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# Simulation Study of Polyglycerol Separation with Distillation Column using Aspen HYSYS

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**Abstract.** Biodiesel production results in glycerol waste can be utilized to be pure glycerol through distillation process. The bottom product of this process, also known as glycerine pitch can be extracted to obtain polyglycerol. To obtain high-purity polyglycerol for various industries, the extract solution must undergo a purification process, typically using distillation. This research investigated the possibility of utilizing extracted solution of glycerine pitch by separating polyglycerol compounds, such as diglycerol and triglycerol, using vacuum distillation. A distillation column was simulated using Aspen HYSYS software to evaluate the separation process. The operating conditions were determined using a shortcut column initially before using the distillation column. Feed concentration was varied to analyze its effect on product composition, percentage of glycerol recovery, and reboiler temperature. The simulation results showed that high-purity glycerol (99%) can be separated from polyglycerol.

**Keywords:** *aspen hysys; distillation; glycerine pitch; polyglycerol; separation.*

## 1 Introduction

The glycerol produced is 10% by weight of the total amount of biodiesel produced with a low level of purity [1]. Glycerol which is commonly used for the needs of the pharmaceutical, medical, cosmetic, toothpaste, food processing industries as a solvent, sweetener or packaging material must have a purity of 95%, while glycerol produced from the biodiesel production process contains a general purity level of 80% and in the presence of water, methanol, salt and Material Organic Non-Glycerol (MONG) [2].

The glycerol produced from the biodiesel production process must go through a purification process which will produce glycerol with a purity of at least 95% because the level of purity produced is still low and cannot be used directly for other processes [3]. The glycerol purification process that is mostly carried out in the biodiesel industry is by using the distillation process. From the distillation process will produce a bottom product in the form of glycerine pitch. Glycerine pitch produced from the glycerol refining process is still classified as waste and has not been utilized properly. Currently, glycerine pitch which is categorized as hazardous waste is only disposed of without any further utilization. With a waste disposal process cost of US$400/ton, it provides an opportunity for research that can utilize glycerine pitch so that it will provide added value to the biodiesel production process. Glycerine pitch composition generally contains 0-30% glycerol, 40-60% Matter Organic Non-Glycerol (MONG), 20-60% ash, and <2% water [4].

The extract solution produced from the glycerine pitch sample extraction process is a mixture of glycerol and polyglycerol [4]. To obtain high-value extracts, the glycerol composition that is still contained in the extract solution must be removed so that only polyglycerol compounds such as diglycerol and triglycerol are present.

One of the separation processes that can be carried out is by using vacuum distillation because of the differences in the boiling points of the three solutions which are quite high. The distillation column is an important tool in the process industry, used to separate liquid mixtures into their components through evaporation and condensation based on their relative volatility difference [5]. The distillation process has been studied from various perspectives in literature [6]. However, analyzing chemical components in a distillation process can involve physical uncertainties and cost considerations, making it essential to carry out chemical analysis in a simulation environment. The use of computational tools facilitates quick, accurate, and easy examination of quality, demand, and cost [7]. This study aimed to evaluate the polyglycerol separation process using distillation column in Aspen HYSYS simulation.

## 2 Methodology

A knowledge-based technique that takes into account both heuristic principles and the researcher's experience was employed for the processes design. Process simulation were carried out utilizing the Aspen HYSYS software. Material balances were obtained as simulation results for each technological scheme. As a result, requirements of raw materials, services, and energy were obtained. In this simulation, several approaches to operating conditions are used to show the results of the distillation process. The feed flow rate at 120 kmol/hour and the components contained in the extract solution are a mixture of glycerol, diglycerol, and triglycerol with varied concentrations. The physicochemical properties of these three components are shown in Table 1 below.

**Table 1** Physicochemical properties of the components in the extract solution.

| Components | Glycerol | Diglycerol | Triglycerol |
|---|---|---|---|
| Molecular Weight | 92 | 106 | 240 |
| Boiling point (ºC) | 290 | 407 | 493 |
| Density (g/cm$^3$) | 1.26 | 1.33 | 1.35 |

The feed is in atmospheric conditions. The pressure of the distillation column used is following the previous study at $2x10^{-3}$ mbar while the operational temperature was set at 200 ºC [8]. The glycerol content in the product is 99%.

