

Process Intensification of Glucose Hydrogenation Through Excess Hydrogen Feed and Hydrogen Recycle Integration for High Purity Sorbitol Production

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Abstract

High-purity sorbitol is an essential intermediate for food, pharmaceutical, and specialty chemical applications. However, conventional glucose hydrogenation flowsheets often face challenges such as low hydrogen utilization and inadequate product purity. This study introduces a process intensification strategy at the flowsheet level, evaluated using simulation software, which combines excess hydrogen feeding with flash-based hydrogen recovery and a recycle purge loop. The approach enhances both the conversion driving force and downstream separation efficiency. Starting from a once-through base case, the intensified configuration increases the H₂-to-glucose molar feed ratio to 4:1 and incorporates mixers, splitters, and a flash separator to recover unreacted hydrogen for recycle, minimizing hydrogen loss and stabilizing reactor hydrogen availability. Simulation results indicate a significant improvement in product quality, raising sorbitol purity from 74 wt% in the base case to 99.37 wt% in the intensified scheme. Overall, the proposed excess-hydrogen-plus-recycle integration offers a scalable solution for achieving >99 wt% sorbitol while optimizing hydrogen management through an improved separation recycle sequence.

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Keywords: Sorbitol; Glucose; Catalytic Hydrogenation; Hydrogen recycle; process intensification

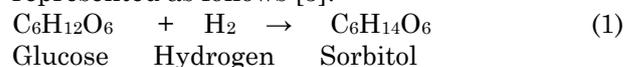
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1. Introduction

Sorbitol, a polyol obtained through the reduction of glucose, exhibits non-cariogenic properties, high chemical stability, and functional roles as a humectant and low-calorie sweetener. These characteristics underpin its extensive use across the food, pharmaceutical, and cosmetic industries [1]. Moreover, its production from renewable carbohydrate sources highlights sorbitol as a strategically significant compound in advancing biorefinery-based chemical processes [2].

Driven by growing consumer demand for environmentally friendly and health conscious products, global sorbitol consumption has shown a steady upward trend [3]. Industrial-scale production now reaches several hundred thousand tons annually, primarily via catalytic hydrogenation of carbohydrate feedstocks such as starch and cellulose [4]. The main reaction for glucose hydrogenation to sorbitol can be represented as follows [5]:



Water serves as the solvent, remaining in the liquid phase and influencing separation processes, though it does not appear as a stoichiometric

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product. To improve conversion efficiency, selectivity, and yield, various strategies have been explored, including adjustments to key operational parameters. For example, the use of Raney nickel (Ni) as a catalyst typically achieves sorbitol purities of around 54% [6]. However, this catalyst presents notable drawbacks, such as high cost and additional treatment steps to mitigate deactivation. Furthermore, experimental studies conducted at elevated temperatures (463 K) have only achieved purities of approximately 70% [5], underscoring the persistent challenge of attaining high product quality under such conditions.

Given the limited purity gains reported in previous studies, a more systematic evaluation of hydrogen availability and recovery is essential to intensify the glucose-to-sorbitol process. In this work, process intensification was achieved through two key modifications: (i) increasing the hydrogen-to-glucose molar feed ratio from the base case to 4:1, and (ii) incorporating a flash-based hydrogen recovery and recycle loop to reutilize unreacted H₂. The impacts of these strategies on reactor performance and downstream separation were assessed using Aspen HYSYS V11 simulations, with the primary objective of attaining maximum sorbitol purity.

2. Method

In the glucose hydrogenation process, the feed comprises glucose as the substrate, water as the solvent, and hydrogen gas as the primary reactant [7]. Sorbitol is produced as the main product and subsequently purified in downstream separation units. The hydrogenation occurs in a gas-liquid multiphase system, where hydrogen remains in the gas phase while glucose and its reaction products stay in the liquid phase [8]. In this study, the physical properties and phase equilibria of all process streams were determined using Aspen HYSYS V11, employing the Non-Random Two-Liquid (NRTL) thermodynamic model.

Aspen HYSYS applies the NRTL model by utilizing pure-component vapor-pressure and VLE data to estimate the necessary binary interaction parameters. The NRTL model, an activity-coefficient-based framework, is widely employed to characterize non-ideal liquid mixtures [9]. By accounting for molecular interactions that deviate from ideality, it enables accurate prediction of component activities and phase behavior. In this study, NRTL was used to calculate phase equilibria, including dew points, bubble points, and liquid-vapor splits, under operating conditions relevant to glucose hydrogenation and sorbitol purification [10]. These thermodynamic properties provide the

foundation for process flowsheet design and performance evaluation.

