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Research Article

Process Optimization of Chlorobenzene Production through the Integration of a Distillation Unit and Mixer in Gas–Liquid Benzene Chlorination

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Abstract

Chlorobenzene is a key intermediate in the synthesis of phenol, aniline, and dichlorodiphenyltrichloroethane (DDT), and is also utilized as a solvent, heat-transfer fluid, and occasionally in dry-cleaning. Given its wide industrial applications, production efficiency is essential for economic viability. This study explores yield enhancement in chlorobenzene manufacture through modifications to the benzene chlorination process. The intensification strategy involved the addition of a distillation column and a mixer unit, while sensitivity analyses were performed using chemical engineering simulation software to assess the influence of operating parameters. The modified process increased chlorobenzene yield from 83% to 98%. Sensitivity analysis revealed that higher benzene feed pressure negatively impacted liquid-phase product yield, whereas a greater benzene-to-chlorine mass flow ratio improved yield by enhancing selectivity toward chlorobenzene formation. These findings demonstrate that process modifications combined with optimized operating conditions can significantly improve chlorobenzene production efficiency.

Keywords: Chlorobenzene; Benzene Chlorination; Yield Optimization; Process integration

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1. Introduction

Monochlorobenzene (C_6H_5Cl), commonly known as chlorobenzene, is a simple aromatic compound that appears as a colorless, highly flammable liquid with a characteristic almond-like odor. Since its first synthesis in the mid-19th century, it has gained significant industrial importance due to its solvent properties and versatile chemical reactivity [1]. Industrially, chlorobenzene is produced through the chlorination of benzene in the presence of Lewis acid catalysts and may also arise as a terminal product in the reductive chlorination of di- and trichlorobenzenes. At present, it serves as a key intermediate in the synthesis of industrial and pharmaceutical compounds, and is widely employed in the manufacture of phenol, aniline, and DDT; as a solvent in paint formulations; and as a heat transfer medium. It is occasionally used in the dry-cleaning industry [2].

Several process optimization studies have applied advanced simulation and integration techniques to improve chlorobenzene production. Aspen Plus modeling has optimized separation parameters such as tray number, feed tray position, and reflux ratio, thereby enhancing benzene–chlorobenzene recovery and overall economic performance [3]. Computational studies using generalized disjunctive programming (GDP) models have further demonstrated that adjusting column configurations-specifically 20 trays with a reflux ratio of 1.05 in the first column and 14 trays with a reflux ratio of 0.3 in the second-can increase conversion from 50% to 69% (4). Heat integration strategies in distillation systems have reduced utility demands by up to 57% [1], while energy

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frameworks integrating heat and power within chlorination processes have lowered operating costs by 35.4% [5]. Collectively, these findings highlight the critical role of column configuration, thermal integration, and operating conditions in determining conversion efficiency, energy consumption, and cost-effectiveness.

Nevertheless, existing literature rarely addresses process modifications that emphasize recycling overhead streams from chlorination and purification units back into the reactor. A promising approach involves adding a distillation unit to remove HCl and a mixer unit to recombine the benzene-rich bottom stream with the purified overhead stream prior to recycling. This configuration ensures a consistent benzene supply to the reactor, reduces fresh feed requirements, and lowers overall energy consumption by improving HCl separation and recycle efficiency. The design is particularly advantageous for gas–liquid phase chlorination at low temperatures, which consumes less energy than gas-gas chlorination and thus provides a more economical route for monochlorobenzene production. Beyond process considerations, sustainability is closely linked to health and environmental concerns. Chlorobenzene exposure has been associated with toxic effects on internal organs and environmental persistence; therefore, plant design must incorporate emission control, HCl recovery, and remediation strategies such as biodegradation. Studies indicate that certain microorganisms can metabolize chlorobenzene in contaminated soil and water, although degradation rates are strongly influenced by aeration and redox conditions [6]. In addition, controlling volatile organic compounds (VOCs) from chlorobenzene emissions has become a focal point. Experimental work has shown that ionic liquids can efficiently capture chlorobenzene and dichlorobenzene from exhaust gases, reducing concentrations to the ppm level [7].

