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Research Article

Improving Purity Propylene Glycol by Modifying Glycerol Hydrogenolysis Process

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Abstract

The chemical industry in Indonesia continues to grow in terms of innovation and technology. One field that has experienced a significant increase is supporting materials, such as propylene glycol. The high demand for propylene glycol in its pure form makes modification of propylene glycol necessary. This modification is done by adjusting the molar ratio of hydrogen to glycerol by 5:1 which involves the process of hydrogenolysis. The physical condition (liquid or gas) of each compound was determined based on the vapor pressure equilibrium data of the pure compound, which was calculated using Hysys software with 2 property packages, namely the NRTL (Non-Random Two-Liquid) model and the uniquac model to determine the actual composition of each component. To obtain a high purity of propylene glycol, the process was modified with the addition of a mixer, recycle, splitter, and two distillation processes. The use of two distillations is used to obtain more optimal results. In the Aspen HYSYS simulation, the reactor used for this process is a conversion reactor because the conversion reactor is the basis of the fluidized bed reactor. From the method that has been carried out, it is found that this modification is very effective in obtaining high propylene glycol purity. The percentage of propylene glycol in the final product increased from 73.5% to 90%.

Keywords: Glycerol; Propylene Glycol; Hydrogenation; Product Purity; Distillations; Hydrogenolysis

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1. Introduction

The growth of the renewable energy industry continues to show an increasing trend from year to year. One form of renewable energy that is being developed is biodiesel, which is an alternative fuel produced through a transesterification process between alcohol and vegetable oil or animal fat [4]. In the biodiesel production process, glycerol is a major by-product, contributing about 10% of the total weight of biodiesel produced. Therefore, it is necessary to develop new technologies to utilize glycerol more effectively by converting it into high-value products [16]. One of the products that can be produced from glycerol is propylene glycol, an organic compound that has various applications in the pharmaceutical, cosmetic, food, and general chemical industries [3]. The main reactions of glycerol hydrogenolysis to propylene glycol defined as [6]:

$$C_3H_8O_{3\,(l)} + H_{2\,(g)} \rightarrow C_3H_8O_{2\,(l)} + H_2O_{\,(g)} \qquad (1)$$

To improve conversion, selectivity, and product yield, various approaches have been applied, including adjustment of operational

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parameters and use of optimized catalysts to create ideal reaction condition [10]. Sari *et al.* tested the hydrogenation of glycerol to propylene glycol using NiO-MgO/Bentonite and NiO-MgO/carbon catalysts, which resulted in glycerol conversions of 50.4% and 52.9%, respectively [13]. However, the use of these catalysts requires high costs and additional processes to handle the inactive catalysts. Furthermore, Seretis & Tsiakaras, conducted glycerol hydrogenolysis experiments at reaction temperatures of 200 °C, 220 °C, and 240 °C, with autogen pressures of 16, 23.5, and 33.5 bar, respectively. These tests showed that the highest glycerol conversion recorded was 73.5% [16]. Based on previous research, the results obtained have experienced a significant increase. However, these results still did not reach 90%. In addition, there is no study that discusses the effect of mole ratio on the conversion obtained. Therefore, modifications are needed in the propylene glycol production process from glycerol hydrogenolysis with a capacity of 20,000 tons per year to achieve higher conversion, namely by setting the molar ratio of hydrogen to glycerol at 5:1, using ASPEN HYSYS V.11 2019 software. This paper also discusses modifications to the basic flowsheet of the propylene glycol production process from glycerol hydrogenolysis, with the aim of increasing the conversion in the process.