The process was analyzed and intensified by systematically varying the molar amount of hydrogen supplied to the reactor and incorporating a hydrogen recycle loop [11]. In the base case, the system operates with a hydrogen-to-glucose molar feed ratio near stoichiometric (approximately 1:1) and without hydrogen recycling. Under these conditions, hydrogen availability in the reactor is limited to the fresh feed, while all unreacted hydrogen exits through the separation section. This configuration reflects conventional operation, characterized by low hydrogen utilization efficiency and limited potential for achieving higher sorbitol purity.

In the intensified configuration, hydrogen is supplied in deliberate excess and combined with a recycle stream to increase both the instantaneous and overall hydrogen-to-glucose molar ratio. The mixed feed achieves a nominal ratio of 4:1 (H₂:glucose), ensuring that the reaction is not limited by hydrogen availability [10]. Operating under excess hydrogen conditions elevates the hydrogen partial pressure in the reactor, thereby enhancing the driving force for hydrogenation, promoting higher glucose conversion, and reducing the concentration of unreacted glucose in the liquid phase. From a selectivity perspective, maintaining a high hydrogen-to-glucose ratio also suppresses side reactions that tend to occur under hydrogen-lean conditions. Consequently, comparing the near-stoichiometric base case with the 4:1 intensified case provides a direct assessment of the influence of hydrogen molar ratio on reaction performance and product quality.

To complement the increase in fresh hydrogen supply, a recycle system was introduced to recover unreacted hydrogen and return it to the reactor feed. In the base case, all excess hydrogen leaving the reactor is vented, meaning the overall hydrogen-to-glucose ratio depends solely on the fresh feed. In contrast, the intensified configuration employs a recycle loop that increases the effective hydrogen molar throughput without a proportional rise in fresh hydrogen consumption. Comparing the two cases, the base configuration represents a once-through hydrogen utilization strategy, whereas the intensified configuration combines excess fresh hydrogen with a closed recycle loop, thereby improving hydrogen utilization efficiency and reducing net hydrogen losses from the system [12].

The reactor feed preparation and operating conditions follow the same structure in both configurations, with the key differences being the hydrogen molar ratio and the presence of a recycle loop. For each case, the mixed feed is directed to a

conversion reactor operating at 408 K and 1 atm [13]. The reactor effluent consists of a gas phase enriched in hydrogen and a liquid phase containing water, sorbitol, and unreacted glucose. The separation sequence is then designed to first remove the most volatile and abundant gas—hydrogen—so that it can either be purged or recycled, depending on the case considered.

In the intensified flowsheet, a flash vessel (V-100) is employed to separate the reactor effluent into two streams: a hydrogen-rich vapor and a liquid phase containing sorbitol, water, and unreacted glucose [14]. The hydrogen-rich vapor, which carries the unreacted hydrogen, is split via a tee valve into two fractions: (i) a purge stream, discharged from the process to prevent the accumulation of inert or undesired gases, and (ii) a recycle stream, routed for further purification. The purge stream ensures compositional stability within the recycle loop, while the recycled fraction increases the total hydrogen available to the reactor without requiring a proportional increase in fresh hydrogen supply.

The hydrogen stream designated for recycle is directed to the splitter unit (X-100) for further purification. In this unit, purified hydrogen is recovered as the overhead product and returned to the feed mixer, thereby increasing the hydrogen-to-glucose molar ratio at the reactor inlet. The bottom stream from X-100, containing water vapor and impurities, is combined with the liquid bottom product from V-100 and sent to subsequent purification steps. This configuration enables the recycle loop not only to maintain an elevated hydrogen molar ratio in the reactor but also to reduce the net fresh hydrogen requirement compared with an equivalent once-through operation at similar hydrogen partial pressure [15].

Finally, the bottom product from V-100, comprising sorbitol, water, and residual glucose, is fed to the T-100 distillation column for concentration and final purification. The column is configured to remove water as the overhead

product, while sorbitol, due to its significantly higher boiling point, remains in the column and is recovered as the bottom product. Under intensified operating conditions, the combined effect of a higher hydrogen-to-glucose molar ratio and an efficient hydrogen recycle loop enables the production of high-purity sorbitol. The modified flowsheet achieves a final purity of 99.37 wt%, compared to only 74 wt% in the base case. This direct comparison underscores the critical role of hydrogen molar ratio and recycle configuration in driving the process-intensification strategy implemented in this work.

3. Results and Discussion

In this case study, sorbitol hydrogenation is performed using glucose and hydrogen as the primary raw materials. The reaction pathway is analogous to that observed in the production of propylene glycol from glycerol via hydrogenolysis. The overall reaction for sorbitol formation is represented in Equation (1). Figure 1 presents the process flowsheet for sorbitol production through this hydrogenolysis pathway.

