

Aniline Process Creation for Conversion Improvement Using Hydrogenation Process

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Abstract

The hydrogenation process of nitrobenzene to aniline is one of the main methods in the chemical industry to produce aniline with high efficiency. This research focuses on optimizing this process through system modification, which includes implementing a recycling flow and adjusting operating conditions such as temperature and pressure. The simulation results show an increase in the conversion of nitrobenzene to aniline by 1.44% after modification, from 96.82% to 98.26%. Although these improvements may seem small, their impact is significant on an industrial scale, especially in reducing raw material waste and energy consumption, making it a more sustainable solution. This study provides valuable insights for improving the efficiency of aniline processes in the context of the global chemical industry.

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Keywords: Nitrobenzene; Aniline production; Hydrogenation; Process modification effect

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1. Introduction

Aniline, a compound with the molecular formula $C_6H_5NH_2$, belong to the class of organic aromatic amines. It consists of a phenyl ring (C_6H_5) linked to an amino group ($-NH_2$) [1]. Aniline is widely used in the chemical industry as a raw material for making dyes, pharmaceutical ingredients, rubber, and polymers such as polyurethane. The hydrogenation of nitrobenzene (NB) stands out as a widely employed model reaction to produce aniline [2]. The proposed mechanism involves the formation of intermediate compounds in the form of nitrobenzene and phenylhydroxylamine. In addition, this mechanism also suggests the possibility of forming by-products such as azoxybenzene, azobenzene, and hydrazobenzene

through the reaction of these intermediate compounds. This process is not only efficient but has also become a model reaction in organic synthesis that is widely researched, considering the importance of aniline on both laboratory and industrial scales. As a compound with high chemical activity, aniline is also often used as a precursor in the synthesis of more complex compounds, making it a key component in many chemical reaction pathways.

Commonly used aniline production methods in industry are by nitrobenzene hydrogenation and phenol amination. However, more yields are obtained when using the nitrobenzene hydrogenation process, so it is used as a commercial route and supplying about 85% of the global aniline production. Aniline production can be carried out either in gaseous or in liquid phase. For the reactions in the vapor phase, fluidized bed and fixed bed reactors are usually used at a temperatures of 200-400 °C and a pressures of 1-

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10 bar with nitrobenzene conversion almost 99.9% [3-4]. Catalysts that were often used in the nitrobenzene hydrogenation process is in the form of metal, such as nickel (Ni) and copper (Cu). The aniline in the isolated species, while having stability in the inverted orthonated form, has a closer energy balance. A commercial process of highly exothermic catalytic hydrogenation of nitrobenzene ($\Delta H^{\circ}_{200\text{ }^{\circ}\text{C}} = -544 \text{ kJ/mol}$) in the vapor and liquid phases was carried out [5-6].

The nitrobenzene hydrogenation process has undergone various developments to increase efficiency and selectivity [7]. One approach that is often applied is adjusting operating parameters such as temperature, pressure, and catalyst type. In addition, research on the integration of recycling processes has shown that this method can significantly reduce raw material waste and increase conversion efficiency [8]. Therefore, further development of this process should not only focus on increasing yields, but also on reducing environmental impacts and production costs. In this research, a simulation was carried out to evaluate the effect of system modification on the conversion of nitrobenzene to aniline by considering aspects of efficiency and sustainability.

2. Methods

2.1 Hydrogenation Process of Nitrobenzene to Aniline

The nitrobenzene hydrogenation process is one of the main methods to produce aniline, which is widely used in the chemical industry [9]. In this process, nitrobenzene ($\text{C}_6\text{H}_5\text{NO}_2$) is reduced to aniline ($\text{C}_6\text{H}_5\text{NH}_2$) using hydrogen gas (H_2) and a catalyst, such as nickel, palladium, or platinum. This reaction is usually carried out under high pressure and well-controlled temperatures to maximize yield and reduce by-product formation. This hydrogenation is the main choice due to its high efficiency, with conversion rates of nitrobenzene to aniline often exceeding 99% [10]. The reactor with weighed portions of the catalyst, nitrobenzene, and methanol was purged three times with nitrogen, then heated to the required temperature in a nitrogen atmosphere. Then the reactor was purged with hydrogen, constant reaction pressure was maintained by a pressure reducer [9].