In the proposed process modification, pressure and mass-flow variables are systematically varied to assess their impact on reactor conversion, distillation efficiency in HCl removal, and the overall yield of monochlorobenzene. The primary objective is to maximize product output through optimized recycle streams and operating parameters, thereby minimizing raw material requirements and reducing energy consumption.

2. Methods

2.1. Monochlorobenzene Production

The most employed method to produce monochlorobenzene is chlorination, wherein a hydrogen atom in a paraffinic or aromatic compound is substituted with a chlorine atom [8]. In the case of chlorobenzene synthesis, this process involves the chlorination of benzene in the presence of a suitable catalyst [9]. Industrially, the process is predominantly conducted via continuous liquid-phase chlorination. Among the available techniques, gas–liquid phase benzene chlorination is favored due to its operation at relatively low temperatures and reduced energy requirements, rendering it more economically viable than the gas–gas phase chlorination alternative. Chlorination of benzene occurred with the reaction of benzene (C_6H_6) with chlorine (Cl_2) to produce chlorobenzene ($C_6H_5Cl_2$) and side product such as dichlorobenzene ($C_6H_4Cl_2$) and HCl. The chemical reactions are represented as follow:

$$C_6H_6\,_{(l)} + Cl_2\,_{(g)} \rightarrow C_6H_5Cl_2\,_{(l)} + HCl\,_{(g)} \quad \Delta H_{r\,298\,K} = -123.39\ \text{kJ/mol} \tag{1}$$

$$C_6H_5Cl_2\,_{(l)} + Cl_2\,_{(g)} \rightarrow C_6H_4Cl_2\,_{(l)} + HCl_{(g)} \quad \Delta H_{r\,298\,K} = -122.13\ \text{kJ/mol} \tag{2}$$

Commonly the production of monochlorobenzene in industries, liquid-phase benzene is reacted with chlorine gas within a reactor system. Chlorine gas is introduced at the base of the reactor through a perforated plate, ensuring effective dispersion and contact with the liquid phase. The reaction is catalyzed by solid ferric chloride ($FeCl_3$), which serves to enhance the reaction rate and suppress undesirable side reactions [10]. The chlorination reaction is conducted in a fixed bed multitube reactor operating under non-isothermal conditions. The reactor is maintained within a temperature range of 20–40 °C, which is critical for minimizing the formation of dichlorobenzene, a by-product that becomes more prevalent at elevated temperatures [11]. This temperature window is considered optimal for the large-scale synthesis of monochlorobenzene. The reactor operates at a pressure of 2.3686 atm (2.4 bar). The feed stream, comprising liquid benzene and chlorine gas, enters the system at 25 °C and atmospheric pressure. The liquid benzene is directed through the reactor tubes packed with $FeCl_3$ catalyst, while chlorine gas is concurrently introduced into the reactor to initiate the chlorination process.

The synthesis of monochlorobenzene proceeds via an exothermic reaction [12]. An exothermic reaction is characterized by the release of heat from the system to the surrounding environment [13]. Consequently, to maintain the reaction within the desired temperature range, an appropriate cooling system is employed. If the chlorination reaction temperature rises excessively, there is a significant risk of an accelerated combustion rate of benzene, potentially resulting in an uncontrolled and hazardous reaction due to the substantial release of hydrogen chloride (HCl) gas into the atmosphere. Therefore, effective cooling is essential to ensure that the reaction temperature remains below 40 °C [14]. In the simulation of the chlorobenzene production process using the gas–liquid phase benzene chlorination method, the system is designed to achieve an annual production capacity of 30,000 metric tons.