2. Method

To improve the purity of propylene glycol through modification of the glycerol hydrogenolysis process, water, glycerol, propylene glycol (PG), and hydrogen are required as components included in the process simulation. In this study, the reaction and separation units involve gas-liquid and liquid-liquid multiphase systems. The physical condition (liquid or gas) of each compound is determined based on the vapor pressure equilibrium data of the pure compound, which is calculated using HYSYS software with 2 property packages, namely the NRTL (Non-Random Two-Liquid) model and the uniquac model to determine the actual composition of each component [15]. NRTL (Non-Random Two-Liquid) is an activity model used in thermodynamics to predict the behavior of non-ideal mixtures, especially in liquid mixtures. This model serves to calculate the activity of components in the mixture. Activity is a parameter that indicates the extent to which a component in a liquid mixture approaches the behavior of the pure substance and its deviation from ideal conditions [18]. NRTL models are used in chemical process simulations to model the interactions between components in a mixture, predict the distribution of each component at various process points, and support overall process optimization [17]. The phase equilibrium of non-ideal mixed systems is calculated using the NRTL Model. These calculations include the determination of dew point, freezing point, as well as various thermodynamic parameters required to analyze and design a process [1].

Our modification of propylene glycol production process is based on the existing literature. We modified the propylene glycol production process directly through the glycerol hydrogenolysis process with a capacity of 30,000 tons per year by adding a distillation equipment at the end of the process so that there are two distillation columns in this propylene glycol production process. The purpose of adding a distillation column in this study is to increase the purity of the product we want, namely high purity propylene glycol. This is based on existing research, where the distillation process removes water and other unwanted compounds that may form during the reaction, thus pushing the reaction equilibrium to the right to produce a purer product and ensure high reaction performance [20].

In this process, a mixer (M-100) is used to mix the materials with an output flow rate of 3328.359 kg/h and a molar ratio of hydrogen to glycerol of 5:1. To minimize side reactions, the amount of hydrogen is deliberately made excessive so as to increase the selectivity of the glycerol introduced into the process and produce a product in the form of propylene glycol with a high yield. After being mixed in the mixer (M-100), the feed from the mixer output stream was then fed into the reactor (R-100). In the Aspen HYSYS simulation, the reactor used for this process is a conversion reactor because the conversion reactor is the basis of the fluidized bed reactor. The conversion reactor operates at a temperature of 547.25 K and a pressure of 4 MPa, where the process conversion obtained is 95%. The reaction that occurs is exothermic, so a cooling jacket is used to maintain the reactor temperature with air at 298 K as the coolant.

The methodology described by Seider [14], which is based on the relative volatility of the compounds used for the synthesis of the separation system. The reactor outlet is a two-phase stream, i.e. from the most volatile to the least volatile consisting of hydrogen, methanol, water, acetol, propylene glycol, ethylene glycol and glycerol. Hydrogen is the first component to be removed, as it is an excess reagent and can be recycled into the process, thus reducing feedstock expenditure. Thus, a flash vessel is used for the separation of hydrogen from the effluent stream under vacuum conditions by evaporation of the more volatile components in a homogeneous mixture [11]. The reactor output enters the separator (V-100) to separate excess hydrogen from the desired product, propylene glycol. Then a tee valve is used in the upper product separator to separate the hydrogen used for recycling from the hydrogen that has been contaminated into by-products and exits as purge steam. purge steam from excess hyrdogen can be used as fuel but the concentration of purge gas must meet standard requirements.

The hydrogen to be used for recycling from the tee valve is passed to the splitter (X-100) for component separation, so as to produce pure hydrogen as the top product to be recycled for re-entry into the feed flow rate and the bottom product (other components), which then enters the distillation column. In contrast, the bottom product from the separator (V-100) together with the bottom product from the splitter (X-100), which contains propylene glycol, is passed through the distillation column. The output of the distillation column is again fed into the last distillation column to obtain propylene glycol with a higher concentration. From the distillation column, the top product obtained consists of water and residual hydrogen gas, while the bottom product obtained is propylene glycol with a high concentration.