The hydrogenation process of nitrobenzene to aniline is a reaction that involves a transition metal catalyst to speed up the reaction. Catalysts based on noble metals such as Rhodium (Rh) in alkaline media provide high efficiency with selectivity reaching 98% [11]. Another alternative is a cheaper cobalt-based catalyst, as described which demonstrated long-term stability under harsh operating conditions [12]. Additionally,

Low-temperature hydrogenation with a nano-Pd catalyst allows a significant reduction in the formation of by-products, making the process more environmentally friendly [13].

The reaction mechanism involves several steps, including the formation of intermediate compounds such as nitrosobenzene ($\text{C}_6\text{H}_5\text{NO}$) and phenylhydroxylamine ($\text{C}_6\text{H}_5\text{NHOH}$). These two compounds are then further reduced to aniline as the final product. However, under certain conditions, by-products such as azobenzene, azoxybenzene, and hydrazobenzene can also be formed due to reactions between these intermediate compounds [2]. Controlling parameters such as temperature and pressure is very important to direct the reaction towards the main product, namely aniline, and minimize the formation of by-products. The chemical reaction [15] is (Figure 1):



2.2 Methods to Conversion Improvement of Aniline Production

One method used to increase conversion in aniline production is by adding a recycling flow to the nitrobenzene hydrogenation process. In this system, part of the reactor output product containing unreacted nitrobenzene will be separated and returned to the reactor for further reactions [16]. This approach ensures that almost all of the nitrobenzene can be converted to aniline, thereby minimizing waste of raw materials and increasing the overall efficiency of the process. This recycling process also allows for more flexible adjustments to reactor operating conditions, such as setting the hydrogen to nitrobenzene ratio, which has a direct impact on selectivity and final aniline yield.

In addition to adding a recycling flow, increasing the reactor temperature is also an effective method for maximizing conversion. An increase in temperature accelerates the rate of the hydrogenation reaction, allowing nitrobenzene to react more quickly with hydrogen gas [17]. The process of aniline production from nitrobenzene hydrogenation before modification is reacting nitrobenzene and hydrogen in reactor with a temperature of 240 °C and a pressure of 2.3 atm to produce mixed aniline. The results of

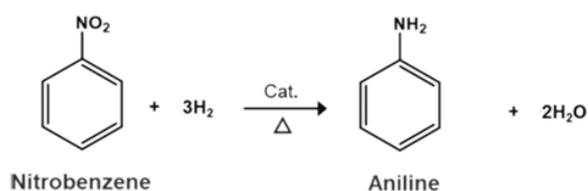


Figure 1. Hydrogenation of nitrobenzene [14]

hydrogenation will be cooled with a cooler until the temperature reaches 40 °C with a pressure of 1.96 atm. Mixed aniline will be separated according to the phase in the separator with temperature and pressure but so that hydrogen will form with the vapor phase which will be disposed of as an excess compound and aniline + water mixture in the vapor phase. The mixture of aniline and water will be heated with a heater until the temperature reaches 130 °C with a pressure of 1.62 atm and separated by a separator and produces the main product in the form of aniline and a by-product in the form of water vapor.

The methods of maximizing and optimizing product yield, product selectivity, and/or reactant conversion against independent variables and operating conditions (reactor pressure, reactor temperature, and reactor mole ratio) [18]. In general, the process of aniline production from nitrobenzene hydrogenation is the reaction of nitrobenzene and hydrogen (high temperature) and then the reaction results will be separated according to the type of material and phase of the mixture by heating or by separation. Controlling the partial pressure of hydrogen during the reaction can minimize unwanted side products [19]. Involves modifying the reactor geometry to increase mass transfer, thereby speeding up the reaction rate without affecting the quality of the primary product [20].

