

# Improving Cyclohexane Yield from Hydrogenation Benzene with a Modified Multistage Separation Design

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## Abstract

Cyclohexane is a compound that is widely used in various industrial applications as a chemical intermediate. Apart from that, this compound can also be used to produce solvents, insecticides and plasticizers. With the large use of cyclohexane, production with high efficiency in terms of yield is required. The aim of this research is to develop a process design for producing cyclohexane from benzene hydrogenation, with the aim of achieving high yield. This research uses an iterative simulation method to compare the basic process and a modified process for cyclohexane production. This process involves creating a simulation model using Aspen HYSYS and adjusting the process parameters until the desired results are achieved. The results of this research indicate that the cyclohexane produced in the modification process achieves a higher percentage of yield product. Based on the implemented process modification, there has been an increase in the yield of cyclohexane produced from 93.49% to 99.90%. Based on the results of this research, the modification process is proven to be able to increase yield and mass flow compared to the basic process system.

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**Keywords:** Cyclohexane; Yield; Hydrogenation; Benzene; Aspen HYSYS

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## 1. Introduction

Hydrogenation of benzene to produce cyclohexane is an industrially important petrochemical process [1]. Cyclohexane are indispensable raw chemical widely used as feedstock of cyclohexanol and cyclohexanone, which are eventually converted into adipic acid and caprolactam for nylon [2]. Cyclohexane is mainly synthesized by hydrogenation of benzene, and the product inevitably contains benzene [3]. In aerobic oxidation processes, thermodynamically stable and kinetically inert C-H bonds in cyclohexane under sustainable and environmentally safe conditions remain a major

challenge in current academic and industrial research. The industrial production of C6 monomers, such as Nylon 6 and Nylon 66 [4], is usually carried out by the liquid phase cyclohexane oxidation process [5]. Cyclohexane is primarily utilized as an intermediate chemical. Approximately 54% of its production is allocated for adipic acid synthesis, which is essential for Nylon-6/6, while 39% is used in caprolactam production for nylon-6. The remaining 7% is employed in various applications, including solvents, insecticides, and plasticizers. The demand for nylon, and consequently for cyclohexane, is increasing by about 6% annually, driven by its use in engineering thermoplastics for resins and films. These thermoplastics are renowned for their high tensile strength, exceptional abrasion resistance, chemical

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stability, and heat resistance, making them increasingly popular for mechanical applications traditionally dominated by metal components [6]. Globally, the total annual production capacity of cyclohexane is approximately 1.800 million gallons, with the U.S. accounting for around 600 million gallons. Chevron-Phillips alone produces 205 million gallons annually in the U.S. Cyclohexane demand is expected to grow at an average annual rate of 2%. However, Japan has experienced a decline in cyclohexane demand due to the adoption of a new adipic acid production method that uses cyclohexene instead of cyclohexane [7].

Both patents and technical literature have been reviewed regarding the conventional production of cyclohexane through benzene hydrogenation. While the processes are generally similar, they vary in the selection of catalysts to achieve a high degree of hydrogenation, ensuring the final product meets required specifications [8]. The main by-products of this process include n-hexane, methyl-cyclopentane, methyl-cyclohexane, methyl-pentane, n-pentane, and methane. Proper control and maintenance of the reactor's temperature are critical to minimizing by-product formation. A significant challenge in cyclohexane production is the deactivation of the reactor's catalyst bed caused by carbon monoxide (CO) in the hydrogen source [9]. To address this, a pre-treatment reactor unit, known as a methanator, is used to convert CO into methane and water before introducing hydrogen into the main reactor. Literature also includes sensitivity analyses on temperature control to determine the optimal phase for the reaction. Although cyclohexane production is predominantly conducted in the vapor phase, some reports suggest that a mixed-vapor phase (a combination of liquid and gas) provides better temperature regulation in the reactor. However, with carefully selected temperatures, pressures, and catalysts, a liquid-phase reaction can effectively minimize by-products while ensuring excellent temperature control in the reactor [10].