3. Result and Discussion

The simulation of propylene glycol production through the glycerol hydrogenolysis process, for both the basic and modified processes, using Aspen HYSYS is shown in Figure 1 and Figure 2. The process flow diagram (PFD) of propylene glycol production through the glycerol hydrogenolysis process, for both the basic and modified processes, is shown in Figure 3 and Figure 4. In this study case, glycerol hydrogenation uses pure glycerol and hydrogen as raw materials. The reaction that occurs in the process of producing propylene glycol from glycerol through glycerol hydrogenolysis is as follows:

$$C_3H_8O_3 + H_2 \rightarrow C_3H_8O_2 + H_2O \quad (2)$$

The process of making propylene glycol from glycerol is modified with ASPEN HYSYS, where separator, tee valve, splitter, mixer, and recycle

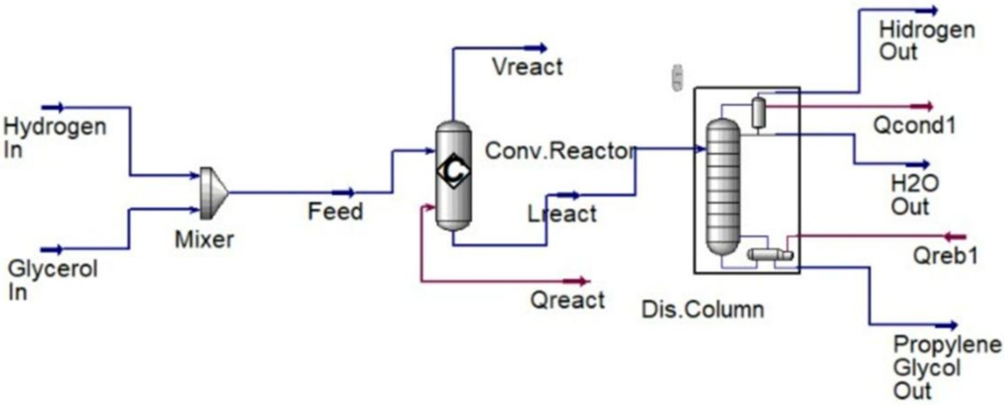

Figure 1. Simulation propylene glycol production before modified with Aspen HYSYS V11

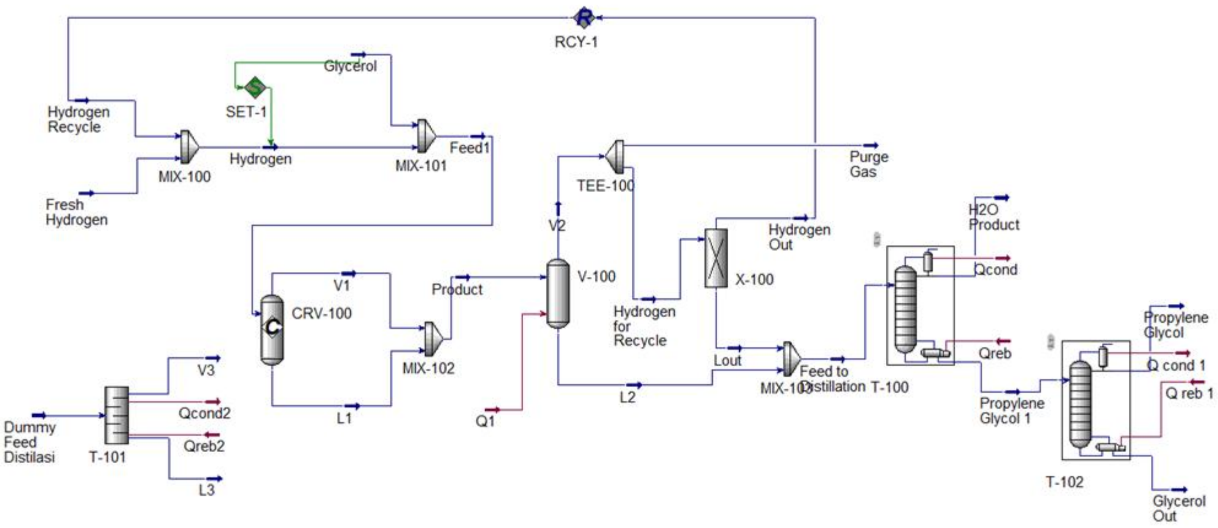

Figure 2. Propylene glycol production flowsheet after modified process

were added. The propylene glycol process flowsheet after modified can be seen in Figure 2.