3. Result and Discussion

3.1 Process Flow Diagram of Hydrogenation Nitrobenzene to Aniline

Figure 2 and Figure 3 illustrate the aniline production process. The process is carried out by mixing nitrobenzene and hydrogen. Where hydrogen is obtained from outside air. The conditions of nitrobenzene and hydrogen need to be adjusted to operating conditions before entering the reactor, where the conversion reactor conditions operate at a temperature of 240°C and a pressure of 2.3 atm. After entering the reactor, two phases will be formed, aniline vapor and aniline liquid phases. Aniline liquid comes out as condensate and the aniline vapor is reprocessed to obtain the main product. The aniline vapor then enters the cooler to be cooled until the temperature drops to 10 °C before entering the distillation column. The distillation column operates at a pressure condition of around 2 atm. Then it produces hydrogen and condensate, a mixture of aniline and steam. The mixture of aniline and steam is heated with a heater to reach a temperature of 170 °C before entering the second distillation column. In this distillation column, the main product will be formed in the form of aniline and the side product will be in the form of water vapor. From this process, aniline production results were obtained with a conversion mole fraction of 97%.

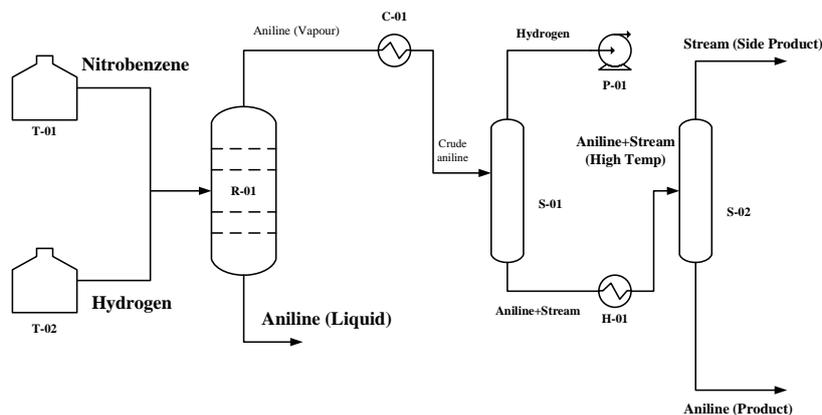


Figure 2. Unmodified process flow diagram of aniline production

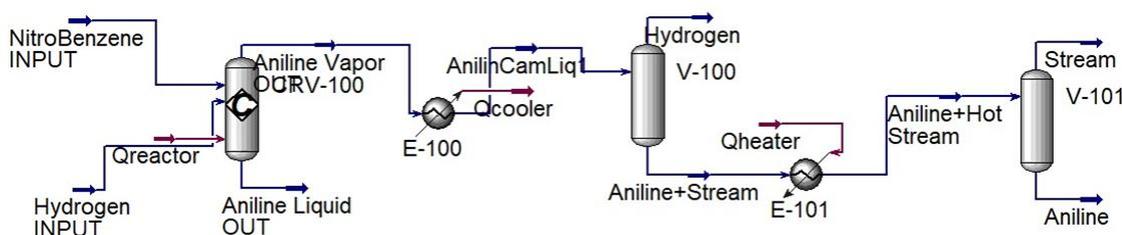


Figure 3. Unmodified process simulation using Aspen HYSYS

Figure 4 and Figure 5 illustrate the production process of modified aniline. This process adds a recycling process. The process is carried out by mixing nitrobenzene and hydrogen. Where hydrogen is obtained from outside air. The conditions of nitrobenzene and hydrogen need to be adjusted to operating conditions before entering the reactor, where the conversion reactor conditions operate at a temperature of 215 °C and a pressure of 1 atm. After entering the reactor, two phases will form, namely the aniline vapor phase and the aniline liquid phase. The aniline liquid comes out as condensate and the aniline vapor is reprocessed to obtain the main product. The aniline vapor then enters the cooler to be cooled until the temperature drops to 10 °C before entering the distillation column. The distillation column operates at a pressure condition of around 0.7 atm. Then it produces hydrogen and condensate, in the form of a mixture of aniline and steam. Because there is a recycling process cycle, the hydrogen produced is recycled by adjusting the operating conditions to suit the operating conditions of the first distillation column, namely hydrogen pressure conditions of 0.7 atm. This