In this case, the modification is proposed by adding recycle, TEE, valve, heat exchanger, separator, and rigorous column. This modification improves yield of cyclohexane of the hydrogenation benzene. In addition, to remove any remaining benzene and minimize the methane formed as a by-product. The aim for this research is to enhancing yield of cyclohexane in the hydrogenation of benzene to produce cyclohexane by adding several unit using Aspen HYSYS V11.

## 2. Methods

The production process model for cyclohexane from hydrogenation of benzene was designed using Aspen HYSYS V11 by adding components cyclohexane, benzene, hydrogen, nitrogen, methane, and H<sub>2</sub>O. The fluid package employed was Peng-Robinson. The selection of Peng-Robinson for this process is due to its involvement with hydrocarbon compounds and its suitability for analyzing gas-liquid-liquid phase equilibrium, and phase equilibrium in multicomponent mixture system. Hence, this equation is suitable for use in the cyclohexane production process [11]. To maximize the cyclohexane yield, a process modification was carried out by adding recycle, TEE, valve, heat exchanger, separator, and rigorous column. The separator unit was added to split the top and bottom product that will proceed to the recycle and rigorous column to separate cyclohexane and benzene. To meet the industrial demand for high-yield cyclohexane, the separation of benzene and cyclohexane is of great significance. However, the molecular dynamic diameters of benzene and cyclohexane are similar, with boiling point difference of only 0.6 °C and an azeotrope will form at 55%/45% [12]. Traditional separation methods such as azeotropic distillation, extraction distillation, adsorption, and extraction are difficult and costly [13]. This process modification can enhance cyclohexane production by minimizing the use of raw materials and energy. Cyclohexane yield can be calculated using Equation (1) [14]:

$$\text{Yield of Cyclohexane (\%)} = \frac{\text{mol of cyclohexane}}{\text{mol of benzene}} \times 100\% \quad (1)$$

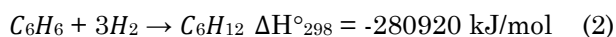
From Equation (1), we have the result of yield of cyclohexane. Table 1 shows comparison between processes with and without modification in terms of cyclohexane yield.

## 3. Results and Discussion

### 3.1 Process Description of Basic Process Before Process Modification

Method for producing cyclohexane by the hydrogenation of benzene. The simulation of cyclohexane production were simulated on Aspen HYSYS V11 processes that obtained were 93.49% in the liquid phase. The basic (unmodified) process flow diagram of cyclohexane production is shown in Figure 1 and the Aspen HYSYS simulation (unmodified) is shown in Figure 2. The Peng-Robinson fluid package was chosen for the simulation process. In the benzene hydrogenation was an exothermic process with the operation condition for the conversion reactor at the temperature of 37.7 °C with a pressure 335 psia.

The feed, containing of liquid benzene at a temperature of 37.8 °C with pressure 110 psia and hydrogen gas at a temperature of 48.9 °C with pressure of 335 psia. Pump use for the stream feed of liquid benzene from 110 psia to 335 psia. To take into consideration for capable of calculating various phases, the Peng-Robinson thermodynamic model was chosen for the process. The chemistry used for this process is presented in Equation (2) [15]:



The raw materials was benzene, hydrogen, and nitrogen (inert), undergo a reaction in a mixer at a temperature 37.7 °C and 335 psia before entering the fixed-bed type reactor (conversion reactor). The purpose of the mixer is to homogenize benzene and hydrogen (pretreatment), ensuring a more homogeneous and evenly mixed compound upon entering the reactor [16]. The fixed bed reactor is chosen and operates at a temperature of 710.6 °C and a pressure of 335 psia. This reactor was chosen for its ability to effectively react with gaseous reactants. Reactor product, comprising hydrogen and cyclohexane with partially no benzene (benzene concentration may be controlled as desired) [17]. The reactor is output gas is then conditioned to operating conditions through a cooler which aims to cool the product before being directed to the separator. Within the separator, residual benzene and cyclohexane is separated from other compound including nitrogen and methane, which are by-products [18]. A single

stage distillation process is employed to further separate these compounds. The distillation process involves separating compounds based on differences in their boiling points [19]. The distillation process takes place at a temperature of 32.2 °C. Methane has a boiling point of -161.5 °C and cyclohexane has a boiling point of 80.7°C [20]. In this distillation, methane evaporates and exits as a gas product, while cyclohexane is collected as a bottom product (liquid) [21].