## 2.1 Determination of the Distillation Column Operating Conditions

The simulation is carried out using a distillation column and the parameters obtained from calculations using the shortcut column. The series of processes in this simulation are shown in Figure 1.

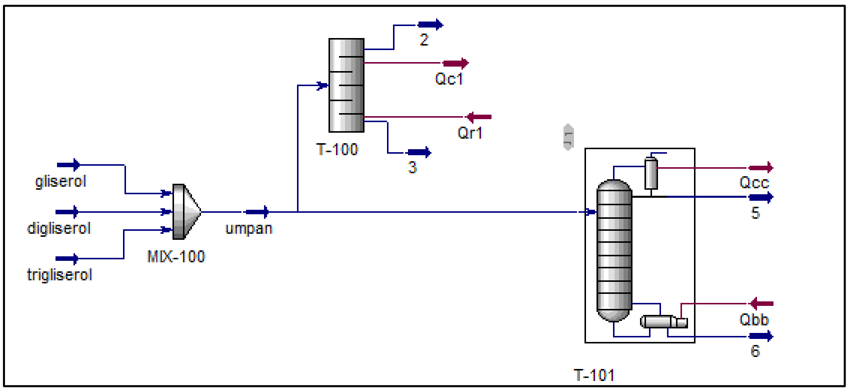


**Figure 1** Process Flow Diagram (PFD) in the simulation of the separation of glycerol in the extract solution

The operating conditions in the distillation column used are derived from distillation simulations using the shortcut column. In the early stages of determining these operating conditions, the feed composition used was 1/3 glycerol, 1/3 diglycerol, and 1/3 triglycerol with the composition and feed conditions as used shown in Figure 2.

## 2.2 Case Study on Feed Concentration Variation

The Feed Concentration is being adjusted to various number while focusing its effect on the reboiler temperature, product composition, and the percentage of glycerol recovery. The set of feed concentrations variation is listed on the Table 1.

**Table 1** Variation in variable magnitude is not fixed in the analysis of the effect of feed concentration on reboiler temperature.

| No. | Components | Flowrate of Glycerol (kmol/h) | Flowrate of Diglycerol (kmol/h) | Flowrate of Tryglycerol (kmol/h) |
|---|---|---|---|---|
| 1 | Glycerol | 10 | 40 | 40 |
| | | 15 | 40 | 40 |
| | | 20 | 40 | 40 |
| | | 25 | 40 | 40 |
| | | 30 | 40 | 40 |
| | | 35 | 40 | 40 |
| | | 40 | 40 | 40 |
| 2 | Diglycerol | 40 | 10 | 40 |
| | | 40 | 15 | 40 |
| | | 40 | 20 | 40 |
| | | 40 | 25 | 40 |
| | | 40 | 30 | 40 |
| | | 40 | 35 | 40 |
| | | 40 | 40 | 40 |
| 3 | Triglycerol | 40 | 40 | 10 |
| | | 40 | 40 | 15 |
| | | 40 | 40 | 20 |
| | | 40 | 40 | 25 |
| | | 40 | 40 | 30 |
| | | 40 | 40 | 35 |
| | | 40 | 40 | 40 |

## 3 Results and Discussion

The simulation is carried out using a distillation column and the parameters obtained from calculations using the shortcut column. By using the process series in Figure 1 and the process conditions in Table 1, the process of separating glycerol in the distillation column was successfully carried out with the resulting product composition shown in Figure 2.