recycling cycle will increase the amount of mixture between aniline and steam formed. The mixture of aniline and steam is heated with a heater until it reaches a temperature of 130°C before entering the second distillation column. In this distillation column, the main product will be formed in the form of aniline and the side product will be in the form of water vapor. From this process, aniline production results were obtained with a mole fraction conversion of 98%. This result improves better than the unmodified process.

3.2 Mass Balance and Energy Balance

The results of the mass balance and energy balances of the aniline production of unmodified process through the hydrogenation process are presented in Tables S1 and S2 (Supporting Information).

3.2 Thermodynamics Consideration

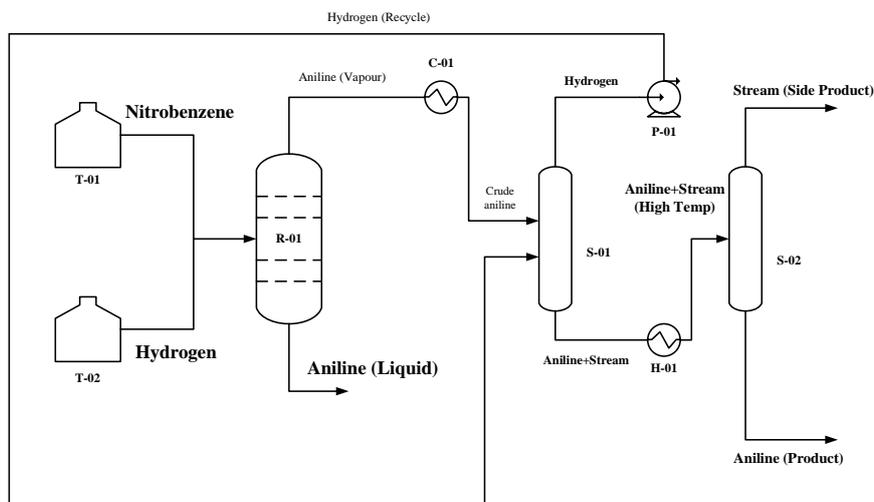
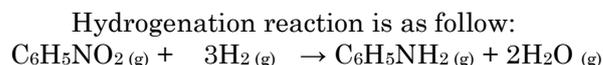


