SEPARATION FERMENTATION PRODUCTS BY USING BATCH DISTILLATION

ZULHAZLEEN BINTI MOHD ZULKIPLI

BACHELOR OF CHEMICAL ENGINEERING

UNIVERSITI MALAYSIA PAHANG

UNIVERSITI MALAYSIA PAHANG

CENTER FOR GRADUATED STUDIES

We centify that the thesis entitled “Production of ethanol by using batch distillation” is written by Zulhazleen binti Mohd Zulkipli. We have examined the final copy of this thesis and in our opinion; it is fully adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering. We herewith recommend that it be accepted in fulfillment of the requirement for the degree of Bachelor of Chemical Engineering.

Name of External Examiner Signature

Institution:

Name of Internal Examiner Signature

Institution:

SEPARATION OF FERMENTATION PRODUCTS USING BATCH DISTILLATION

ZULHAZLEEN BINTI MOHD ZULKIPLI

Thesis submitted in fulfillment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

JUNE 2012

SUPERVISOR'S DECLARATION

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

Signature
Name of Supervisor:
Position:
Date:

STUDENT'S DECLARATION

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

Signature

Name:

ID Number:

Date:

ACKNOWLEDGEMENTS

I would like to be grateful to Allah s.w.t because with his will I was able to complete the experiment and report in 2012. First of all i would like to express gratitude to my supervisor DR. Anwaruddin Hisyam for the guidance and encourangement that had been given throughout the experiment . All the lesson that had been given is much indeed appreciated by me.

A big contribution and hard worked from all of staffs in the chemical laboratory of University Malaysia Pahang during the experiment was truly help the progression to completed the inquiriment of my program.The process in conducting the experiment makes me realized that working enviroment is really though. This experience is really important to me for future. The program brought me realized that in working , working together and respect each other is important.

Great deals appreciated go to the my faculty , Chemical Engineering and Natural Resources Faculty in order to give the best guidance for training students . I also would like to thank for my friends that share the knowledge in order to complete the report and special thanks especially to my parents Mohd Zulkipli bin Abdullah and Siti Saleha Yusoff that continuously give the support in order to complete my studies.

ABSTRACT

This research aims to study the separation processes of fermentation products by using batch distillation. Technically, the main objective of the research is to study behavior of the purification of fermentation products of local nira by utilizing batch distillation . The major product from the fermentation process is ethanol. The scope of study includes the investigation of composition profile of the distillate and the others parameter to the composition profile. Nira is a fermentation product that to be expected to produce ethanol by undergo the separation process using batch distillation. The result shows the concentration can be obtained by using the standard curve . By utilizing batch distillation, the optimun condition in separation components in vinegar from nypa palm can be determine. For the effect of time the concentration of ethanol become concentrated as increasing the time. At 74˚C-76˚C the optimum product can be collected because in this stage the particle of ethanol have been nearly reached the boiling point which means the particle have enough energy to escape from the liquid form to the vapors form. The concentration of acetic acid is higher than ethanol because most of ethanol particle have been converted to the acetic acid. The highest ethanol concentration should be after 20 hours fermentation, however the vinegar solution that has been used in this experiment is more than 1 days.

ABSTRAK

Kajian ini fokus untuk mengkaji process pemisahan produk penapaian menggunakan penyulingan berperingkat. Secara teknikal, objektif untuk kajian ini adalah untuk mengkaji sifat penulenen produk penapaian dari produk tempatan iaitu cuka nipah menggunakan penyulingan beperingkat. Produk utama untuk proses penapaian ini ialah etanol. Skop kajian merangkumi kajian kandungan produk dari penyulingan dan parameter untuk menghasilkan produk. Cuka nipah dijangka menghasilkan etanol menggunakan penyulingan beperingkat. Hasilnya kepekatan boleh didapati melalui lengkung standard . dengan menggunakan penyulingan beperingkat keadaan optimum dalam process pemisahan komponen didalam cuka nipah boleh di dapati. Untuk kesan perubahan masa , kepekatan etanol bertambah apabila masa bertambah. Pada suhu 74℃-76℃ kepekatan produk adalah paling tinggi kerana partikel etanol menghampiri takat didih dan bermakna partikel mempunyai cukup tenaga untuk terbebas dari keaadaan cecair kepada gas. Kepekatan asid asetic lagi tinggi daripada etanol kerana partikel etanol telah bertukar kepada asid asetic. Kepekatan etanol paling tinggi sepatutnya selepas 20 jam proses penapaian, tetapi larutan cuka yang digunakan dalam eksperimen ini melebihi satu hari.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

1.1: Introduction

The reasearch objective is to determine the behavior of the purification of fermentation products of local nira by utilizing batch distillation process. Fermentation can produce alcohol (ethanol) by undergoing continuosly fermenting unit. Ethanol is commonly prepared by fermentation of sugars or other biomass feedstock. In fermentation process fermentable materials are added with microbe. During the fermentation, the microbe cells consume the biomass feedstock in beaker and convert the feedstock into the alcohol as they continue to grow.

Fermentation processes from any material that contains sugar could derive ethanol. The varied raw materials used in the manufacture of ethanol via fermentation are conveniently classified into three main types of raw materials: sugars, starches, and cellulose materials. Sugars (from sugarcane, sugar beets, molasses, and fruits) can be converted into ethanol directly by Yan Lin & Shuzo Tanaka et al, 2005.

Starches that from corn, cassava, potatoes, and root crops must first be hydrolyzed to fermentable sugars by the action of enzymes from malt or molds. Cellulose (from wood, agricultural residues, waste sulfite liquor from pulp, and paper mills) must likewise be converted into sugars, generally by the action of mineral acids. Once simple sugars are formed, enzymes from microorganisms can readily ferment them to ethanol (Yan Lin & Shuzo Tanaka et al, 2005).