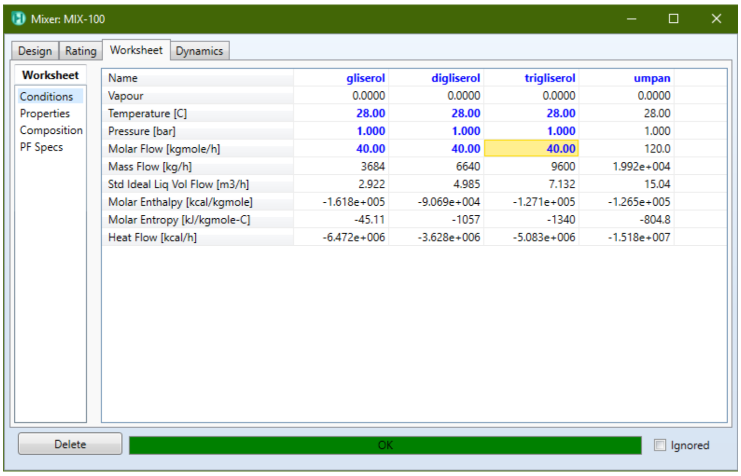


**Figure 2** Distillation feed conditions

By using the feed conditions as shown in Figure 2 condenser pressure of 2 mbar and a reboiler pressure of 3 mbar, a minimum reflux ratio of 0.28 will be produced. The actual number of reflux ratios used is the most optimum, which is 1.2 times the minimum amount of reflux [9]. From these data, design data will be obtained that can be used in simulations in the distillation column. The distillation column design data to be used is shown in Figure 3 below.

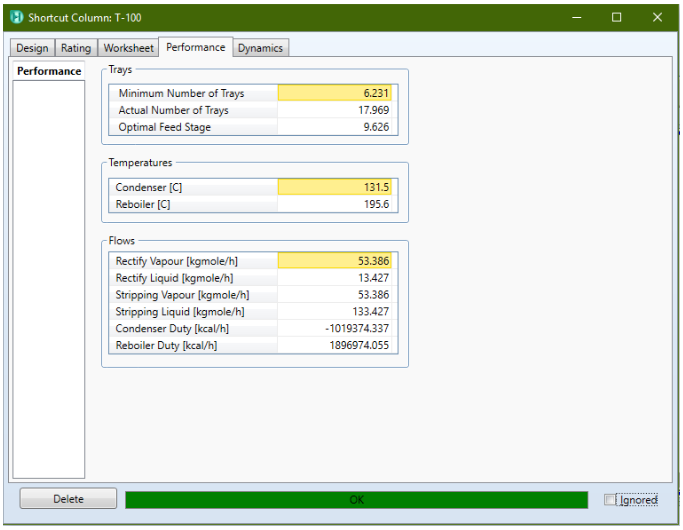


**Figure 3** Distillation column design process conditions

From Figure 3, it can be seen that using distillation at a pressure of $2x10^{-3}$ mbar can separate glycerol maximally. The glycerol content in the product solution is only 0.001 or 0.1% and the remainder is a mixture of diglycerol and triglycerol with respective levels of 49.75% and 50.15%. The level of purity of glycerol produced in this separation process is also very high, namely 99.2% with 0.8% being diglycerol carried into the distillate solution as shown in Figure 4.

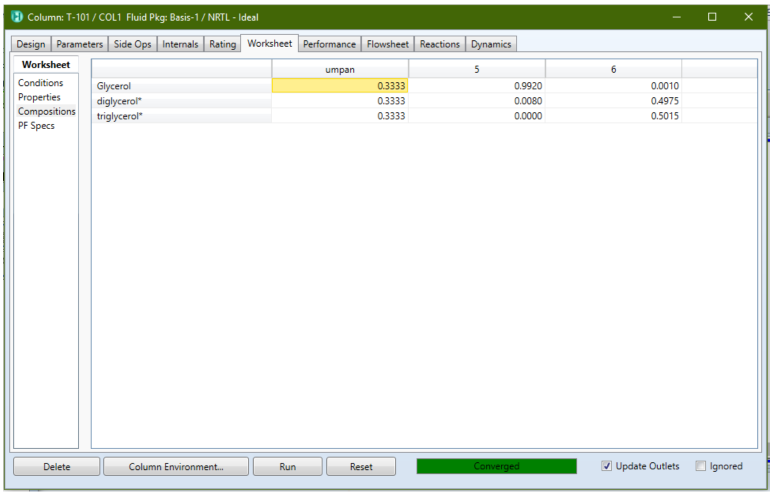


**Figure 4** The final composition of the distillation process

The parameters used in the distillation column can be seen in Figure 5 below. Besides the approach that being used on the initial setup on shortcut column, the results on the shortcut column were also applied on the distillation column setup. The total condenser system is being used in this simulation, so that the top product is entirely in the form of a liquid phase.

