In the multi-vessel column (also called multi-effect column), several batch distillation columns are coupled by vapor and liquid streams with vessels (holdup tanks) between, but with only one reboiler and one condenser. One objective is to minimize the energy consumption, and this is analogous to integrated (multi-effect) continuous columns like the Petlyuk column. The Petlyuk column configuration is a fully thermally coupled distillation column arrangement where two or more columns are linked together through vapor and liquid streams without reboilers or condensers between the columns or simply one column with a dividing wall inside one column shell. Such complex column arrangements are shown to offer large potential savings in energy compared with sequences of simple columns for multi-component continuous distillation separation systems (Hilmen, 2000)

Several authors have presented comparative studies of conventional batch distillation columns and the middle vessel batch configuration (Hasebe *et al.*, 1992; Mujtaba and Macchietto, 1992; Barolo *et al.*, 1998). They compared the performance of the middle vessel batch distillation column with conventional batch distillation for separating ideal ternary mixtures. The middle vessel batch distillation column was shown to have the best separation performance. The idea of using a middle vessel was later extended to a multi-vessel batch column configuration with total reflux operation for separating more than three components (Wittgens, 1996). He claimed that the multi-vessel batch distillation column is potentially more energy efficient than the corresponding sequence of continuous distillation columns for mixtures with more than five components. Furthermore, Hasebe *et al.* (1997) gave optimization results on optimal vessel holdup policy in the closed multi-vessel column for ideal mixtures in terms of minimum batch time. They proposed to charge the feed to the reboiler and gradually increase the holdups of the other vessels up to pre-calculated (optimized) values and then run in total reflux mode until the desired product purities are reached in each vessel.

A generalization of the multivessel column for mixtures with more than three components is a configuration with $n_c$-2 vessels where $n_c$ is the number of components in the mixture. This column is normally denoted a multivessel column configuration. In this configuration, feed is possible to be charged in different places: still pot, intermediate vessels and top vessel. Hasebe *et al.* (1997) introduced this type of configuration as multi-effect batch distillation as previously discussed above.

Compared to the more traditional column configurations, where the products are withdrawn over the top, one at a time and the use of off-cut fractions to fulfill product quality specifications, there are at least two advantages with the multivessel column. First, the operation is much simpler since no products change-over are required during the operation. Second, the energy requirement may be less due to the multi-effect nature of the separation, where the heat required for the separation is supplied only to the reboiler/still pot and cooling is done only at the top. This happens if the columns and vessels are installed vertically in such a way that the liquid flows with gravity.

Performing the separation of multicomponent mixture under total reflux, individual components will accumulate according to their boiling points along the column, provided sufficient storage capacity is available and non-azeotropic mixture is separated. The products can be collected directly from middle-vessels, top vessel and, to some cases, bottom vessel (reboiler). In this operation mode, the operation is very much simpler. No reflux control is required. The only profile that must be well understood is the composition change in each vessel as a function of time. Again, Hasebe *et al.* (1999) investigated the optimal operation policy for total reflux of multi-effect batch distillation, which minimizes energy consumption. The results of optimizations for a binary system showed that separation performance of the total reflux column was increased remarkably by optimizing the reboiler holdup as a function of time. The authors also stated that for ternary systems the separation performance of the multi-effect batch distillation (MEBAD) is better than the conventional batch column even if the holdup of each vessel is kept constant during the entire operation period.

## 2.4 DESIGN ASPECTS OF BATCH DISTILLATION COLUMN

A distillation column is designed based on the separation purpose, operating conditions, and the component properties of the mixture. Both plate and packed columns are widely used in chemical industries. Several papers have discussed about the separation behavior in both types of column. To find the comparable models for plate and packed columns, Salimi and Depeyre (1996) studied the comparison between dynamic behavior of a batch packed and plate column. They summarized the findings and presented in a table as shown in Table 2.1.

Designing a randomly packed column is a subtle combination of art and science. Packed columns are most frequently used to remove contaminants from a gas stream in absorption. However, packed columns can also be used to remove volatile components from a liquid stream by contacting it with an inert gas (stripping). In addition, they are also used in distillation applications where the separation is particularly difficult due to close boiling components. However, the design methods are similar for any of the process scenarios.