The primary reaction involves the production of cyclohexane through the interaction of benzene and hydrogen. This main reaction, there are one by-product, which is methane [22]. For the determination of the nature of the reaction exothermic and the direction of the reaction irreversible, it is necessary to calculate the standard heat of reaction ( $\Delta H_{298K}^\circ$ ) at 1 bar and 298 K based on standard heat of formation of the reactants and products. The value of  $\Delta H_f^\circ$  and  $\Delta G_f^\circ$  can be seen in Table 1.

Standard heat of reaction at 298 K ( $\Delta H_{298K}^\circ$ ):

$$\begin{aligned} (\Delta H_{298K}^\circ) &= \sum \Delta H_f^\circ \text{ product} - \sum \Delta H_f^\circ \text{ reactan} \\ &= (\Delta H_f^\circ C_6H_{12}) - (\Delta H_f^\circ C_6H_6 + \Delta H_f^\circ H_2) \\ &= (-123.14 \text{ kJ/mol}) - (82.93 \text{ kJ/mol}) \\ &= (-206.07 \text{ kJ/mol}) \end{aligned}$$

Based on the calculations, we get the value  $\Delta H_{298K}^\circ = -206.07 \text{ kJ/mol}$ , which is a negative value so that the reaction is exothermic.

Table 1. The value of  $\Delta H_f^\circ$  and  $\Delta G_f^\circ$  [23]

Compound	Molecular Formula	$\Delta H_f^\circ$	$\Delta G_f^\circ$
Benzene	C <sub>6</sub> H <sub>6</sub>	82.93	129.66
Hydrogen	H <sub>2</sub>	0	0
Cyclohexane	C <sub>6</sub> H <sub>12</sub>	-123.14	31.76

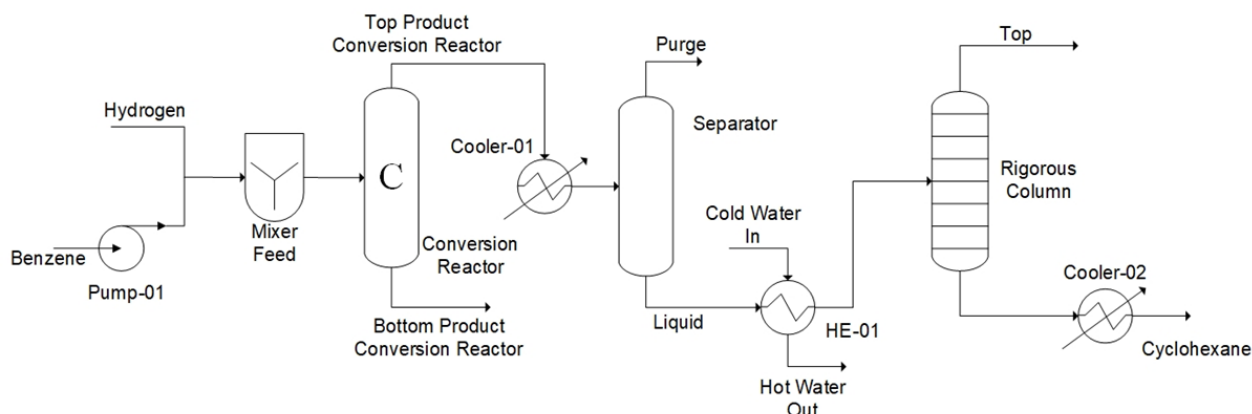


Figure 1. The basic (unmodified) process flow diagram of cyclohexane [20]