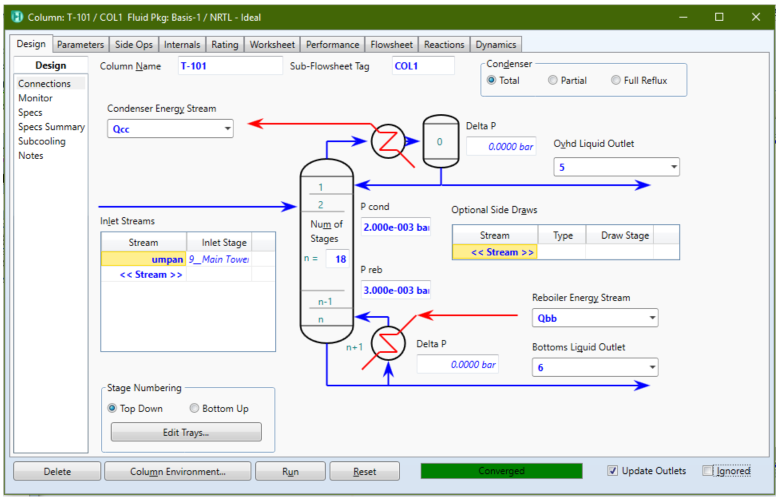


**Figure 5** Distillation column in the glycerol separation process

### 3.1 Effect of Feed Concentration Variations on the Reboiler Temperature

A change in feed concentration can affect the temperature in the reboiler. The effect of variations in bait concentration can be seen using a case study on HYSYS. This analysis uses fixed parameters in the form of the number of stages of 14 stages and the number of reflux ratios of 0.839.

A case study was conducted to see the effect of changes in the composition of each component on the achieved reboiler temperature. This can be done by selecting fixed variables and variable variables in the analysis process. The fixed variable set in this analysis is the concentration of glycerol in the product solution, where the expected concentration is 0.001. The variable that changes or is not fixed in this analysis is the flow rate of the components fed in the distillation so that it will affect the concentration of each component in the feed stream. The results of this analysis are shown in Fig 6 below

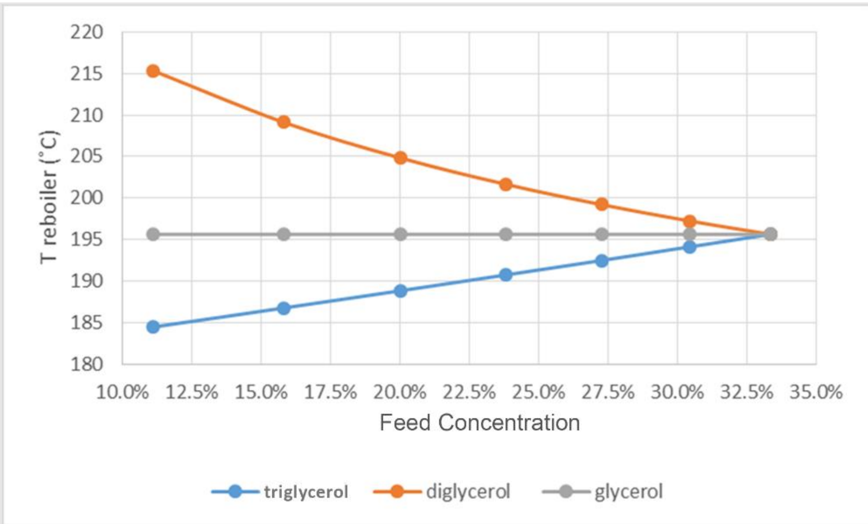


**Figure 6** Effect of changes in feed concentration on reboiler temperature

Figure 6 shows that a change in feed concentration will affect the reboiler temperature achieved to produce a product composition which is expected not to contain more than 0.001 or 0.1% glycerol. Changes in the concentration of each component have different effects. In the triglycerol component, an increase in concentration will result in a higher reboiler temperature, whereas in the diglycerol component, changes in the concentration of the feed will decrease the reboiler temperature achieved.

Reboiler temperature is an important parameter in process selection and operating conditions for a separation process. The use of reboiler temperatures above 200 ˚C is not recommended for selection because there is a possibility of cracking.

## 3.2 Effect of Feed Concentration Variations on Product Composition

Changes in feed concentration are also able to affect the quality of the product produced. This analysis was carried out using a fixed variable in the form of a reboiler temperature of 180 ˚C, so that a change in the composition of the resulting product will be seen with a change in the composition of each component in the feed stream. The results of the analysis are shown in Figure 7.