**Table 2. 1** Model comparison between packed and plate columns

| Equation | Packed | Plate |
|---|---|---|
| Thermodynamics | • Equilibrium<br>• Non-equilibrium | • Equilibrium<br>• Non-equilibrium |
| Mass and energy balance | • Differential<br>• Finite difference approximation | • Differential<br>• Finite difference approximation |
| Hydraulics | • Continuous contact<br>• Big liquid holdup<br>• Small total pressure drop | • Step by step contact<br>• Small liquid holdup<br>• Big total pressure drop |

(Salimi and Depeyre, 1996)

The first step in designing a packed tower is more science than art. The equilibrium data between the distillation components is required for the analysis. If tabulated data for the system is unavailable for distillation, equilibrium data can be predicted by selecting the appropriate thermodynamic properties. Senol (2001) studied the mass transfer and modeling of randomly-packed distillation column. His article dealt with the performance of a randomly packed distillation column depending on the effective vapor-liquid interfacial area and the flood ratio. He mainly focused on an analytical interpretation of conditions optimizing height equivalent to a theoretical plate (HETP) and interfacial area.

More comprehensively, a study on packed column has also been performed by Yang and Chuang (2000). They presented a new approach to simulation of distillation in packed column by modifying equilibrium stage model. The new model incorporated mass transfer efficiency and convective heat transfer along the packed column.

For batch distillation column, unfortunately, there is no specific literature for designing the batch column. However, since the basic principle is similar to the continuous one, thus the procedures of continuous column design can be simply used to design the batch column.

Distillation columns are designed using VLE (vapor liquid equilibrium) data for the mixtures to be separated. The vapor-liquid equilibrium characteristics (indicated by the shape of the equilibrium curve) of the mixture will determine the number of stages required for the separation. This is illustrated clearly by applying the McCabe-Thiele method to design a binary column (Benitez, 2009).

The McCabe-Thiele approach is a graphical one, and uses the VLE plot to determine the theoretical number of stages required to effect the separation of a binary mixture. It assumes constant molar overflow and this implies that:

1. molal heats of vaporization of the components are roughly the same
2. heat effects (heats of solution, heat losses to and from column, etc.) are negligible
3. for every mole of vapor condensed, one mole of liquid is vaporized

For the design procedure, given the VLE diagram of the binary mixture, operating lines are drawn first. For common components, this diagram can be easily obtained from literatures or, alternatively, be generated from commercial software, such as Aspen Plus.

1. Operating lines define the mass balance relationships between the liquid and vapor phases in the column.
2. There is one operating line for the bottom (stripping) section of the column, and one for the top (rectification or enriching) section of the column.
3. Use of the constant molar overflow assumption also ensures that the operating lines are straight lines.

### 2.4.1 Selecting type and size of packing

This is where the art of designing packed columns begins. Some people believe that there are stringent rules surrounding the choice between random and structured packing. Random packing is simply understood as the type that comes in a sack and it is simply dumped into the column. Structured packing may come in bales or intricate designs that are stacked in specific patterns. This is probably one of those areas of engineering where past experience in the application is the best guide. Structured packing is used are in very low pressure drop applications and for increasing the capacity of an existing column (Brown, 1980).

Random packings have various shape, material, and size. Random column packings are inexpensive alternative to increase a tower's capacity and efficiency. Some examples of random column packings are presented in Figure 2.10.

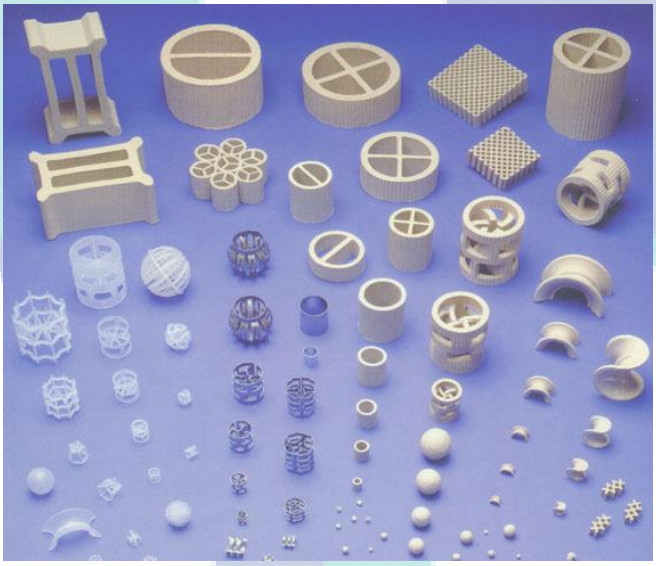

**Figure 2. 10** Several types of random packings

On the other hand, structured column packing is designed for a column where the vapor rate is high enough. Some types of the structured column packings are shown in Figure 2.11.