2.2. Thermodynamic Review

A thermodynamic review is used to determine the characteristics of the reaction, described by the values of ΔH_r, ΔG_r, and K [15]. It is used to determine whether the reaction is exothermic or endothermic and reversible or irreversible. The value of $\Delta H^{\circ}_{r\,298K}$ for the main reaction is obtained by Equations (1) and (2).

$$H_{\text{total reaction}} = \Delta H_{\text{reaction 1}} + \Delta H_{\text{reaction 2}} = \text{-}244.520 \text{ kJ/mol} \qquad (3)$$

Based on the calculation, obtained $\Delta H^{\circ}_{reaction}$ = -244.529 kJ/mol with negative value that indicates the reaction is exothermic that releases heat. Data $\Delta G^{\circ}_{f\,298K}$ of each component to determine ΔG_r at 298K can be seen in Table 1. The value of $\Delta G^{\circ}_{r\,298K}$ for the main reaction is obtained by:

$$\Delta G^{\circ}_{reaction\,1} = \Delta G^{\circ}_{f\,product} - \Delta G^{\circ}_{f reactant} = (G^{\circ}_{f\,HCl} + G^{\circ}_{f\,298K\,C6H5Cl}) - (G^{\circ}_{f\,HCl} + G^{\circ}_{f\,C6H6}) = -125800\,J/mol$$

The value of equilibrium constant (K) at 298 K can be calculated by:

$$\ln K_{298} = \frac{-\Delta G^{\circ}_{r\,298}}{RT} \qquad (4)$$

$$\ln K_{298} = \frac{-(-125800\frac{J}{mol})}{-8.314\frac{J}{mol.K}\times 298\,K}$$

$$K_{298} = 1.126 \times 10^{22}$$

For reaction temperature 313 K (40 °C), the equilibrium constant (K) can be calculated by:

$$\ln\frac{K_{\mathrm{T}}}{K_{298}} = \frac{\Delta H^{\circ}_{f\,298K}}{R} \times \left(\frac{1}{T} - \frac{1}{T_{298}}\right) \qquad (5)$$

Table 1. Gibbs free energy formation of compounds.

Compounds	Molecular Formula	$\Delta G_{f\,298\,K}$ (kJ/mol)
Benzene	C_6H_6	129.66
Chlorine	Cl_2	0
Chlorobenzene	C_6H_5Cl	99.16
Hydrochloric Acid	HCl	-95.30
1,4-Dichlorobenzene	$C_6H_4Cl_2$	77.15

$$\ln\frac{K_{313}}{1.126\times 10^{22}} = \frac{-123390\frac{J}{mol}}{8.314\frac{J}{mol.K}} \times \left(\frac{1}{313\,K} - \frac{1}{298\,K}\right)$$

$$K_{313} = 1.035 \times 10^{21}$$

Based on the calculation, the equilibrium constant value of the main reaction operates at 40 °C (K_{313}) = 1.035×10^{21}, which is higher than 1 so the reaction is irreversible. Calculation of ΔG°_f Reaction (2) as a side reaction:

$$\Delta G^{\circ}_{reaction\,2} = \sum \Delta H^{\circ}_{f\,product} \sum \Delta G^{\circ}_{f reactant} = (G^{\circ}_{f\,HCl} + G^{\circ}_{f\,298K\,C6H5Cl}) - (G^{\circ}_{f\,HCl} + G^{\circ}_{f\,C6H6})$$

$$\Delta G^{\circ}_{f\,298K} = \text{-}\,R\,T\,\text{In}\,K_{298}$$

$$\ln K_{298} = \frac{-\Delta G^{\circ}_{f\,298}}{RT}$$

$$\ln K_{298} = \frac{-117310\frac{J}{mol}}{-8.314\frac{J}{mol.K}\times 298\,K} = 47.349$$

$$K_{298} = 3.659 \times 10^{20}$$

At operating temperature 313 K (40 °C):