Fermentation product:

$$C_6H_{12}O_6 \longrightarrow 2\ CH_3CH_2OH + 2\ CO_2$$

It is usually carried out in a batch still to which a column equivalent to a number of equilibrium stages is attached. In a batch distillation process, operation occur discontinuously. 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. Batch process is economical if it is in small volume. Batch process are made up from a series of batch and semicontinues steps. So, the process is flexible in changing production rate or product formulation. For any process synthesis the prediction of the concentration path of distillate and bottom of a batch distillation is necessary especially for mixtures with complex structure, e.g. with one ore more azeotropes. Many authors have investigated these concentration paths (Reinders and de Minjer, 1940; Ewe11 and Welch, 1945). Some of their results can be explained by a modified Rayleigh equation (herepresented for batch distillation operated as stripping column):

$$\frac{dx_{Da}}{dx_{Db}} = \frac{x_{Da} - x_{Ba}}{x_{Db} - x_{Bb}}$$

The demand of the ethanol production and consumption has grown considerably mainly as a renewable fuel. As ethanol is soluble in water, it can be used in a variety of different products in industry. By using batch distillation process behavior the optimal condition could be determined to obtain ethanol.

1.2: Problem Statement

The ethanol produced by fermentation ranges in concentration from a few percent up to about 14 percent by Shakhashiri et al.,2009. Nearly all fuel ethanol is produced by fermentation of corn glucose in the US or sucrose in Brazil by MacDonald et al, 2001 and Rosillo-Calle & Cortez et al, 1998. A latest technology is used for a country with a significant agronomic-based economy for fuel ethanol fermentation. This is possible because, during the last two decades, technology for ethanol production has

been developed to the point at which large-scale production will be a reality in the next few years.

In order to improve the production of ethanol by separation the fermentation product, batch distillation is suitable in less production. Batch distillation can be adjusted the temperature and the feed of raw material.

1.3: Objective

The main objective of the research is to study behavior of the purification of fermentation products of local ‘nira’ by utilizing batch distillation . By utilizing batch distillation, the optimun condition in separation components in ‘nira’.

1.4: Scope of Study

This research will be carry out by divided into four step. Firstly is preparation of raw material, secondly is check the standard solution concentration of ethanol and acetic acid by using UV-Vis spectrophotometers , followed by study the effect of parameters in distillation process and lastly check the product concentration again by using UV-Vis spectrophotometers.

Preparation of raw material is done by filter the vinegar from nypa palm by using vacuum filter. This process will avoid any impurities in the solution. Secondly is to check the standard solution for the ethanol by plotting the graph at different concentration versus absorbent. Thirdly is study the composition profile of distillate as a function of time. Lastly, the concentration for ethanol and acetic acid will be collected by compare absorbent from UV-Vis spectrophotometers to the standard graph solution.

1.5: Rationale and Significance

The world's ethanol production is continually drop in 2012 and will continue to next 2 years. This basically means that to make up for that loss some analysts think a new demand source is needed if the prices continue to remain in the current price range. Ethanol demand keep increasing due to the used extensively as a solvent in the manufacture of varnishes and perfumes, as a disinfectant and mostly used for gasoline additive and furthermore, ethanol is less toxic than the other alcohols. The depletion of world energy supply creates interest to find alternative source to produce ethanol.

By utilizing the optimum batch distillation, the production ethanol will be increase. The parameter was studied in order to find the better condition in production of ethanol. The separation could occur due to the differential of boiling point of water that is 100 ^{o}C at the atmospheric pressure. It has been recognized that if ethanol separation is combined with fermentation there will be a reduction in the cost of process. When ethanol is removed directly from the fermentor, or by recycling the contents of a continuous fermentor through a separation device, which retains cell viability, it is possible to completely convert a much more concentrated feed .

CHAPTER 2

LITERATURE REVIEW

2.1 Distillation

Distillation of a feed mixture in given composition is placed in a single stage separator (a still pot, retort or flask) and is heated to boiling. The vapor is collected and condensed to a distillate. The composition of the remaining liquid and the distillate product are determined by expressed in functions of time. There are several reasons for running a batch process such as this:

1) Small capacity doesn't warrant continuous operation
2) Separation is to be done only occasionally
3) Separation is preparative to produce a new product
4) Upstream operations are batch wise or feed stocks vary with time or from batch to batch
5) Feed materials are not appropriate for a continuous flow system.

Integrates both sides:

$$\int \frac{dx}{x(k-1)} = \int \frac{dW}{W} \longrightarrow \frac{1}{(k-1)} \ln \frac{x}{x_0} = \ln \frac{w}{w_0}$$

2.2 Alcohol

Alcohol are compounds in which one or more hydrogen atoms in alkane have been replaced by an –OH group.the point of an alcohol is always much higher than alkane with the same number of carbon atoms. The boiling points of the alcohols increase as the number carbon atoms increases (Jim Clark,2003).

Alcohol is a process of separation alcohol (more volatile) from water (less volatile) from the solution. The solution will be heated and condesated the alcohol at the distillate will be collected as a high alcohol strength liquid. The volatilities of the substances from the distillation process deviate predicted by using Raoult's Law. In batch distillation, the separation of two components will stop when the volatilities become same and the boiling point remains static.

2.3 Properties of Ethanol

Ethanol and water form a constant boiling point of the mixture at 95.6% v/v EtOH with a boiling point 78.2 ^{o}C . This temperature is close with the boiling point of pure ethanol that is 78.5^{o}C (Shakhashiri et al.,2009). The separation could occur due to the differential of boiling point of water that is 100 ^{o}C at the atmospheric pressure.

Ethanol is a monohydric primary alcohol. It melts at -117.3°C and boils at 78.5°C. It is miscible with water in all proportions and is separated from water only with difficulty; ethanol that is completely free of water is called absolute ethanol. Ethanol forms a constant-boiling mixture, or azeotrope, with water that contains 95% ethanol and 5% water and that boils at 78.15°C; since the boiling point of this binary azeotrope is below that of pure ethanol, absolute ethanol cannot be obtained by simple distillation. However, if benzene is added to 95% ethanol, a ternary azeotrope of benzene, ethanol, and water, with boiling point 64.9°C, can form; since the proportion of water to ethanol in this azeotrope is greater than that in 95% ethanol, the water can be removed from 95% ethanol by adding benzene and distilling off this azeotrope (Ang Dek Chang et al.,2001).Because small amounts of benzene may remain, absolute ethanol prepared by this process is poisonous.