Gibbs energy ( $\Delta G_{298K}^{\circ}$ ):

$$\begin{aligned}\Delta G_{298K}^{\circ} &= \sum \Delta G_f^{\circ} \text{ product} - \sum \Delta G_f^{\circ} \text{ reactant} \\ &= (\Delta G_f^{\circ} \text{C}_6\text{H}_{12}) - (\Delta G_f^{\circ} \text{C}_6\text{H}_6 + \Delta G_f^{\circ} \text{H}_2) \\ &= 31.76 \text{ kJ/mol} - (129.66 \text{ kJ/mol}) \\ &= -97.9 \text{ kJ/mol} \\ \Delta G_{298K}^{\circ} &= -R T \ln K_{298} \\ \ln K_{298} &= \frac{\Delta G_{298K}^{\circ}}{-R T} \\ \ln K_{298} &= \frac{-97.9 \text{ kJ/mol}}{-(0.008314 \frac{\text{kJ}}{\text{mol} \cdot \text{K}})(298 \text{ K})} \\ \ln K_{298} &= 39.5145 \\ K_{298} &= 1.4485 \times 10^{17}\end{aligned}$$

At operating reactor temperature of 148.9 °C (421.9 K):

$$\begin{aligned}\ln \frac{K_T}{K_{298}} &= -\frac{\Delta H_{298K}^{\circ}}{R} \left( \frac{1}{T} - \frac{1}{T_{298}} \right) \\ \ln \frac{K_{421.9}}{1.4485 \times 10^{17}} &= -\frac{-206.07 \frac{\text{kJ}}{\text{mol}}}{8.314 \frac{\text{kJ}}{\text{mol} \cdot \text{K}}} \left( \frac{1}{421.9 \text{ K}} - \frac{1}{298 \text{ K}} \right) \\ \ln \frac{K_{421.9}}{1.4485 \times 10^{17}} &= -24.4259 \\ K_{421.9} &= 3.5719 \times 10^6\end{aligned}$$

Since the value of the equilibrium constant is relatively large, the reaction is irreversible, that is, to the right.

### 3.2 Process Modification

To maximize the cyclohexane yield, a process modification is carried out by adding recycle, TEE, valve, heat exchanger, separator, and rigorous column. The separator unit was added to split the top and bottom product that will proceed to the recycle and rigorous column to separate cyclohexane and benzene. This process modification can enhance cyclohexane purity by minimizing the use of raw materials. The recycling process has led to increase the purity of materials recycled which can reduce the reliance on raw materials [24]. Based on the implemented process modification, there has been an increase in the yield of cyclohexane produced from 93.49% to 99.90%. The modified process flow diagram of cyclohexane production is shown in Figure 3 and the Aspen HYSYS simulation (modified) is shown in Figure 4, while the mass and energy balance, compositions, and energy streams are presented in Table S1, S2, and S3 (Supporting Information), respectively.

Making cyclohexane using benzene at a temperature 37.8 °C and 335 psia, hydrogen, and nitrogen as raw materials, undergo in a mixer at a temperature 48.9 °C and 335 psia before entering the fixed-bed type reactor (conversion

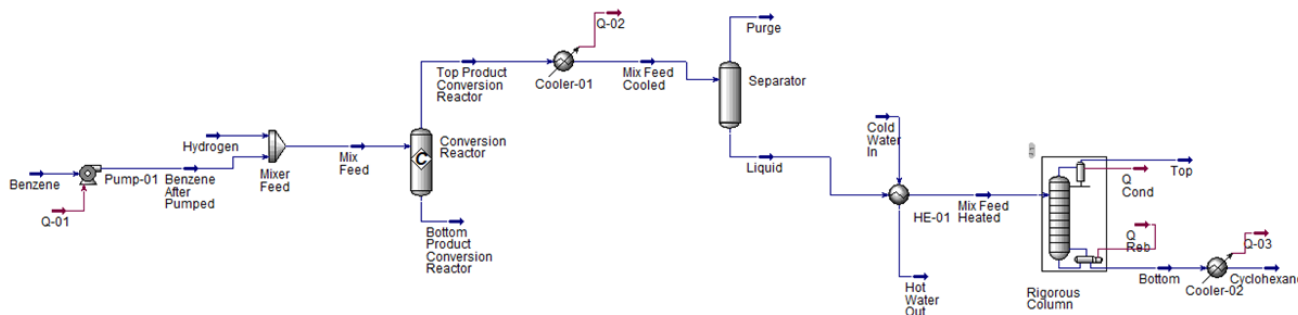


Figure 2. Aspen HYSYS simulation (unmodified / before modification)