$$\text{In}\frac{K_{\mathrm{T}}}{K_{298}} = -\frac{\Delta H_{f\,298}}{R}\left(\frac{1}{T} - \frac{1}{T_{298}}\right)$$

$$\text{In}\frac{K_{313}}{3.659\times 10^{20}} = -\frac{-121130\frac{J}{mol}}{8.314\frac{J}{mol.K}}\left(\frac{1}{313\,K} - \frac{1}{298\,K}\right)$$

$$K_{313} = 3.514 \times 10^{19}$$

From the calculation, the value of $K_{313} > 1$, thus, the side reaction is irreversible.

Accordingly, production of monochlorobenzene is preferably conducted via gas–liquid phase benzene chlorination. In this process, benzene (C_6H_6) reacts with chlorine (Cl_2) to form chlorobenzene (C_6H_5Cl), accompanied by by-products such as dichlorobenzene ($C_6H_4Cl_2$) and hydrogen chloride (HCl) [16]. Process flow diagram illustrating benzene chlorination without any modifications is presented in Figure 1.

2.3. Optimization Method

To maximize the yield of chlorobenzene, it is necessary to make modifications in the process. A process modification was implemented by introducing a recycle stream from the top product of the chlorination reactor, redirecting it back into the same reactor. This recycling strategy was realized through the addition of a distillation unit and a mixer unit. The distillation unit was incorporated to function as an HCl separator, receiving a feed primarily composed of hydrogen chloride from the chlorination reactor's output. Concurrently, the mixer unit was installed to merge the bottom stream from the distillation unit (serving as the HCl remover) with the top stream from the purification unit, thereby forming a closed-loop recycle system. The resulting recycled stream, enriched with unreacted benzene (C_6H_6), is subsequently fed back into the chlorination reactor. This process modification contributes to improved chlorobenzene production by reducing the consumption of raw materials and lowering energy requirements.

The implementation of the process modification demonstrates an increase in chlorobenzene yield by redirecting the output stream from the chlorination reactor back into the same reactor for recycling. Chlorobenzene yield can be calculated using Equation (1):

$$\text{Yield of chlorobenzene } (\%) = (\text{mol of chlorobenzene}) / (\text{mol of benzene}) \times 100\% \qquad (6)$$

3. Results and Discussion

3.1. Adjusted Process Flowsheet and Simulation

To increase chlorobenzene yield, the process is improved by introducing a recycle stream, from the top product of the chlorination reactor back into the reactor. This recycling step is enabled by adding a distillation unit and a mixer. The distillation column functions primarily to remove HCl, as its feed mainly consists of HCl from the chlorination reactor outlet. The mixer is then used to blend the bottom product of the distillation unit (HCl remover) with the top product from the purification unit, forming the recycle loop. The recycled stream, which contains a high concentration of unreacted benzene (C_6H_6), is subsequently directed back to the chlorination reactor. This modification helps increase chlorobenzene production while reducing raw material consumption and energy use. The simulation of the modified benzene chlorination process is illustrated in Figure 2 by adding highlighted units in blue color. The revised process flow diagram (PFD), incorporating one additional distillation unit and one mixer, is presented in Figure 3.