Ethanol reacts with certain acid to form ester, with acetic acid it forms ethyl acetate. Ethanol can also be oxidise to form diethyl ether or at higher temperature , ethylene (The Columbia Electronic Encyclopedia, 2000).

2.4 Ethanol Fermentation

Ethanol is well known for use in alcoholic beverages, and the vast majority of ethanol for use as fuel, is produced by fermentation. By using yeast that usually *Saccharomyces cerevisiae*, the reaction occur to metabolize sugar in the absence of oxygen and then they produce ethanol and carbon dioxide. The chemical equation below summarizes the conversion:

$$C_6H_{12}O_6 \rightarrow 2\ CH_3CH_2OH + 2\ CO_2$$

It has been recognized that if ethanol separation is combined with fermentation there will be a reduction in the cost of process. When ethanol is removed directly from the fermentor, or by recycling the contents of a continuous fermentor through a separation device, which retains cell viability, it is possible to completely convert a much more concentrated feed .

Ethanol could be produced either synthetically by direct hydration of ethylene which is a product of natural gas, or by fermentation process which involved the conversion of fermentable sugar into ethanol by microorganism such as Zymomonas mobilis and Saccharomyces cerevisiae. Except for human consumption, synthetic ethanol is widely used due to its lower production costs. However, the synthetic ethanol is relied on non-renewable resources. Therefore, if ethanol is to be widely used as a chemical feedstock and as fuel in future, the production of ethanol via fermentation process using renewable biomass is essential (Ang Dek Chang,2001).

2.5 Ethanol Uses In Chemical Industry

Ethanol has been used in about 50-60% in industrial field as a solvent in the formulation of numerous commercial products. Fermentation ethanol is preferred for use in the formulation of medicinal products such as tincture of iodine , merthiolate , cough syrups , and elixirs (Herman Harry Szamant,1989)

15 % of industrial uses ethanol is in production of esters. Carboxylic acids will be react with ethanol when warmed together in the presence of a few drops of concentrated sulphuric acid in order to observed the smell of the esters formed (Jim Clark, 2004).

Ethanol can be easily converted through dehydration process to produce diethyl ether (DEE) which is a source of compression-ignition fuel with higher energy density than from ethanol. In the trasportation sector , ethanol produced from the biomass as future fuel for spark-ignited engines because of high octane quality.

2.6 UV-Vis Spectrophotometer

Ultraviolet (UV) and Visible (VIS) light can cause electronic transitions. When a molecule absorbs UV-VIS radiation, the absorbed energy excites an electron into an empty, higher energy orbital. The absorbance of energy can be plotted against the wavelength to yield a UV-VIS spectrum. UV-VIS spectroscopy has many uses including detection of eluting components in high performance liquid chromatography (HPLC), determination of the oxidation state of a metal center of a cofactor , or determination of the maximum absorbance of a compound prior to a photochemical reaction. Most organic compounds that absorb UV-VIS radiation contain conjugated pi-bonds. Both the shape of the peak and the wavelength of maximum absorbance (l_{max}) give information about the structure of the compound (Department of Chemical Wake Forest College, 2012)

Ultraviolet radiation has wavelengths of 200-400 nm. Visible light has wavelengths of 400-800 nm. Plastic cuvettes can be used to hold a sample if you wish to scan only the visible region. Since plastic absorbs UV radiation, more expensive quartzcuvettes are used when ultraviolet scans are desired.

For conviniece of reference, definitions of various spectral regions have been set by Joint Comittee on Nomenclature in Applied Spectroscopy.

Table 2.1 :Wavelength For Various Spectral Regions

Region	Wavelength
Far ultraviolet	10-200
Near ultraviolet	200-380
Visible	380-780
Near infrared	780-3000
Middle infrared	3000-30,000
Far infrared	30,000-300,000
Microwave	300,000-1,000,000,000

Source: http://www.molecularinfo.com/MTM/UV.pdf

CHAPTER 3

METHODOLOGY

3.1 Chemical and Raw Material

Chemical used in this experiment are 1M acetic acid and 99.7% v/v pure ethanol. This chemical was used as the standard solution in order to find concentration of ethanol and acetic acid in nypa palm vinegar solution. The only material is a product fermentation that is vinegar from nypa palm. By utilizing the optimum batch distillation, the production of ethanol from the vinegar of nypa palm can be increase.

3.2 Equipment

In this study, there are some important equipment that is used. Firstly are vacuum pump suction, UV-Vis spectrophotometer and batch distillation. Vacuum pump suction is used to separate the impurities from the material to increase the accuracy. UV-Vis spectrophotometer is used to determine the concentration of ethanol and acetic acid in the solution of vinegar of nypa palm. Batch distillation is used to separate the components in the vinegar.

3.3 Experimental Procedure

3.3.1 Preparation of Raw Material

Initially , the solution of vinegar from nypa palm was removed the impurities by using vacuum pump suction. The solution will be filtered and the color of solution become clearrer. The impurities was removed because it may effect the result due to the disturbance that may occured.

3.3.2 Preparation Standard Curve

This process was including using the UV-Vis spectrophotometer to determine the concentration of solution. Acetic acid and ethanol solution was set at four different concentrations. The type of cuvette is glass because the wavelength for both acetic acid and ethanol is below than 350nm. By using UV-Vis spectrophotometer to determine the absorbent, the concentration versus absorbent was plotting. This graph then can be used to find the concentration of vinegar.