Figure 4. Process flow diagram of modified process of aniline production

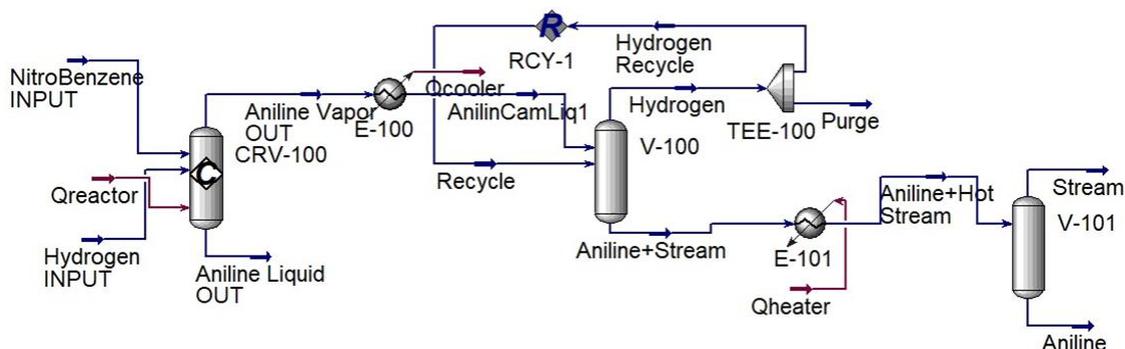


Figure 5. Aspen HYSYS simulation of modified process of aniline production

For the determination of the nature of the reaction (exothermic/endothermic) and the direction of the reaction (reversible/irreversible), it is necessary to calculate the standard heat of formation (ΔH_f°) at 1 bar and 298 K of the reactants and products. The value of ΔH_f° and ΔG_f° can be seen in Table 1 [21].

Standard heat of reaction (ΔH_R°) calculation:

$$\Delta H_R^\circ = \sum \Delta H_f^\circ \text{ product} - \sum \Delta H_f^\circ \text{ reactant}$$

$$= (\Delta H_f^\circ \text{ C}_6\text{H}_5\text{NH}_2 + \Delta H_f^\circ \text{ H}_2\text{O}) - (\Delta H_f^\circ \text{ C}_6\text{H}_5\text{NO}_2 + \Delta H_f^\circ \text{ H}_2)$$

$$= (86.86 + (-241.80)) - (67.60 + 0) = -222.55 \text{ kJ/mol}$$

Based on the calculations that have been made, we get the value $\Delta H_R^\circ = -222.55 \text{ kJ/mol}$ (negative value) so the reaction is exothermic.

Gibbs energy (ΔG_f°) calculation:

$$\Delta G_f^\circ = \sum \Delta G_f^\circ \text{ product} - \sum \Delta G_f^\circ \text{ reactant}$$

$$= (\Delta G_f^\circ \text{ C}_6\text{H}_5\text{NH}_2 + \Delta G_f^\circ \text{ H}_2\text{O}) - (\Delta G_f^\circ \text{ C}_6\text{H}_5\text{NO}_2 + \Delta G_f^\circ \text{ H}_2)$$

$$= (166.69 + (-228.60)) - (158.00 + 0) = -219.91 \text{ kJ/mol}$$

Equilibrium constant (K_2) in the standard state

$$\Delta G_f^\circ = -RT \ln K$$

$$\ln K_2 = -\frac{\Delta G_f^\circ}{RT} = \frac{219910 \text{ J/mol}}{8,314 \text{ J/mol} \times 298 \text{ K}}$$

$$\ln K_2 = 88.760$$

$$K_2 = 4.48594$$

Equilibrium constant (K_1) at $T = 240 \text{ }^\circ\text{C}$

$$\ln \frac{K_1}{K_2} = \frac{-\Delta H_R^\circ}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)$$

Where, K_1 is equilibrium constant at $25 \text{ }^\circ\text{C}$, K_2 is equilibrium constant at operating temperature, T_1 is standard temperature ($25 \text{ }^\circ\text{C}$), T_2 is operating temperature ($240 \text{ }^\circ\text{C}$), R is ideal gas constant, ΔH_R° is standard heat of reaction at $25 \text{ }^\circ\text{C}$.

$$\ln \frac{K_1}{4.48594} = \frac{222550 \text{ J/mol}}{8.314 \text{ J/mol} \cdot \text{K}} \left(\frac{1}{513 \text{ K}} - \frac{1}{298 \text{ K}} \right)$$

$$\ln \frac{K_1}{4.48594} = -37.646$$

$$4.4725 \times 10^{-17} = \frac{K_1}{4.48594}$$

$$K_1 = 2.00633 \times 10^{-16}$$

Table 1. The value of ΔH_f° and ΔG_f° of compounds [21].