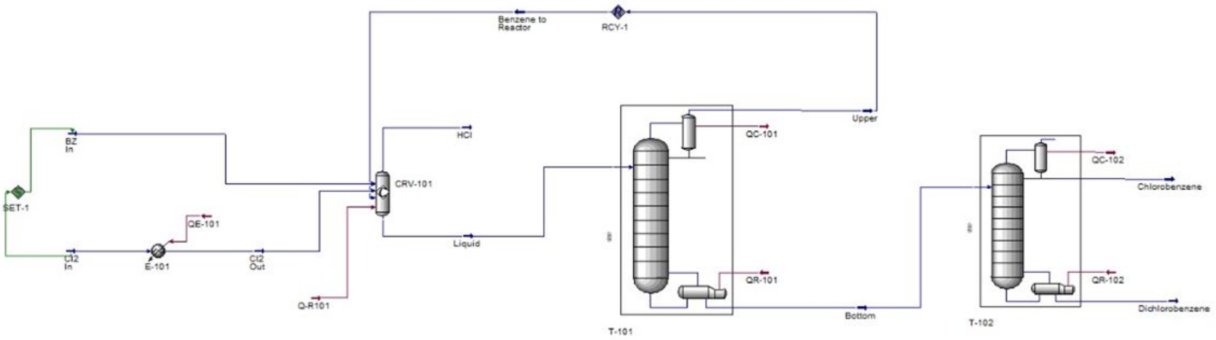

Figure 1. Basic flow diagram of benzene chlorination.

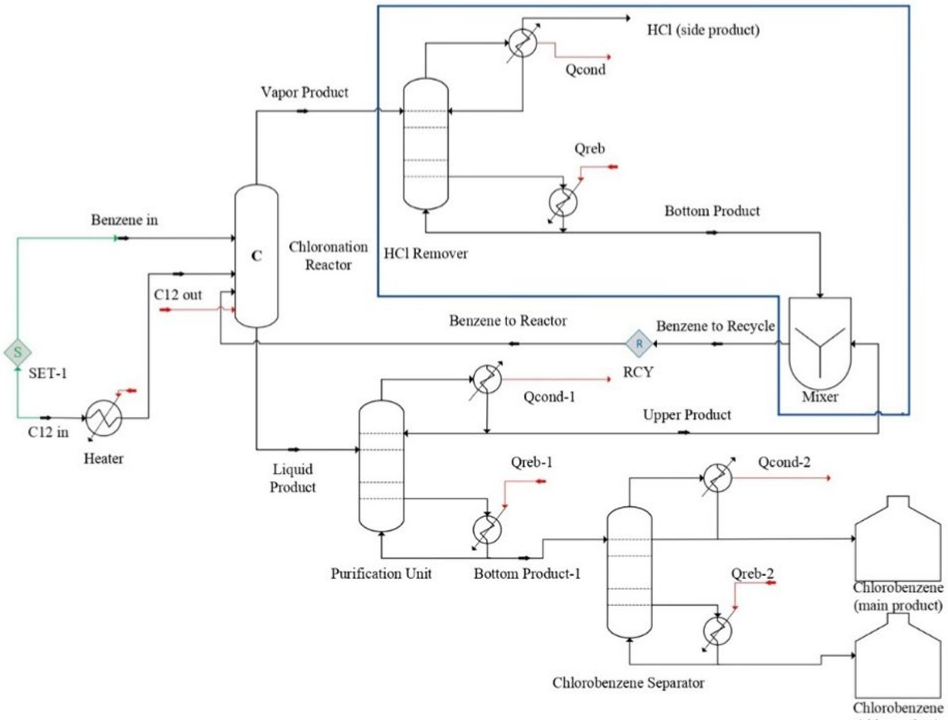

Figure 2. Process flow diagram (PFD) of modified process of benzene chlorination.

3.2. Enhanced Product Yield through Process Modification with an Added Distillation Unit

The enhancement of chlorobenzene yield is primarily achieved through the addition of a distillation unit to the process flowsheet. The newly incorporated distillation column serves as an HCl removal unit, receiving a stream that consists largely of HCl from the chlorination reactor outlet. By effectively separating HCl, the distillation unit enables cleaner downstream processing and allows a greater portion of unreacted benzene to be recovered. This recovered benzene, obtained from the bottom product of the distillation unit, is then directed to a mixer where it is combined with the overhead stream from the purification unit, forming a recycle loop. The recycle stream, rich in unreacted benzene (C_6H_6), is subsequently returned to the chlorination reactor, increasing the overall conversion of benzene to chlorobenzene. This modification reduces fresh benzene requirements and lowers energy consumption, while significantly improving chlorobenzene yield. Based on Equation (1), the chlorobenzene yield was determined. Table 2 presents a comparison of the chlorobenzene yield between the unmodified and modified process configurations.