Figure 3.1: UV-Vis spectrophotometer

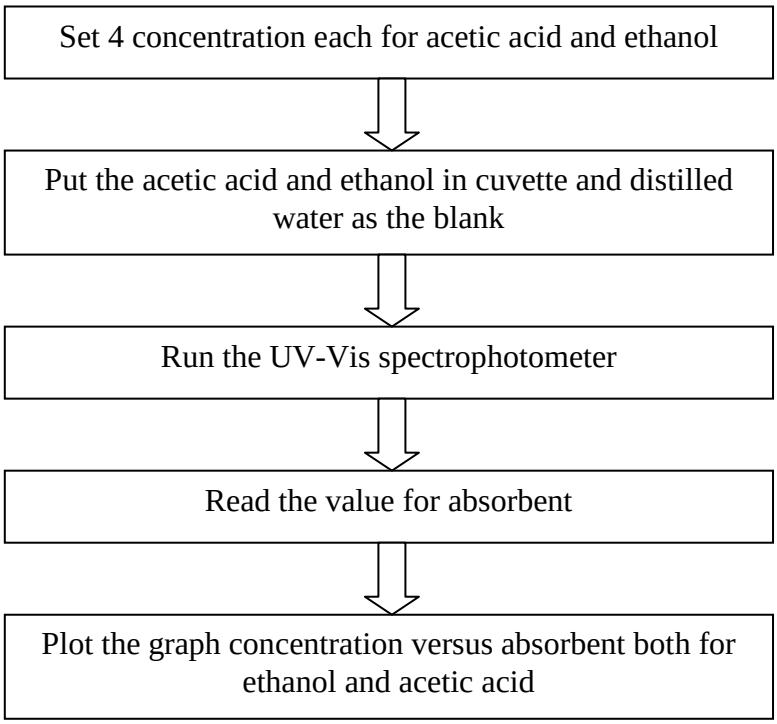

Figure 3.2: Preparation Standard Graph for Ethanol and Acetic Acid

3.3.3 Evaluation Parameter by Using Batch Distillation

This step is using batch distillation to separate components in vinegar. By conducting the batch distillation experiment, temperature and volume of vinegar was recorded each 30 minutes for four times. The product then was collected in order to find the concentration later by using UV-Vis spectrophotometer. The boiling point of ethanol is 78 ˚C so the experiment was stop when reaching the boiling point of ethanol.

Figure 3.3 : Batch Distillation Process

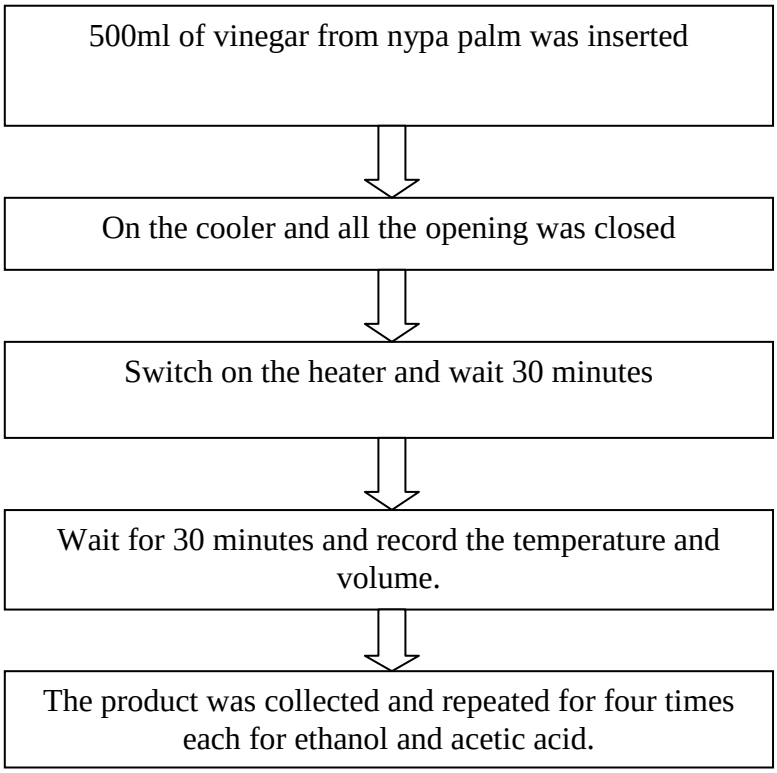

Figure 3.4 : Batch Distillation Process Step

3.3.4 Determine the Concentration

The concentration of ethanol and acetic acid will be determine by refering to the standard curve solution . Each 30 minutes, 60 minutes, 90 minutes and 120 minutes was collected the product sample. To determine the sample concentration once again UV-Vis spectrophotometer was used to find the concentration. The wavelength for ethanol is at 240 nm whereas acetic acid is 207 nm.

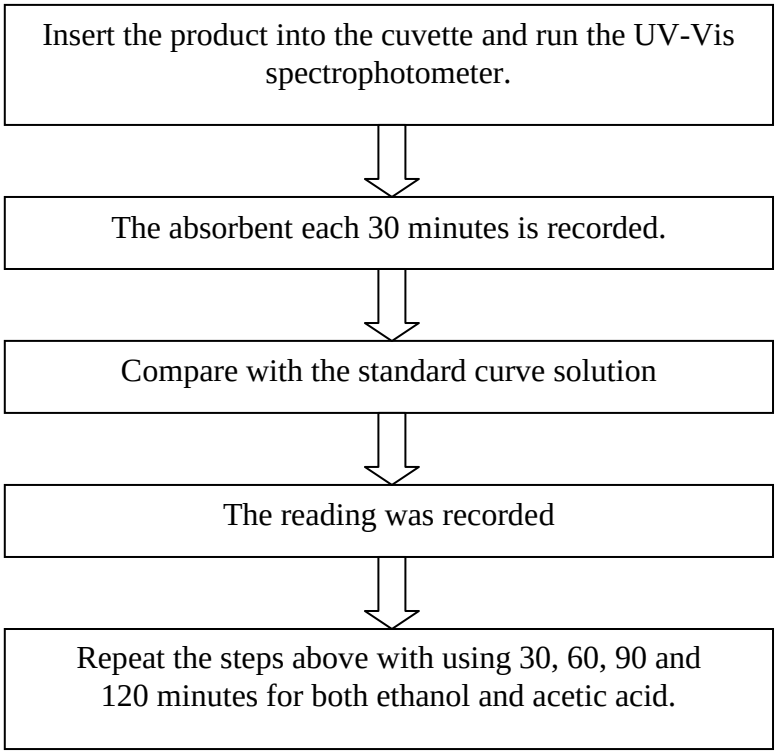

Figure 3.5: Determine the Concentration of Product

CHAPTER 4

RESULT AND DISCUSSION

4.1 Background

This chapter is discussed about the result obtained from the experiment. The experiment was repeated three times to compare the result. Each experiment will determine the concentration ethanol and acetic acid, temperature, and also volume collected for 30 minutes, 60 minutes, 90 minutes and 120 minutes. However the main result is to compare the concentration ethanol and acetic acid for each time.