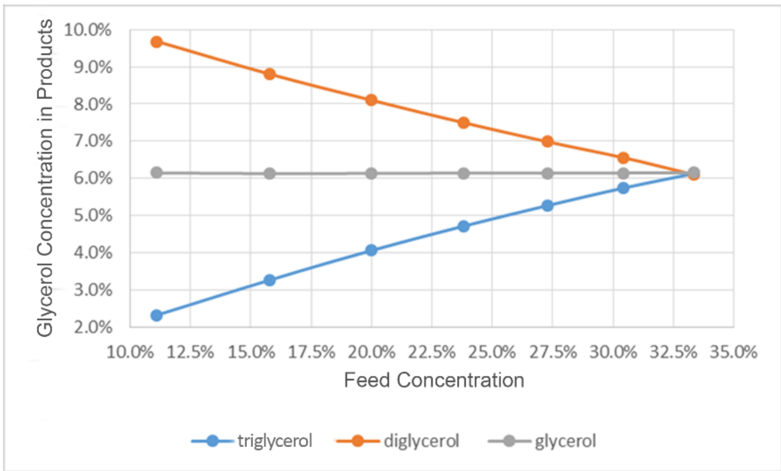


**Figure 7** Effect of changes in feed concentration on product composition

Figure 7 shows that a change in feed concentration will affect the product composition achieved in the separation process using a temperature of 180 ˚C. Changes in the concentration of each component have different effects. In the triglycerol component, an increase in concentration will result in a lower glycerol concentration in the product. This indicates that the glycerol separation process is getting better, while in the diglycerol component, a change in concentration in the feed will increase the glycerol concentration in the product, thus indicating a poor separation process because it is still high levels of glycerol which is still carried into the product solution.

## 3.3 Effect of Reboiler Temperature Variations on Percentage of Glycerol Recovery

The next analysis was carried out to see the effect of changing the temperature of the reboiler used on the % recovery of glycerol in the distillate solution. This analysis was carried out using a non-fixed variable in the form of reboiler temperature. Reboiler temperature is varied to see the effect on the quality of the product produced. The temperature variations used are in the range of 160 ˚C – 190 ˚C with a temperature rise level of 5 ˚C.

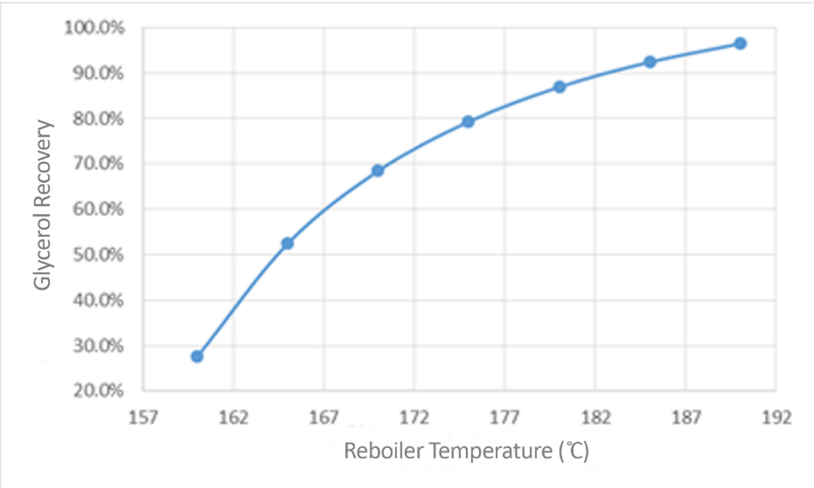


**Figure 8** Effect of reboiler temperature on recovery of glycerol in the distillate

The higher the recovery of glycerol in the distillate solution indicates the better the separation process. A high amount of recovery in the distillate solution can indicate that the amount of glycerol separated from the mixture is higher. The results of this analysis can be shown in Figure 8.

## 4 Conclusions

The simulation results show that the process of separating glycerol from the extract solution can be carried out using vacuum distillation at a condenser pressure of $2x10^{-3}$ mbar and the resulting reboiler temperature does not exceed 200 ˚C.

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