Table 2. Comparison process without modification and with modification.

Process	Chlorobenzene Yield (%)
Without modification	83%
With modification	98%

The observed enhancement in chlorobenzene yield from 83% to 98% is primarily attributable to the incorporation of a recycle stream that facilitates the recovery and reintegration of unreacted benzene into the chlorination reactor. In the original process configuration, a significant fraction of benzene remained unconverted and was subsequently lost in downstream processing, thereby constraining the overall conversion efficiency. The implementation of a distillation unit to selectively remove hydrogen chloride (HCl) enables the isolation of a benzene-enriched stream, which is then recycled. This closed-loop operation effectively increases the residence time and reactant availability within the reactor, promoting more complete conversion of benzene under controlled reaction conditions. Consequently, the process achieves a substantial

Table 3. Sensitivity analysis of operating condition of reactor by varying pressure of benzene.

Pressure of benzene entering reactor (psia)	Chlorobenzene mole fraction leaving the Reactor
12.5	0.6427
13	0.6408
13.5	0.6389
14	0.6371
14.5	0.6383

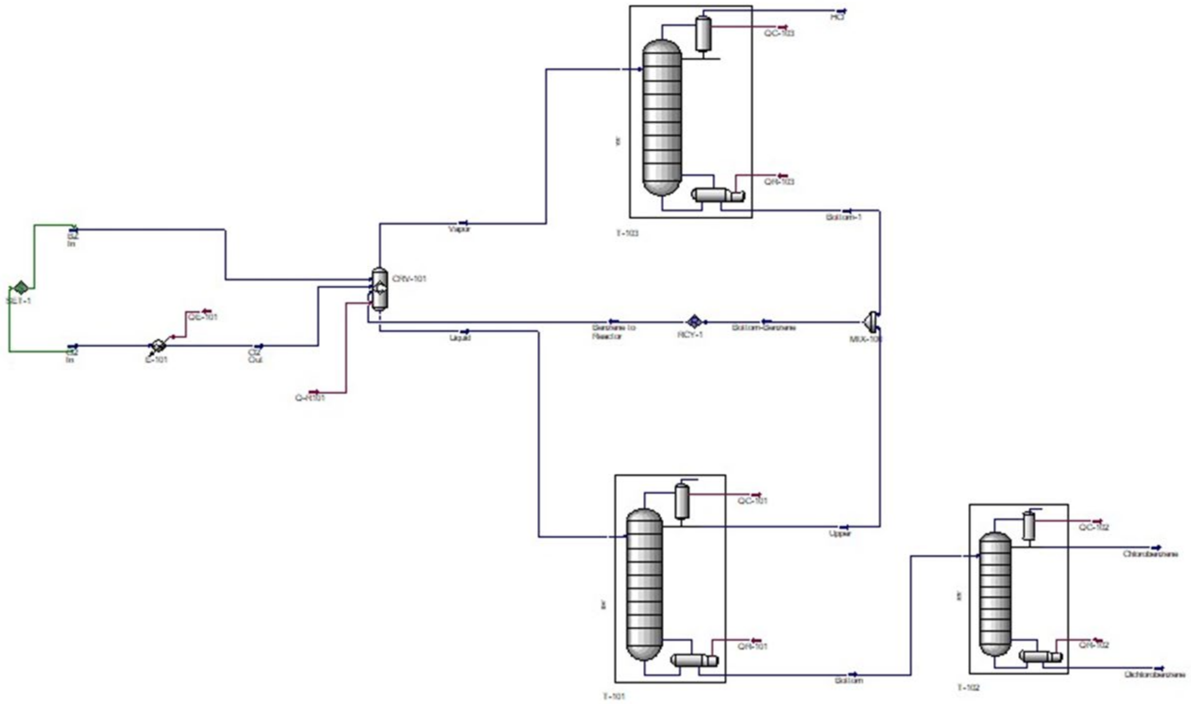

Figure 3. Aspen HYSYS process simulation of the modified process of benzene chlorination.