4.2 Standard Curve

Standard curve is plotted for absorbent versus concentration in order to get the value of concentration for duration time parameters. Standard curve for ethanol is plotted using several of initial concentration starting from 19.94 g/L, 39.88 g/L, 59.82 g/L, and 79.76 g/L whereas the standard curve for acetic acid was 0.05 M, 0.10M,0.15 M and 0.2M. By using UV-Vis spectrophotometer, absorbent value can be obtained. This standard curve is used to predict the concentration at certain parameters for the others. **Table 4.1** shows the values for absorbent according to initial concentrations for ethanol while **Figure 4.1** in Appendix A shows the graph of absorbent versus concentration for ethanol. **Table 4.2** shows the values for absorbent according to initial concentrations for acetic acid starting from 0.05 M, 0.10 M, 0.15 M, and 0.2 M while **Figure 4.2** in appendix A shows the graph of absorbent versus concentration for acetic acid. The absorbent **Table 4.6** and **Table 4.7** for each experiment for ethanol and acetic acid in Appendix A.

Table 4.1: Absorbent for Standard Concentration Ethanol

Absorbent	Concentration (g/L)
0.786	19.94
1.082	39.88
1.401	59.82
1.724	79.76

Table 4.2: Absorbent for Standard Concentration Acetic Acid

Absorbent	Concentration (M)
0.196	0.05
0.347	0.10
0.525	0.15
0.717	0.20

4.3 Effect of Parameter

Appendix B shows the calculation of acetic acid concentration. By using the graph from **Figure 4.1** and **Figure 4.2** in Appendix A, the concentrations of acetic acid and ethanol in product can be determine at certain time. The result for the effect of parameter shows in **Table 4.3, Table 4.4,** and **Table 4.5**.

Table 4.3: Effect of Parameter in Batch Distillation Experiment 1

total volume = 500ml
temperature = 27 ˚C

Time, min	temp, ˚C	Conc. Ethanol, g/L	Conc. Acetic acid, g/L	volume ,ml
30	70	1.00	14.6522	49.2
60	72	1.60	14.7126	51.5
90	74	4.00	14.7723	56.2
120	78	5.00	14.8324	43.4

total= 200.3ml

residue= 235ml

Table 4.4: Effect of Parameter in Batch Distillation Experiment 2

Total volume = 500ml

Temperature = 27˚C

Time, min	temp, C	Conc. Ethanol, g/L	Conc. Acetic acid, g/L	volume ,ml
30	69	5.00	14.7126	50.00
60	73	5.40	14.7126	50.00
90	76	6.00	14.7126	57.40
120	78	6.30	14.7723	44.50

total= 200.9ml

residue=232ml

Table 4.5: Effect of Parameter in Batch Distillation Experiment 3

Total volume = 500 ml

Temperature = 27 ˚C

Time, min	temp, C	Conc. Ethanol, g/L	Conc. Acetic acid, g/L	volume ,ml
30	71	6.40	14.6822	50.00
60	73	7.60	14.7126	51.00
90	76	7.70	14.7723	56.50
120	78	7.90	14.8023	42.70