The PG process flowsheet can be seen in Figure 2. Glycerol and hydrogen are mixed in mixer M-100 before entering conversion reactor CRV-100. Conversion reactors can be used to quickly calculate reaction products also might be used in place of a plug flow reactor with multiple reactions inside a recycle loop in order to save time. Another reason for the conversion reactor been used in this simulation is because conversion reactor is a basic for the fluid bed reactor [5]. The reaction in the reactor was carried out with a temperature of 547.25 K, a pressure of 4 MPa, and a mole ratio of glycerol and hydrogen of 1:5. Amount of hydrogen is made into excess with the aim of minimizing the occurrence of reactions side, so that the selectivity of the glycerol included in the process will increase and produce propylene glycol with a high yield [2]. The temperatures are set to 547.25 K (274.1°C) at the reactor, because the increase in reaction temperature from 210 to 270 °C led to a large increase in the glycerol conversion from 21.9 to 99.6% [8]. At these conditions, the conversion of glycerol is not dependent on its concentration, so glycerol's stream is not diluted [9]. Vasiliadou & Lemonidou [19] conducted research at a reaction temperature of 200-270 ^{o}C. The results showed that higher temperature (270 ^{o}C) is favorable for glycerol hydrogenolysis. The glycerol conversion rate increased with pressure because there were more H2 molecules available around the glycerol. But, the higher pressure (>4.5 MPa) is favorable for the formation of degradation products of propylene glycol, resulting in decreased selectivity of propylene glycol [7]. The selectivity toward propylene glycol slightly increased from 72.3 to 73.0% and further sharply decreased to 63.0% at 6 MPa. Therefore, the optimal pressure to obtain the maximum yield was 4 MPa [12].

Then, the reactor output (68.5% hydrogen vapor product and 59.3% PG liquid product) enters the separation section. The mixture from MIX-102 is separated using flash vessel V-100 with a pressure of 4 MPa and a temperature of 547.15 K so that hydrogen becomes the top product (vapor) and PG becomes the liquid phase. The top product of the flash vessel enters the TEE-100 splitter to remove some of the gas. Part of the gas enters the splitter X-100 to separate all hydrogen from the gas which is then recycled, while the lower product enters the distillation column T-100. In addition, the bottom product of flash vessel V-100 is also fed to distillation. The distillation process aims to increase the concentration of PG. The upper yield of distillation is hydrogen gas (84.4%), while the lower yield is PG with a percentage of 90%. The lower yield is subjected to a further distillation process to obtain a purer yield of propylene glycol by entering the distillation column T-101. The upper yield of the second distillation is propylene glycol gas (99.5%), while the lower yield is glycerol and little amount of propylene glycol with a percentage of 90%. Based on the relationship between the reactant mole ratio and PG yield, the simulation in HYSYS V.11 is in accordance with the theory.

The reactor effluent primarily consists of propylene glycol, hydrogen, unreacted glycerol, and by-products. It is then sent to the separation section, where excess hydrogen is recovered using a flash vessel, a tee valve, and a splitter. The recovered hydrogen is recycled and mixed with pure hydrogen to maintain a 5:1 molar ratio of hydrogen to glycerol at the process inlet [4]. At the start of the reaction, hydrogen is used in excess. If unused hydrogen is not recycled by the end of the process, production costs will rise due to the high material requirements. Recycling hydrogen helps mitigate these costs. The separator reduces the distillation load, optimizes yield, and minimizes the heat required for the process. Additionally, the separator facilitates the recovery of hydrogen, which is subsequently recycled. The splitter is used to separate components with similar compositions, ensuring pure hydrogen is obtained for recycling. Ultimately, this process results in a purer propylene glycol product.

The calculation of heat/enthalpy of the reaction at the operating temperature (547.25 K) (Tables 1 and 2). Based on the purity data between the basic propylene glycol production process and the modified propylene glycol production process (Table 3), it can be compared that propylene glycol with the modified process has a higher purity of 99%, with a difference of 26% between the two processes.