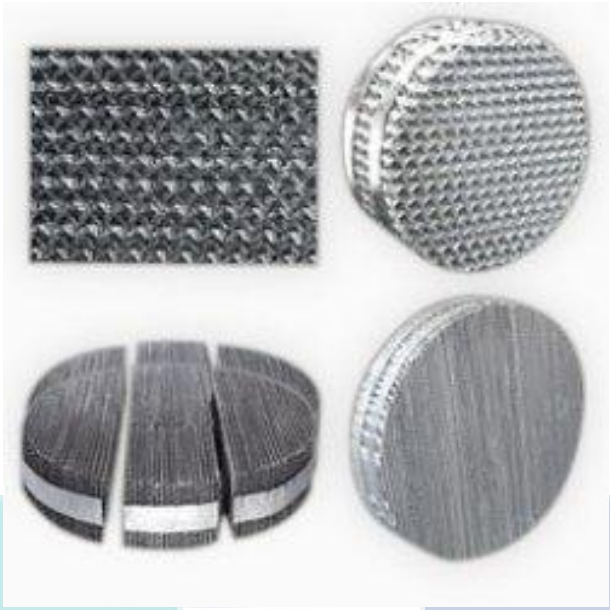

**Figure 2. 11** Several types of structured packings

### 2.4.2 Determining the column diameter

Most methods for determining the size of randomly packed towers are derived from the Sherwood correlation. A design gas rate can be determined with the help of the figure below which is based on correlation from the Sherwood equation.

These guidelines are designed around "flooding pressure drops" documented in literature. In other words, for most cases, designing with these pressure drops should help designers avoid flooding. An equation below can be used to calculate the x-axis value, and then is used to obtain the Sherwood Number in y-axis of the graph.

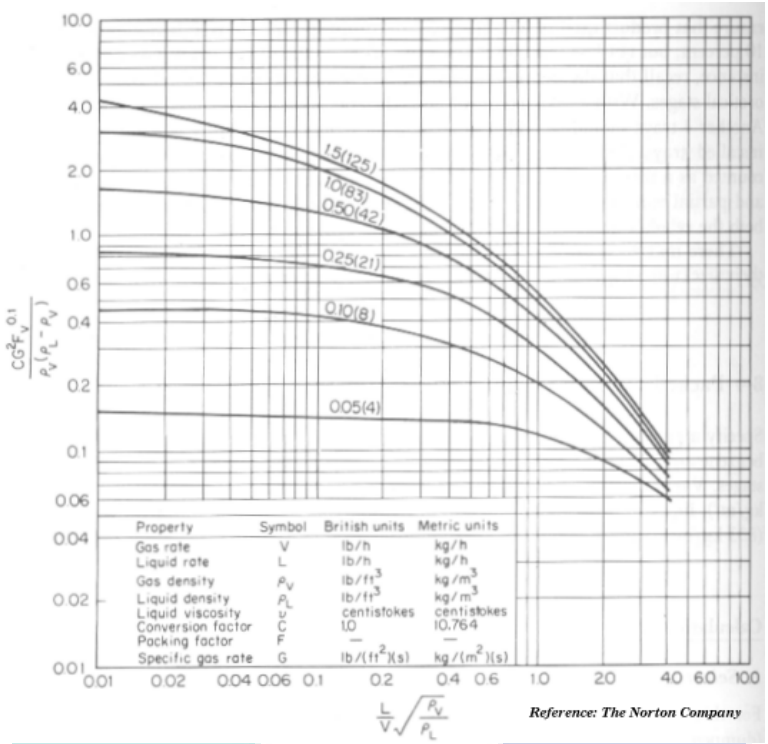


**Figure 2. 12** Sherwood equation for estimating the packing size (Treybal, 1981)

### 2.4.3 Determining the height of column

Packed columns are often used for distillation when the separation is relatively easy and the required column diameter is not very large. They are generally less expensive than plate columns and have a lower pressure drop. The main disadvantage is the difficulty in getting good liquid distribution, particularly for large diameter columns or very tall columns. Even if liquid is spread evenly over the packing at the top of the column, liquid tends to move towards the wall and to flow through the packing in preferred channel (McCabe *et al.*, 2001).

Perhaps the most interesting step in designing a packed column is deciding how tall to build the column. HETP (Height Equivalent to a Theoretical Plate) is a method to estimate the height of the packing that is equivalent to a certain number of stages. This is suitable for preliminary design of a packed column. However, for higher demand of accuracy, it should be consulted to the professional packing manufacturers.