total=200.2ml

residue= 237ml

4.4 Concentration versus Time

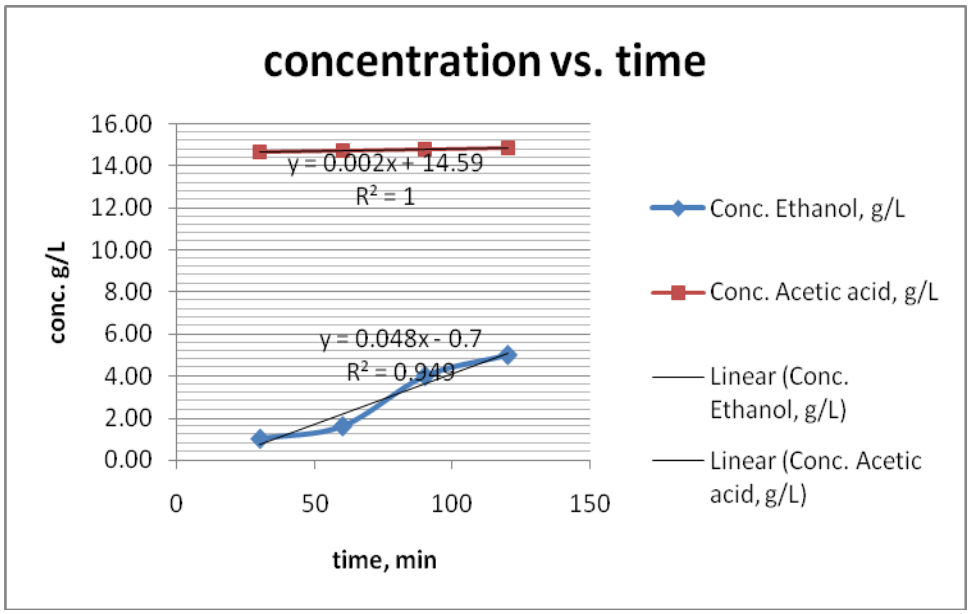

Figure 4.3: Graph Concentration versus Time for Experiment 1

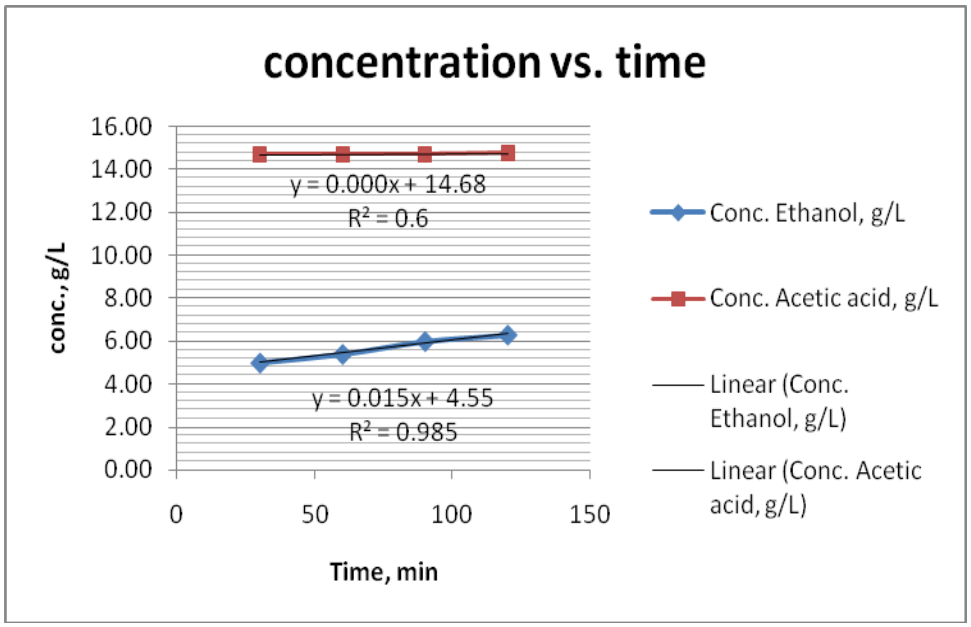

Figure 4.4: Graph Concentration versus Time for Experiment 2

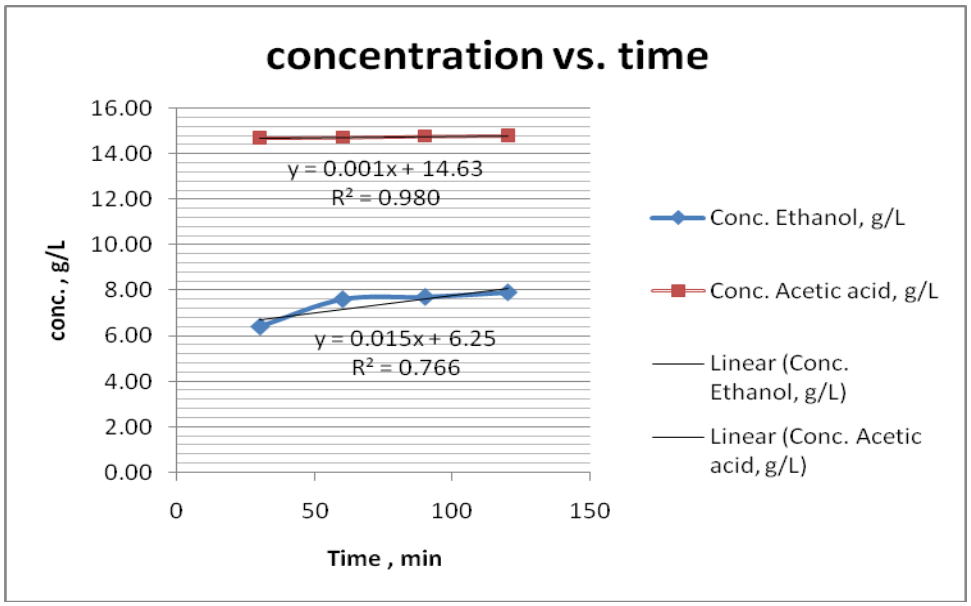

Figure 4.5: Graph Concentration versus Time for Experiment 3

The concentration ethanol keep increasing as illustrated in **Figure 4.3, Figure 4.4** and **Figure 4.5**. In order to produce ethanol, the distillation process was stopped when reached 78˚C which is boiling point of ethanol. The highest collection of ethanol was at 90 minutes which is the highest volume for each experiment 1, 2, and 3. At this time the particles rapidly distillates due to the particle of ethanol have high energy to escape from the other particle and will be condensed by condenser. As a liquid is heated, particles will evaporate off the liquid surface. It is when the pressure of these escaping particles is equal to the external air pressure on the surface of the liquid, that bubbles of gas start to form in the liquid. After reaching the 78˚C the collection of volume product become decreasing due to the ethanol particle continues to decrease.

At 78℃ the highest concentration of ethanol and acetic acid was determined. This is due to the concentration become more and more concentrated as the product keeps heating in distilling pot with high purifying process. The fermentation product have low content of alcohol and acetic acid therefore distillation is used to concentrate the solution. As the solution is heated , the formed of vapors will contain more ethanol and acetic acid than water due to the both ethanol and acetic acid are more volatile than the water. The vapors which is rich the ethanol and acetic acid then will be separated from the liquid form. After that, the vapors are then put in contact with the condenser apparatus. Due to the lower temperature the vapor will condense and keep repeating until more concentrated ethanol and acetic acid is obtained.

The concentration of acetic acid does not change much because the boiling point of acetic is over than 118℃. This shows that the acetic acid component does not distillates completely. From the **Figure 4.4** the concentration was same for the 30 minutes, 60 minutes and 90 minutes. Only some of acetic acid escapes to formed vapors. Usually vinegar can be defined as a solution that contains 5% of acetic acid because acetic acid (ethanoic acid) is the major component in vinegar.

As illustrated in the graph from **Figure 4.3, Figure 4.4,** and **Figure 4.5** the residue volume still high. This is due to the residue still containing of water and acetic

acid. The experiment only stops at 78˚C while the boiling point for water is 100˚ C and acetic acid at 118˚ C. The percent loss of volume of solution was calculated in appendix C. The percent loss for each experiment was quite high that are 12.98%, 13.22% and 12.56%. Most of the loss of volume is assume to be sticking to the wall of batch distillation.