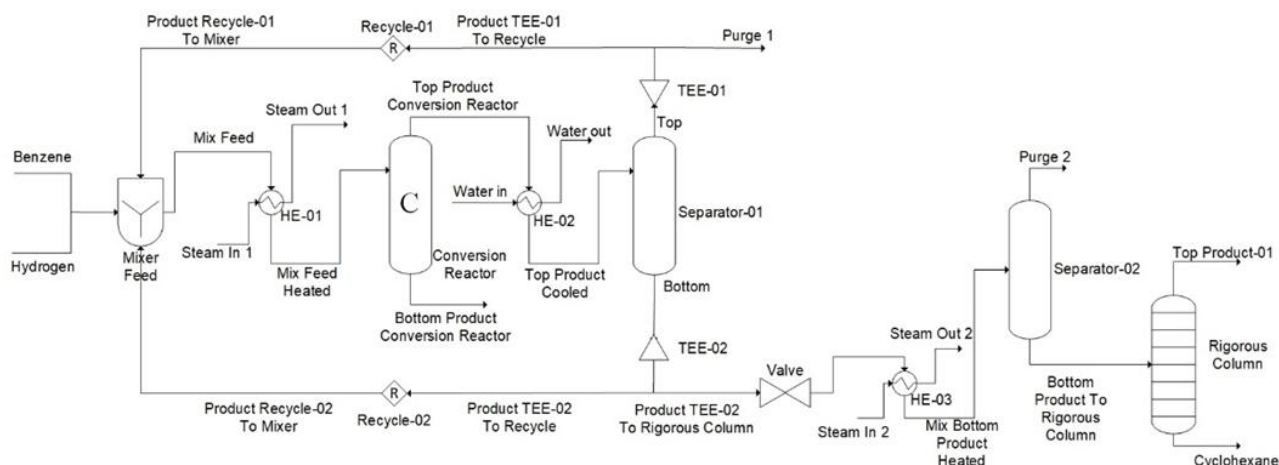


Figure 3. The modified process flow diagram of cyclohexane

reactor). Mixer is used to homogenize benzene and hydrogen (pretreatment), and ensuring a more homogeneous and evenly mixed compound upon entering the reactor.

In this modified process, to achieve high yield is needed tools, such as recycle, TEE, valve, heat exchanger, separator, and rigorous column. From separator, it can divided two stream of top and bottom product. The top product will be divided into two streams by the TEE. A purge stream is formed which will be released into the atmosphere and a product which will be recycled back to the mixer. By adding a separator, it can increase the yield of the product because it can remove dissolved gas to stabilize the liquid part of the fluid [25]. Stream recycle from TEE-01 with the specification 48.9 °C and 310 psia. The bottom product from Separator will be used to increase cyclohexane purity. The TEE-02 unit divides two streams, namely the recycle stream and the stream to be separated. Stream recycle from TEE-02 with the specification 48.9 °C and 310 psia will be recycled back to the mixer. The other stream from TEE-02 will be used to decreased pressure using valve from 310 psia 250 psia. By adding heat exchanger, to increase temperature from 48.9 °C to 200 °C because it can vaporizing. Separator used to increase the yield of the product. Outlet of liquid stream separator used to separated cyclohexane using Rigorous Column. Using Rigorous Column with the temperature of 200°C and pressure of 250 psia to separate methane and cyclohexane. The use of Rigorous Column is able to produce optimal results [26]. This is influenced by the equilibrium of the reaction and pressure on the column to achieve maximum results and efficient separation [27]. Cyclohexane as a product and methane as a side

product. Simulation of cyclohexane product is shown in Figure 4.

### 3.3 Improvement of Yield Product Due to the Process Modification

The results of the process modification show an improvement in the yield of cyclohexane. Based on the implemented process modification, yield of cyclohexane produced was 99.90%. To determine the yield percentage of two simulated processes, calculations were carried out using Equation (3). Table 2 compares the yield percentage in process without modification and with modification. The calculation of the percentage yield without modification process can be seen from the equation below with the data used from Aspen HYSYS V11.

$$\text{Yield of Cyclohexane (\%)} = \frac{0.9990}{1} \times 100\% = 99.90\% \quad (3)$$

With rigorous column will influenced by the equilibrium of the reaction and pressure on the column to achieve maximum results and efficient separation [27]. Cyclohexane as a product and methane as a side product. Simulation of cyclohexane product is shown in Figure 4.