Table 1. Gas heat capacity constant of each compound

Compounds Name	Cp (Joule/(mol.K))				
	A	B	C	D	E
Glycerol	132.145	0.86007	-0.00197450	1.81E-06	0
Hydrogen	25.399	0.020178	-0.00003859	3.19E-08	-8.76E-12
Propylene Glycol	118.614	0.67283	-0.00183770	2.13E-06	0
Water	33.933	-0.0084186	0.00002991	1.78E-08	3.69E-12

$$\Delta H_{f500} = \Delta H_{f298} + \int_{298}^{547.25} C_p \, dT$$

$$C_p = \int_{298}^{547.25} (\mathrm{A} + \mathrm{BT} + \mathrm{CT}^2 + \mathrm{DT}^3 + \mathrm{ET}^4) dT$$

$$C_p = \left[\mathrm{AT} + \frac{1}{2}\mathrm{BT}^2 + \frac{1}{3}\mathrm{CT}^3 + \frac{1}{4}\mathrm{DT}^4 + \frac{1}{5}\mathrm{ET}^5\right]_{298}^{547.25}$$

$$\Delta H_{f547.25} = \Delta H_{f298} + \left[\mathrm{AT} + \frac{1}{2}\mathrm{BT}^2 + \frac{1}{3}\mathrm{CT}^3 + \frac{1}{4}\mathrm{DT}^4 + \frac{1}{5}\mathrm{ET}^5\right]_{298}^{547.25}$$

Glycerol ($C_3H_8O_3$):

$$\Delta H_{f547.25} = -582800 \frac{J}{mol} + \left[132.145T + \frac{1}{2}(0.86007)T^2 + \frac{1}{3}(-0.00197450)T^3 + \frac{1}{4}(1.81 \times 10^{-6})T^4\right]_{298}^{547.25} = -512698.236 \, J/mol$$

Hydrogen (H_2):

$$\Delta H_{f547.25} = 0 \frac{J}{mol} + \left[25.399T + \frac{1}{2}(0.020178)T^2 + \frac{1}{3}(-0.00003859)T^3 + \frac{1}{4}(3.19 \times 10^{-8})T^4\right]_{298}^{547.25} = 7422.71129 \, J/mol$$

Propylene Glycol ($C_3H_8O_2$):

$$\Delta H_{f547.25} = -421500 \frac{J}{mol} + \left[118.614T + \frac{1}{2}(0.67283)T^2 + \frac{1}{3}(-0.00183770)T^3 + \frac{1}{4}(2.13 \times 10^{-6})T^4\right]_{298}^{547.25} = -361683.54 \, J/mol$$

Water (H_2O):

$$\Delta H_{f547.25} = -241800 \frac{J}{mol} + \left[33.933T + \frac{1}{2}(-0.0084186)T^2 + \frac{1}{3}(0.00002991)T^3 + \frac{1}{4}(-1.78 \times 10^{-8})T^4 + \frac{1}{5}(8.76 \times 10^{-12})T^5\right]_{298}^{547.25} = 7422.71129 \, J/mol$$

Total ΔH of the reaction:

$C_3H_8O_3(l) + H_2(g) \rightarrow C_3H_8O_2(l) + H_2O(g)$

$$\Delta H_{f298} = \left[\left(\Delta H_{f298} C_3H_8O_{2(l)} + \Delta H_{f298} H_2O_{(l)}\right) - \left(\Delta H_{f298} C_3H_8O_{3(l)} + \Delta H_{f298} H_{2(g)}\right)\right] J/mol$$

$$\Delta H_{f298} = [(-421500 + (-241800)) - (-582800 + 0)] \frac{J}{mol} = -80500 \, J/mol$$

$$\Delta H_{f547.25} = \left[\left(\Delta H_{f547.25} C_3H_8O_{2(l)} + \Delta H_{f547.25} H_2O_{(l)}\right) - \left(\Delta H_{f547.25} C_3H_8O_{3(l)} + \Delta H_{f547.25} H_{2(g)}\right)\right] J/mol$$

$$\Delta H_{f547.25} = [(-361683.54 + (-233188.39)) - (-512698.236 + 7422.71129)] \frac{J}{mol} = -89596.407 \, J/mol$$