reduction in raw material losses and a marked improvement in product yield, culminating in the elevated chlorobenzene yield of 98%. Detailed mass and energy balance results from Aspen HYSYS simulations are presented in Supporting information: Table S1 and Table S2 (PDF).

3.3 Enhanced Product Yield through Process Modification with Added Mixer Unit

The implementation of the process modification demonstrates an increase in chlorobenzene yield by incorporating a mixer unit into the recycle loop. In this modified system, the mixer functions to combine unreacted benzene from the purification unit with the recycle stream originating from the chlorination section. This blended stream, which contains a higher concentration of benzene (C_6H_6), is then redirected to the chlorination reactor, improving reactant availability and enhancing conversion. By integrating the mixer unit, the process can utilize unreacted materials more efficiently, thereby reducing raw material consumption and lowering energy requirements. A comparison of the chlorobenzene yield between the original and modified processes is presented in Table 2. Mass and energy balances for both systems, obtained through Aspen HYSYS simulation, are provided in Supporting information: Table S1 and Table S2 (PDF).

3.4 Evaluation of Reactor Operation Sensitivity

The sensitivity analysis was performed by varying the independent variable, namely benzene pressure, to observe its effect on the dependent variable, the liquid chlorobenzene mole fraction. The case study employed pressure variations of 12.5 psia, 13 psia, 13.5 psia, 14 psia, and 14.5 psia. Table 3 summarizes the outcomes of this sensitivity analysis, showing how changes in benzene pressure influence the chlorobenzene mole fraction at the reactor effluent.

The findings of the case study reveal that increasing benzene pressure leads to a reduction in the yield of liquid products exiting the reactor. Chlorobenzene, as the principal product, necessitates a high yield upon exiting the reactor to optimize the separation process [17]. To reduce the load on the separation unit, the process may operate at lower benzene pressure, since higher reactant pressures demand costly treatment and storage facilities [18]. By applying reduced benzene pressure, energy efficiency is achieved as the separation unit's burden is minimized. Figure 4 illustrates how pressure influences the sensitivity of benzene, while Figure 5 illustrates how benzene pressure influences the chlorobenzene mole fraction.

A sensitivity analysis was further carried out by varying the independent variable, namely the ratio of benzene mass flow to chlorine gas mass flow, to examine its effect on the dependent variable, the liquid chlorobenzene mole fraction. The case study was performed with ratio variations of 0.87, 0.88, 0.89, 0.90, and 0.91. The results of this analysis, showing the influence of the benzene-to-chlorine mass flow ratio on the chlorobenzene mole fraction, are presented in the following table.

The results of the case study demonstrate that an increase in the benzene-to-chlorine mass

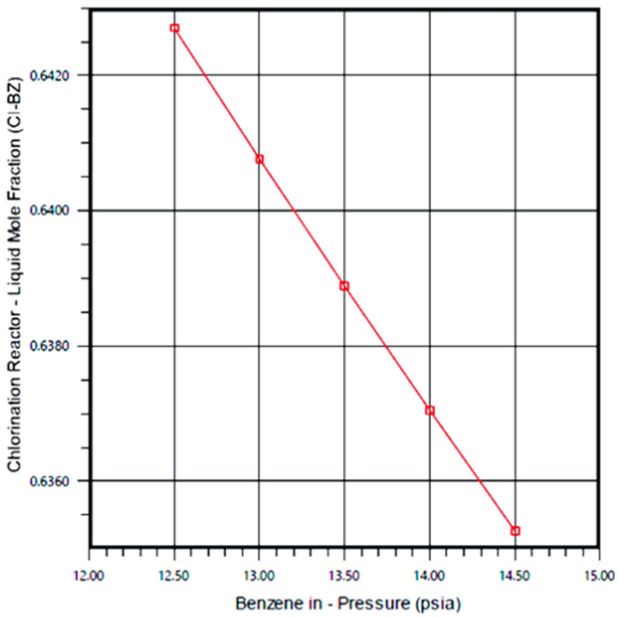

Figure 4. Sensitivity analysis of benzene partial pressure.