Table 2. Comparison process without modification and with modification

Process	Cyclohexane Purity (%)
Without modification	93.49
With modification	99.90

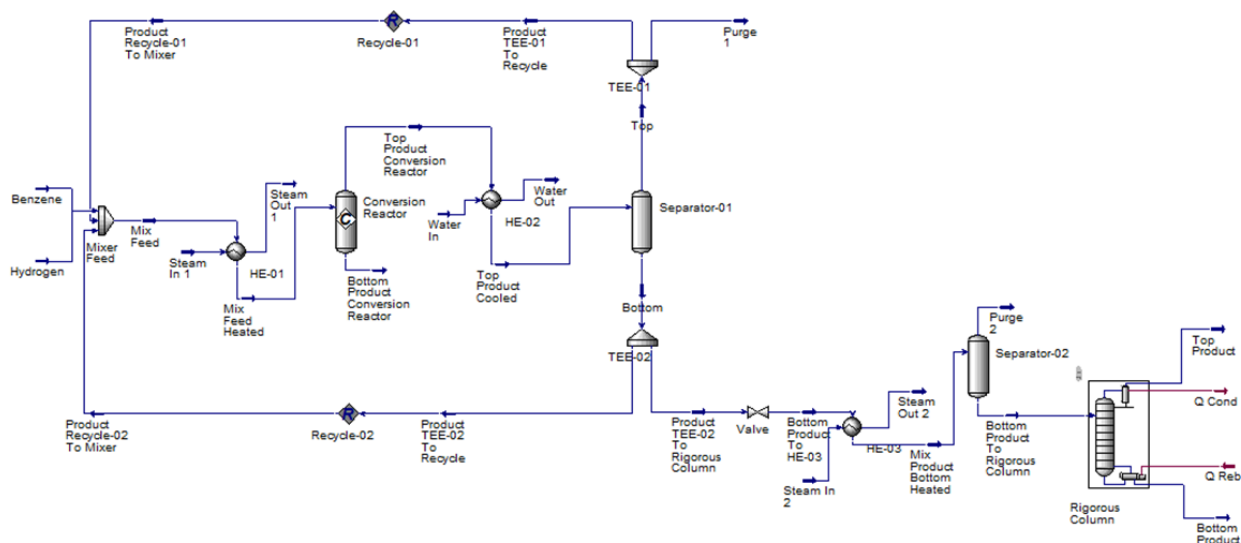


Figure 4. Aspen HYSYS simulation of the modified process of cyclohexane production.

#### 4. Conclusion

Process modifications in cyclohexane production must be undertaken to achieve higher production of yield. The modification process is carried out by adding recycle, TEE, and rigorous column. The cyclohexane yield in the modification process had an increase from 93.49% to 99.90%. By considering these aspects, the modification process was proven to be more efficient than the basic process and was in accordance with the research objectives. Thus, factories can optimize the use of raw materials, minimize by-products, and increase product yields. The suggestion that should be done is how engineers should find other modification methods to lower the energy usage for cyclohexane production without significantly reducing the quality of the improved process results and provide a simpler process than the current one.

#### CRedit Author Statement

Author Contributions: N.F. Faiq: Conceptualization, Methodology, Investigation, Software, Visualization, Writing, Review & Editing, Supervision; A.N. Ghifari: Conceptualization, Methodology, Visualization, Writing, Review & Editing, Project Administration, Validation; M.N. Failasofi: Conceptualization, Methodology, Formal Analysis, Resources, Validation, Writing; M.A. Wafi: Conceptualization, Methodology, Investigation, Resources, Data Curation, Writing; D.Hafizh : Conceptualization, Methodology, Investigation, Resources, Data Curation, Writing. All authors have read and agreed to the published version of the manuscript.

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