The reaction is carried out at a temperature of 547.25 K. From the above calculations, the values of ΔH_{r298} and $\Delta H_{r547.25}$ are −80500 J/mol and −89596.407 J/mol, respectively. Thus, it can be concluded that the occurring reaction is exothermic.

Calculation of Gibbs Energy:

$C_3H_8O_3(l) + H_2(g) \rightarrow C_3H_8O_2(l) + H_2O(g)$

$$\Delta G_{f298} = \left[\left(G_{f298} C_3H_8O_{2(l)} + G_{f298} H_2O_{(g)}\right) - \left(G_{f298} C_3H_8O_{3(l)} + G_{f298} H_{2(g)}\right)\right] J/mol$$

$$\Delta G_{f298} = \left[\left(-304480 + (-228600)\right) - (-448490 + 0)\right] \frac{J}{mol} = -84590 \, J/mol$$

Calculation of reaction equilibrium constant at operating temperature (547.25 K):

$$\Delta G_{298} = -RT \ln K_{298}$$

$$\ln K_{298} = -\frac{\Delta G_{298}}{RT}$$

$$\ln K_{298} = -\frac{-84590 \, J/mol}{(8.314 \frac{J}{mol.K})(298K)}$$

$$K_{298} = 6.7268 \times 10^{14}$$

$$\ln \frac{K_{547.25}}{K_{298}} = \frac{(\Delta H_{r\,298})}{R} \times \left(\frac{1}{547.25K} - \frac{1}{298K}\right)$$

$$\ln \frac{K_{547.25}}{6.7268 \times 10^{14}} = \frac{\left(-(-80500 \frac{J}{mol})\right)}{8.314 \frac{J}{mol.K}} \times \left(\frac{1}{547.25K} - \frac{1}{298K}\right)$$

$$K_{547.25} = 2.517 \times 10^8$$

Table 2. Data on ΔH_{f298} and ΔG_{f298} compounds [20]

Compounds Name	ΔH_{f298} (kJ/mol)	ΔG_{f298} (kJ/mol)
Glycerol	-582.8	-448.49
Hydrogen	0	0
Propylene Glycol	-421.5	-304.48
Water	-241.8	-228.6

Table 3. Comparison of propylene glycol purity between before and after modified process

Purity of propylene glycol before modified process (%)	Purity of propylene glycol after modified process (%)
73	99

The obtained value of K_{500} for this side reaction is 2.517×10^{8}. Due the large equilibrium constant value, the reaction is irreversible.

4. Conclusion

Modifications in the production of propylene glycol from glycerol were made to obtain high product purity. In modeling and simulating the production process of propylene glycol from hydrogen and glycerol through hydrogenation process, ASPEN HYSYS V.11 is used. To obtain a high purity of propylene glycol, the process was modified with the addition of a mixer, recycle, splitter, and two distillation processes. The use of two distillations is used to obtain more optimal results. The purity of propylene glycol obtained when using one distillation column is only 90%, so to increase product purity a second distillation is required. The use of two distillations with the incoming feed being the result of the first distillation shows an increase in propylene glycol purity, which is 99%.