From the **Figure 4.3, Figure 4.4,** and **Figure 4.5** the concentration of acetic acid is more than ethanol for each experiment. This is due to the most of the ethanol has been converted to the acetic acid. The concentration of ethanol was found to increase rapidly during the first 20 hours of fermentation. The ethanol might have been used as a carbon source by yeast for its growth after 22^{nd} hours, when the concentration of glucose started to deplete (Bauchop and Elsden et al, 1960; Copella and Dhurjati et al 1989). The yeast then converted the ethanol to the acetic acid because the solution of vinegar is more than 1 days. Usually after 34 hours the ethanol will shows the ethanol become decreasing. As we can see the **Figure 4.5** the concentration ethanol become to reached its limit in producing the ethanol.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The objective of the research is to study behavior of the purification of fermentation products of local ‘nira’ by utilizing batch distillation . In this study the purification process shown a positive result by conducting the batch fermentation. The concentration of ethanol become concentrated as increasing the time. The maximun concentration of ethanol isat 120 minutes with concentration 7.9 g/L.

By utilizing batch distillation, the optimun condition in separation components in vinegar from nypa palm can be determine. At 74˚C-76˚C the optimum product can be collected because in this stage the particle of ethanol have been nearly reached the boiling point which means the particle have enough energy to escape from the liquid form to the vapors form. The particle rapidly move to the air and then will be cooled by the condenser and be collected at receiving flask. By temperature 78˚C the volume of product become decreasing because all the ethanol solution has been vaporized.

The graph from **Figure 4.3, Figure 4.4,** and **Figure 4.5** shows that the concentration of acetic acid is more higher than ethanol because most of ethanol particle have been converted to the acetic acid. The highest ethanol concentration should be after 20 hours fermentation , however the vinegar solution that has been used in this experiment is more than 1 days.

5.2 Recommendations

This study can be improved by using others analysis method such as spectroscopic analysis like atomic absorption spectrometer (AAS), and Fourier transform infrared (FTIR). By using this analysis, the percentage of error can be reduced.

The average percentage loss is 12.92% which are contributing high volume for the product. The volume loss should be considered as the product also because the residue is the solution that left behind and unconverted to the product.

There are a few precaution in conducting the experiment by using batch distillation.Since this is a very energy intensive experiment a lot of heat is lost to the environment through the apparatus. Consequently a lot of the equipment gets extremely hot. Ensure that gloves are worn at all times when touching such equipment and to avoid contact with hot surfaces as much as possible. Besides that, the steam condensate and the product solution both exit the apparatus at extremely high temperatures. Wear thick gloves whenever collecting these two liquids. Be very careful during the collection since the hot liquid or steam can splash and cause burns and injuries. Lastly, leave the cooling water flow on even after closing the steam line and feed supply line. Cooling water cools down the apparatus after the experiment is over to ensure that there is no overheating of equipment causing potential damage or safety concerns.

Lastly , UV-Vis spectrophotometer need to be careful in handling. This is due the wrong techniques will produce wrong value. The value will obviously wrong from the actual value. The percentage error in handling UV-Vis spectrophotometer is high if lack the knowledge. Wear a glove and mask to avoid any injuries.

REFERENCES

Department of Chemical Wake Forest College (2012). UV-Vis Spectrophotometer. Chemistry Department . Retrieved on May 27, 2012 from http://www.wfu.edu/chem/courses/organic/UV/index.html

Shahabedin Eslami , Abdolreza Aroujalian , Babak Bonakdarpour , Ahamdreza Raessi (2007).Coupling of Pervaporation system with Fermentation Process. Journal Coupling of Pervaporation system with Fermentation Process . Retrieved November 19, 2011 from http://www.nt.ntnu.no/users/skoge/prost/proceedings/ecce6_sep07/upload/2454.pdf

Cardona, C.A. & O.J. Sánchez (2004) Modeling of Batch Extractive Fermentation for the Fuel Ethanol Production. Retrieved October 11, 2011 from http://biotecnologia.ucaldas.edu.co/publicaciones/Ponencia%20PRES05.pdf

Properties of Ethanol. Retrieved October 10, 2011 from, http://www.infoplease.com/ce6/sci/A0858037.html

I. Rodriguez-Donis, E. Pardillo-Fontdevila , V. Gerbaud , X. Joulia (2001).Synthesis experiments and simulation of heterogeneous batch distillation processes. *Journal of Computers and Chemical Engineering* . 25,799–806 .

R. Dussel and J.Stichlmair (2003) . Separation of azeotropic mixtures by batch distillation using an entrainer. *Journal of Computers and Chemical Engineering.* 19,113-118.

Michael Hanke and Pu li (2000) . Simulated annealing for the optimization of batch distillation process. *Journal of Computers and Chemical Engineering* . 24, 1-8.

Understanding Ethanol Fuel production and use. Retrieved October 1 2011 from, http://www.appropedia.org/Understanding_Ethanol_Fuel_Production_and_Use

Ang Dek Chang (2001). Production of ethanol by genetically modified Saccharomyces cerevislae using sago starch as substrate. Retrieved October 1, 2011 from, http://psasir.upm.edu.my/11856/1/FSMB_2001_39_A.pdf

G.E. Guidoboni (2002). Continuous fermentation systems for alcohol production. *Journal of Enzyme and Microbial Technology*. 6(5) 194-200.

Eva-Katrine Hilmen (2000). Separation of Azeotropic Mixtures: Tools for Analysis and Studies on Batch Distillation Operation. Retrieved October 9 from, http://www.chemeng.ntnu.no/thesis_e/download/2000/hilmen/Thesis_Hilmen.pdf

UV-Vis Spectrocopy . Retrieved 27 May 2012 from , http://www.wfu.edu/chem/courses/organic/UV/index.html

Yan Lin and Shuzo Tanaka (2006). Ethanol fermentation from biomass resources : current state and prospects. *Journal of Microbial Biotechnol*. 69, 627-642.