## 2.5 PROCESS CONTROL OF BATCH DISTILLATION

Due to the nature of dynamic processes of batch distillation, process control has become one of most important elements in batch distillation. Many researchers have performed investigations on the process control of batch distillation column, especially in the sequential process control (SPC) where one event follows another until a job is completed. Skogestad (1992) presented a more comprehensive re

view on the operation of distillation columns in general, including dynamic modeling and simulation and also the process control. Furthermore, he also released some ideas regarding the distillation operation.

Farschman and Diwekar (1998) specifically emphasized the control of middle vessel column with dual composition control. The author assumed that the control of middle vessel column is similar to that of the continuous column if the column is operated under variable reflux/variable reboil ratio.

In the operation of MEBAD as previously mentioned by Hasebe *et al*. (1997), the control of MEBAD has also been discussed by Noda *et al*. (2000). The holdups of the reboiler, the vessel, and the reflux drum cannot be changed independently, because the sum of the holdups is constant. Thus, in case of ternary separation by the MEBAD, three controlled variables, *i.e.* compositions of three products, need to be controlled by two manipulated variables, *i.e.* holdups of the middle vessel and the reflux drum. The real advantage of their research was the application of on-line optimization system of pilot scale multi-effect batch distillation system.

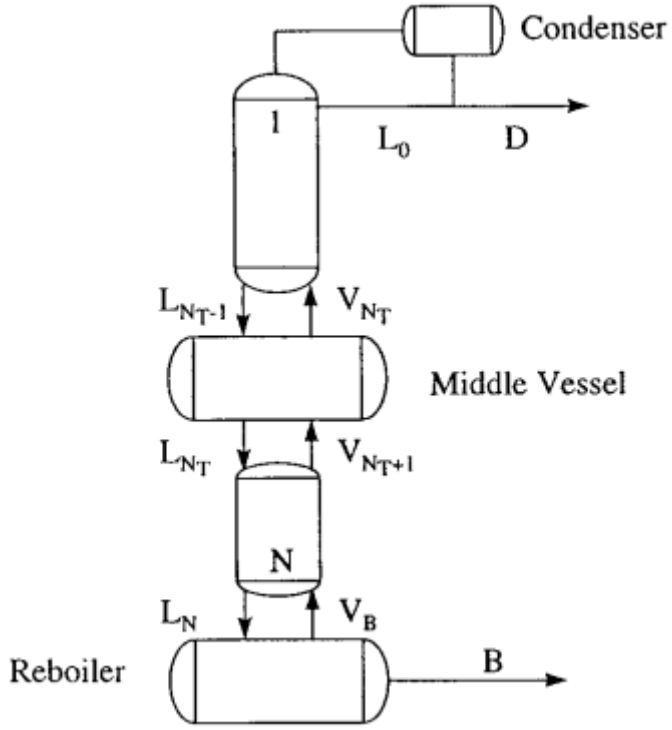


**Figure 2. 13** Middle vessel column as illustrated by Diwekar (1998)

In addition, Kano *et al.* (2003) performed a simulation of proposing predictive inferential control for batch distillation. In the predictive inferential control system, future compositions predicted from on-line measured process variables are controlled instead of the estimates of current compositions. The key concept of the predictive inferential control is to realize feedback control with a feed forward effect by the use of the inherent nature of a distillation column.

## 2.6 MOTIVATION OF THE RESEARCH

From the research history in the application of batch distillation, it is noted that batch distillation has been extensively explored; from design to operation, from simple to complex column. Some additional features have also been introduced, such as product vessels for multivessel column, cyclic operation, dual reflux drum mode, and heat-integrated aspects. Most of the past research studied only focusing on the operation and the column behavior, while the product vessels did not receive enough attention. More specifically, however, investigations on the product vessel of multivessel batch column look to leave opportunities to study further. The author

found that a deeper attention should be paid to at least two aspects of the product vessels of multivessel batch column:

1. The possibility of designing the product vessel that is able to be adjusted dependent on the separation strategy. Moreover, the product vessel is made controllable. By doing this, a better separation strategy can be proposed to handle various kind of separation, including unknown feed composition.
2. The introduction of slop vessels, which act as slop cuts in the conventional batch column. The slop vessels are installed along the column for compensating undesired components from respective product vessels. Therefore, the product purity achievable in the product vessel can be increased.

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