CRediT Author Statement

Author Contributions: M. Hapsari: Conceptualization, Methodology, Validation, Investigation, Resources, Formal Analysis, Writing - Original Draft, Writing - Review and Editing, Visualization, Supervision; H. M. Istanto: Conceptualization, Methodology, Formal Analysis, Data Curation, Writing - Original Draft, Writing - Review & Editing, Visualization, Software, Project Administration; A. R. Fiya: Conceptualization, Methodology, Validation, Writing - Original Draft, Formal Analysis; A. R. Azizah: Conceptualization, Methodology, Investigation, Resources, Writing - Original Draft; N. Fuzyawan: Formal analysis, Data

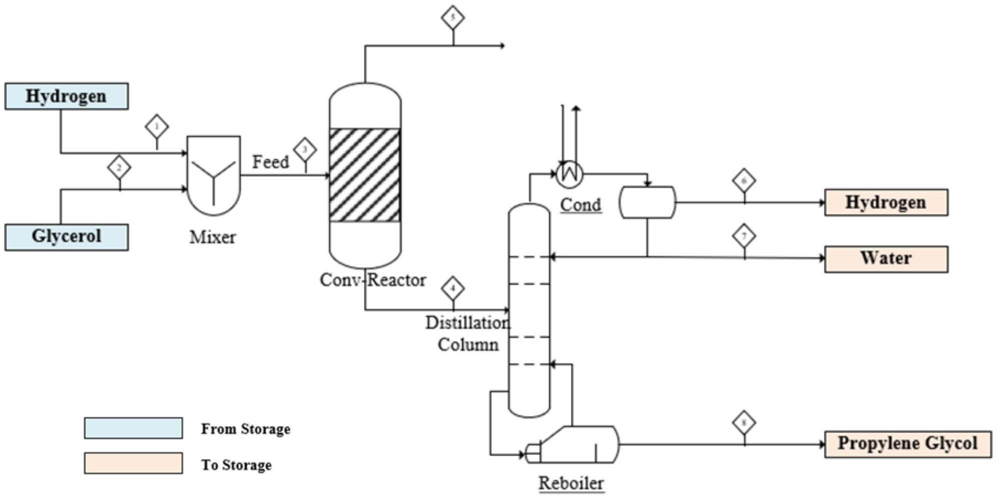

Figure 3. Process flow diagram of propylene glycol production before modification

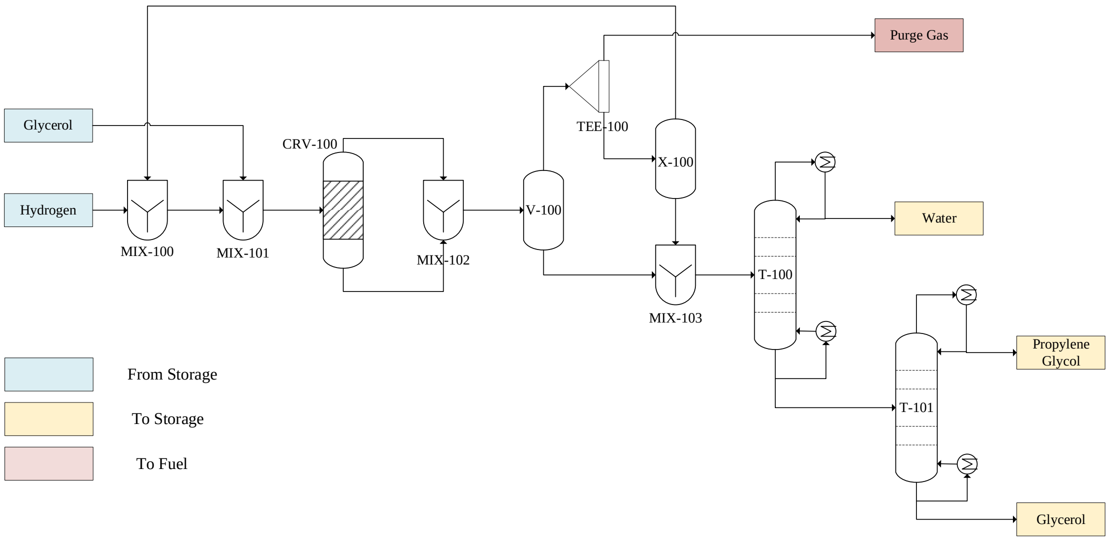

Figure 4. Process flow diagram of propylene glycol production after

curation, Visualization. All authors have read and agreed to the published version of the manuscript.

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