APPENDIX A

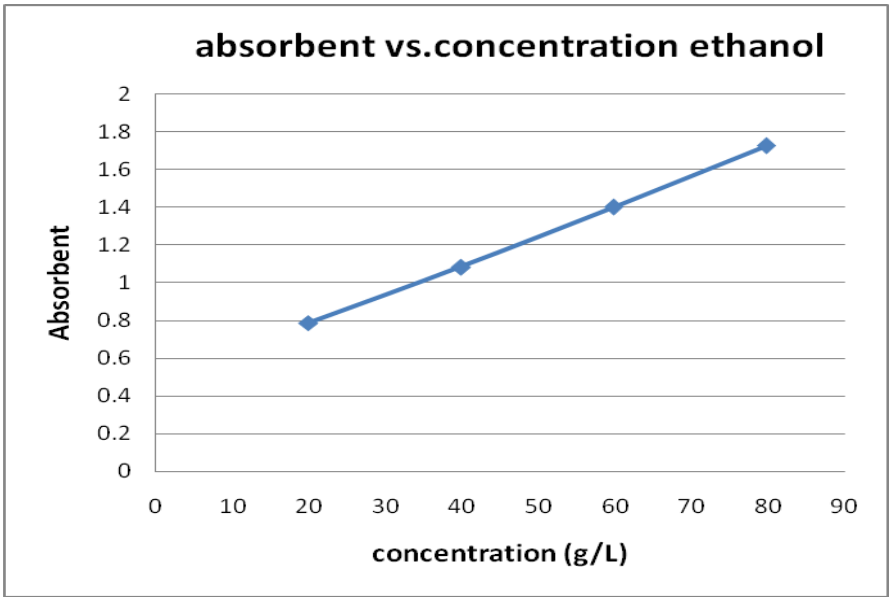

Figure 4.1: Graph Standard Solution Absorbent versus Concentration of Ethanol

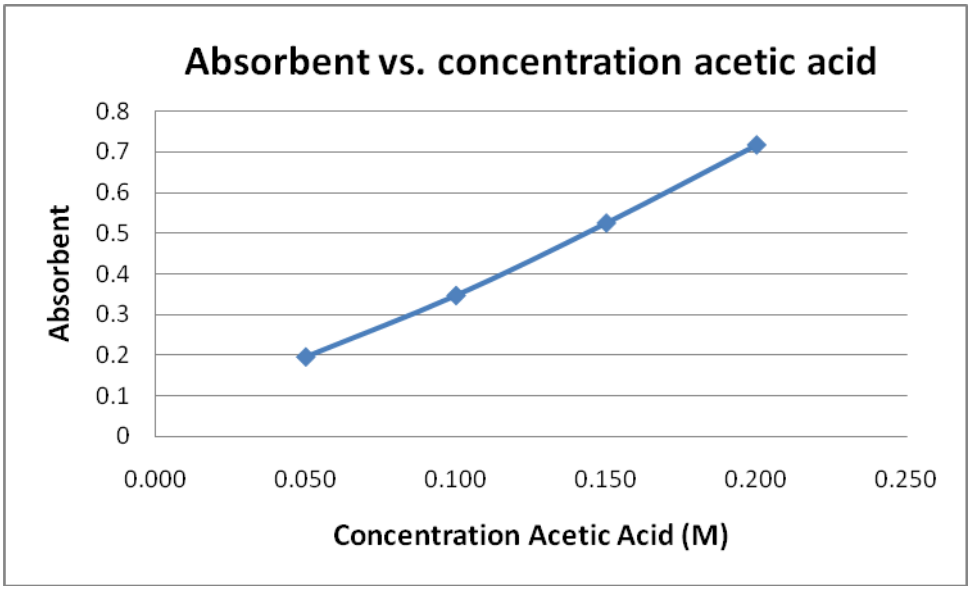

Figure 4.2: Graph Standard Solution Absorbent vs. Concentration of Acetic Acid

Table 4.6 : Absorbent for Acetic Acid each Experiment

time (min)	absorbent		
	Exp. 1	Exp. 2	Exp. 3
30	0.760	0.880	0.760
60	0.880	0.880	0.880
90	0.900	0.880	0.900
120	0.910	0.900	0.905

Table 4.7 : Absorbent for Ethanol each Experiment

time (min)	absorbent		
	Exp. 1	Exp. 2	Exp. 3
30	0.510	0.552	0.590
60	0.526	0.558	0.603
90	0.542	0.576	0.605
120	0.552	0.586	0.609

APPENDIX B

1. Calculation concentration of acetic acid

Molecular weight acetic acid = 60.05 g/mol

Experiment 1

M_1= 0.244 mol/ L × 60.05 g/mol
= 14.6522 g/L

M_2= 0.245 mol/ L × 60.05 g/mol
= 14.7126 g/L

M_3= 0.246 mol/ L × 60.05 g/mol
= 14.7723 g/L

M_4= 0.247mol/ L × 60.05 g/mol
= 14.8324 g/L

Experiment 2

M_1= 0.245 mol/ L × 60.05 g/mol
= 14.7126 g/L

M_2= 0.245 mol/ L × 60.05 g/mol
= 14.7126 g/L

M_3= 0.245 mol/ L × 60.05 g/mol
= 14.7126 g/L

M_4= 0.246 mol/ L × 60.05 g/mol

= 14.7723 g/L

Experiment 3

M_1= 0.2445 mol/ L × 60.05 g/mol

= 14.6822 g/L

M_2= 0.245 mol/ L × 60.05 g/mol

= 14.7126 g/L

M_3= 0.246 mol/ L × 60.05 g/mol

= 14.7723 g/L

M_4= 0.2465 mol/ L × 60.05 g/mol

= 14.8023 g/L

APPENDIX C

1. Percent loss of volume of solution

Experiment 1

$$percent\ loss = \frac{500ml - (product + residue)}{500ml} \times 100\%$$

$$= \frac{500ml - (200.1ml + 235ml)}{500ml} \times 100\%$$

$$= 12.98\%$$

Experiment 2

$$percent\ loss = \frac{500ml - (product + residue)}{500ml} \times 100\%$$

$$= \frac{500ml - (201.9ml + 232ml)}{500ml} \times 100\%$$

$$= 13.22\%$$

Experiment 3

$$percent\ loss = \frac{500ml - (product + residue)}{500ml} \times 100\%$$

$$= \frac{500ml - (200.2ml + 237ml)}{500ml} \times 100\%$$

$$= 12.56\%$$

APPENDIX D

1. Figure for experiment

Figure 1:Vacuum pump suction for filter the impurities in vinegar

Figure 2: Glass Cuvette for UV-Vis Spectrophotometer

Figure 3 :Heating the Vinegar in Distilling Pot

Figure 4: Receiving Flask Collect the Product

Figure 5 : Residue from Batch Distillation