The sorbitol production process from glucose was modified in Aspen HYSYS by integrating additional unit operations, namely a separator, tee valve, splitter, mixer, heat exchanger, and a hydrogen recycle system, to enhance separation efficiency and optimize hydrogen recovery. The flowsheet for the modified process is shown in Figure 2.

The process flowsheet shown in Figure 2 illustrates that glucose and hydrogen are first

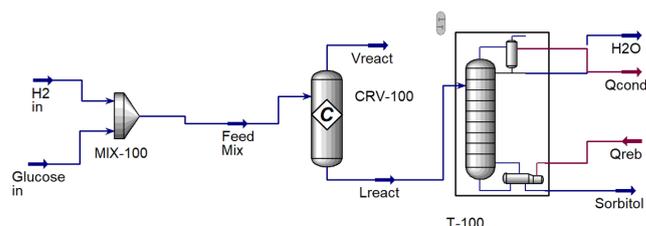


Figure 1. Process simulation of the sorbitol production process before modification.

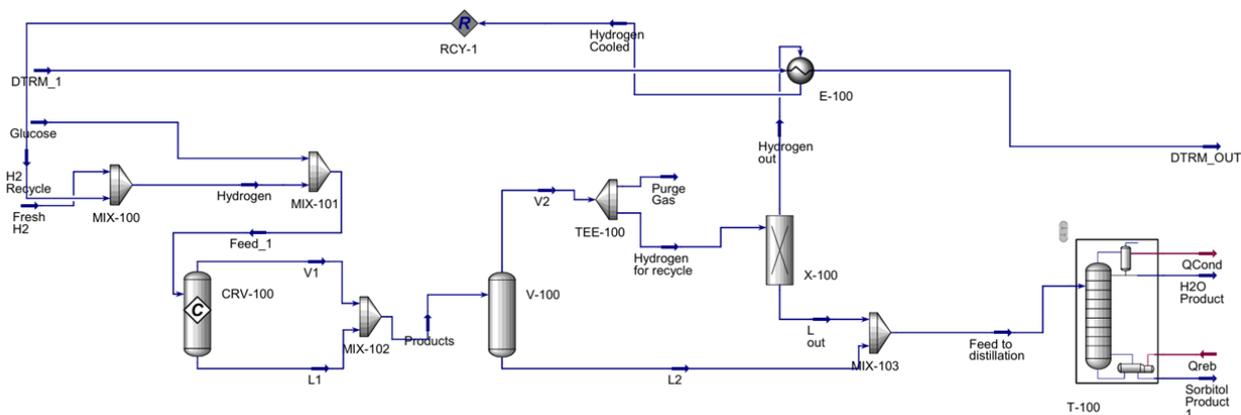


Figure 2. Process simulation of the modified sorbitol production process.

combined in mixing units M-100 and M-101 before entering the conversion reactor (CRV-100). This reactor is selected for its ability to rapidly determine reaction products and its suitability for systems incorporating hydrogen recycle. The hydrogenation reaction is carried out under operating conditions adapted from literature and refined within the simulation: a temperature of 408 K, a pressure of 1 atm, and a hydrogen-to-glucose molar ratio of 4:1. Supplying hydrogen in excess increases its partial pressure, enhances the driving force for hydrogenation, suppresses side reactions such as mannitol formation, and maintains high selectivity toward sorbitol.

Under these operating conditions, the HYSYS simulation indicates that most of the glucose is successfully converted to sorbitol. The reactor effluent forms a single homogeneous liquid phase. According to the simulation results, sorbitol purity at the reactor outlet reaches 94 wt%, while the remaining fraction consists primarily of water, along with unreacted glucose and a small amount of dissolved hydrogen. Despite the high conversion, the substantial water content results in a relatively low sorbitol concentration in the reactor output, necessitating additional purification steps.

The reactor effluent then enters the initial separation stage. The mixture from MIX-102 is fed to the flash vessel (V-100), where hydrogen is removed as the overhead vapor product, while the liquid phase, containing sorbitol, water, and unreacted glucose, is withdrawn as the bottom product. The overhead stream is directed to the TEE-100 splitter, which divides it into two fractions: a small purge stream to prevent the accumulation of inert gases and a recycle stream for further purification. The recycle stream enters the X-100 splitter, where purified hydrogen is recovered and returned to MIX-100. This hydrogen recycle loop is critical for maintaining the desired hydrogen-to-glucose molar ratio at the reactor inlet and for reducing the demand for fresh hydrogen feed.

The bottom product from V-100 is routed through MIX-103 to the T-100 distillation column for concentration and final purification. The column is configured to separate water as the overhead product, while sorbitol, due to its significantly higher boiling point, is recovered as the bottom product. Separation efficiency is enhanced through the use of a reboiler (Qreb) and a condenser (Qcond). Based on HYSYS simulation results, the bottom stream of T-100 yields sorbitol with a purity of 99.37 wt%, whereas the overhead stream consists primarily of water with trace impurities.