Compounds	Molecular Formula	ΔH_f° (kJ/mol)	ΔG_f° (kJ/mol)
Nitrobenzene	$\text{C}_6\text{H}_5\text{NO}_2$	67.60	158.00
Hydrogen	H_2	0	0
Aniline	$\text{C}_6\text{H}_5\text{NH}_2$	86.86	166.69
Water	H_2O	-241.80	-228.60

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

The process of hydrogenation of nitrobenzene to aniline is exothermic, with energy released during the reaction. The optimally controlling the reaction temperature can increase conversion efficiency without triggering the formation of side products [22]. The important role of enthalpy and entropy in reaction stability, especially at low pressure [23]. DFT (Density Functional Theory) based thermodynamic simulations can predict ideal conditions for producing aniline with a selectivity level of up to 99%, making it easier to optimize on an industrial scale [24].

3.3 Process Modification for Increasing Conversion of Nitrobenzene.

Increaseing conversion in the nitrobenzene hydrogenation process is carried out using a series of tools such as reactor, separators, cooler and heater to achieve high conversion (Table 2). First, the reactant input will enter the CRV-100 (Reactor) to react and form aniline, then aniline gas flows. After that, it will be burned with E-100 (Cooler) and flowed for discount into top product and bottom product through 2 Separators: V-100 and V-101, with interheater: E-101. The hydrogen output from the V-100 Separator is recycled and put back into the V-100. In the non-modified design, the nitrobenzene conversion reached 97%, while after process modification the nitrobenzene conversion reached 98%.

An increase in temperature accelerates the rate of the hydrogenation reaction, allowing nitrobenzene to react more quickly with hydrogen gas [17]. The process of aniline production from nitrobenzene hydrogenation before modification is reacting nitrobenzene and hydrogen in reactor with a temperature of $240 \text{ }^\circ\text{C}$ and a pressure of 2.3 atm to produce mixed aniline. The process of aniline production from nitrobenzene hydrogenation after modification is reacting nitrobenzene and hydrogen in reactor with a temperature of $215 \text{ }^\circ\text{C}$ and a pressure of 1 atm to produce mixed aniline.

Table 2. Conversion of nitrobenzene to aniline before and after process modification

Process Type	Nitrobenzene conversion
Before Modification	97%
After Modification	98%

Modification of the nitrobenzene hydrogenation process involves innovation in the reactor and the use of new technology to increase efficiency. The use of a catalyst-coated reactor can increase the contact area between the catalyst and the raw material, thereby increasing conversion [25]. The efficiency of product separation using a hybrid membrane system that better separates aniline from water [26]. The use of graphene-based catalysts with transition metal doping can minimize the formation of by-products, providing economic benefits on an industrial scale [27]. Increasing energy efficiency in heating and cooling systems in the process flow can reduce energy consumption by up to 10% [28]. Emphasized the importance of controlling the pressure of recycled hydrogen to maintain reaction stability and increase selectivity up to 98% [29].

4. Conclusion

Benzene hydrogenation is generally carried out using a reaction system in a reactor and separated using a separator. After modifying the nitrobenzene hydrogenation process system in the manufacture of aniline by providing a recycling process and changing operating conditions, changes in operating conditions can affect the product conversion results. Fluid circulation, reduced heat and pressure are known to increase the aniline conversion yield. This is proven by simulations after modification with reduced heat and pressure, as well as the addition of a recycling process. The general process simulation results produce an aniline conversion of 97%, while the conversion results from the simulation results after modification can reach 98%. From the comparison of these two results, conversion after reducing temperature and pressure, as well as adding a recycling process, can increase conversion by 1%. This increase certainly has a quite good impact on the scale of industrial production. Although these modifications are not significant, they can improve overall process efficiency and provide economic benefits by reducing raw material waste and energy consumption, making it a more sustainable solution in aniline production.

CRedit Author Statement

Author Contributions: A. N. Rohmah: Conceptualization, Methodology, Resource, Data Curation, Writing, Software, Formal Analysis, Review and Editing; A. R. Palupi: Conceptualization, Methodology, Resource, Writing, Supervision, Writing Draft, Review and Editing, Projects Administration; A. N. H. M. Manulang: Writing Draft, Review and Editing; H. D. B. Erawati: Writing Draft, Review and Editing,

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

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