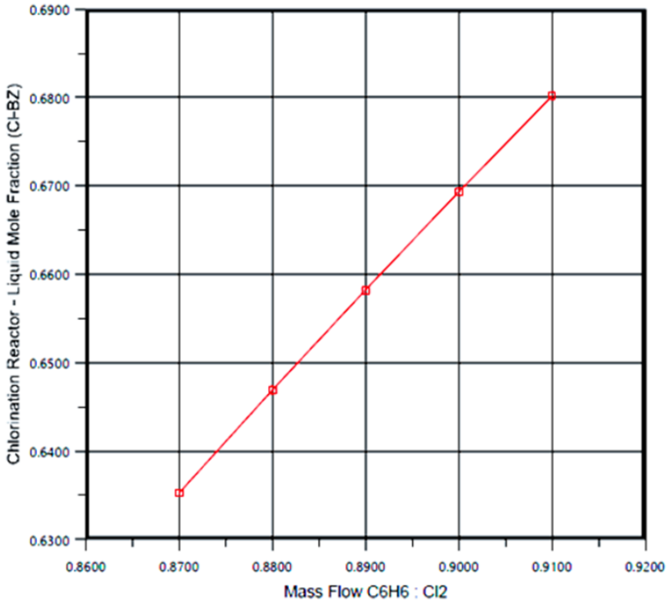

Figure 5. Sensitivity analysis of the ratio of benzene mass flow to chlorine gas mass flow.

flow ratio enhances the yield of liquid products exiting the reactor. Consistent with the findings of Sadeghi and Ahangar [19] higher mass flow rates promote greater production of the primary product, whereas reductions in mass flow tend to favor the formation of by-products. Furthermore, Figure 5 illustrates the relationship between benzene partial pressure and the mole fraction of chlorobenzene, highlighting the operational sensitivity of the system.

4. Conclusion

Process modifications in chlorobenzene production are essential to enhance both energy and mass efficiency. Such modifications enable factories to optimize the utilization of raw materials and energy, minimize environmental impacts, improve product yield, and strengthen overall operational performance. As a result of the implemented modifications, the yield of chlorobenzene has increased significantly, from 83% to 98%. Nevertheless, further investigations into the benzene chlorination process remain necessary. In particular, comprehensive economic analyses are required to develop the process so that it becomes more energy-efficient and cost-effective in terms of raw material consumption. Equally important is the assessment of environmental impacts, given the growing global emphasis on sustainability and the seriousness with which environmental issues are being addressed in this decade. Pollution arising from industrial production represents a critical concern that is increasingly subject to regulatory frameworks and enforcement.

CRedit Author Statement

Author Contributions: T.S. Hisanah: Conceptualization, Methodology, Investigation, Resources, Data Curation, Writing, Review and Editing, Project Administration; K. Raisa: Conceptualization, Methodology, Formal Analysis, Data Curation, Writting Draft Preparation, Visualization, Software, Supervision; N. Apriturlina: Conceptualization, Methodology, Formal Analysis, Data Curation, Writting Draft Preparation; E. Sidhik: Investigation, Resources, Visualization, Software, Supervision. N.I. Akbar: Investigation, Resources, Visualization, Software, Supervision. Y.C.N. Sirait: Investigation, Resources, Writing, Review, and Editing. All authors have read and agreed to the published version of the manuscript.

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