The reactor waste and streams from the separation units primarily contain hydrogen,

water, unreacted glucose, and trace by-products. Hydrogen is recovered through the flash vessel, tee, and splitter, then recycled to the front end of the process to reduce fresh hydrogen demand and minimize production costs. The separator also helps lower the distillation load, improving separation efficiency and reducing the heat duty required in the distillation column. Ultimately, this integrated sequence of reaction, separation, and purification steps enables the production of high-purity sorbitol (99.37 wt%), as confirmed by Aspen HYSYS simulation results.

When compared to the original process flowsheet, the performance improvements achieved through modification become evident. The base configuration consisted only of a mixer, the CRV-100 reactor, and a single distillation unit, without a flash vessel, splitter, tee valve, or hydrogen recycle loop. In this setup, the hydrogen-to-glucose molar ratio was limited to approximately 1:1, resulting in insufficient hydrogen partial pressure to achieve optimal hydrogenation. The absence of a recycle pathway further increased fresh hydrogen consumption and reduced overall reaction efficiency. These limitations contributed to the low sorbitol purity observed in the base case, approximately 74 wt% in the bottom product, along with a high water content due to constraints in the separation system.

The implementation of process modifications, namely the addition of a flash vessel, splitter, hydrogen recycle unit, and an increased hydrogen-to-glucose molar ratio, has been shown to significantly enhance overall process performance. These changes improve reaction efficiency by maintaining adequate hydrogen partial pressure, optimize component separation, reduce fresh material consumption, and ultimately deliver a sorbitol product with substantially higher purity. Overall, the revised flowsheet elevates product quality from 74 wt% to 99.37 wt%, demonstrating that the modified configuration is markedly more effective, economical, and selective than the original design.

In the base case, glucose hydrogenation to sorbitol was simulated using a conventional flowsheet comprising a mixer, a fixed-conversion reactor, and a single distillation column. Fresh hydrogen and aqueous glucose were mixed and fed to the reactor without any hydrogen recycle, maintaining a hydrogen-to-glucose molar feed ratio close to stoichiometric (approximately 1:1). Under these conditions, the reactor outlet contained a substantial amount of unreacted hydrogen, which was sent directly to the separation section along with the liquid phase. The distillation column produced a sorbitol-rich bottoms stream with a purity of 74 wt%, while a

large fraction of hydrogen exited in the overhead stream, indicating limited product purity and inefficient hydrogen utilization.

To intensify the process, the flowsheet was systematically modified by increasing the hydrogen-to-glucose molar ratio to 4:1 and introducing a hydrogen recovery and recycle loop. In the intensified configuration, fresh hydrogen is combined with recycled hydrogen and mixed with the glucose feed before entering the reactor, resulting in higher hydrogen partial pressure and a greater driving force for hydrogenation. The reactor effluent is then directed to a flash separator and splitting system, which separates the hydrogen-rich vapor from the sorbitol-rich liquid phase. A portion of the recovered hydrogen is recycled to the reactor feed, while the remainder is purged to prevent inert gas accumulation. The liquid stream is subsequently fed to the distillation column, where sorbitol is recovered as the bottom product with markedly higher purity.

The quantitative impact of process intensification is summarized in Table 1. In the base case, the fresh hydrogen feed was 7.996 kmol.h⁻¹ with no recycle applied, resulting in 40.01 kmol.h⁻¹ of hydrogen leaving the system. The sorbitol product flow was 19.31 kmol.h⁻¹ at a purity of 74 wt%. In the intensified configuration, the hydrogen-to-glucose molar ratio was increased to approximately 4:1 by raising the fresh hydrogen feed to 31.75 kmol.h⁻¹ and introducing a recycle stream of 2.857 kmol.h⁻¹, while maintaining the glucose feed at 67.04 kmol.h⁻¹. Consequently, the hydrogen purge decreased to 2.857 kmol.h⁻¹, and sorbitol purity improved to 99.37 wt%. The lower sorbitol molar flow in the product stream reflects a more concentrated product, as a larger fraction of water and light components is removed in the distillate.

Overall, these results demonstrate that combining excess hydrogen feed with a hydrogen

recovery–recycle loop significantly enhances both hydrogen utilization and separation efficiency. Elevated hydrogen partial pressure in the reactor promotes the desired hydrogenation pathway while suppressing side reactions, whereas upstream hydrogen removal and recycling reduce the gas load on the distillation column. Consequently, the intensified process flow diagram (PFD) (Figure 3) achieves ultra-high-purity sorbitol (99.37 wt%) compared to only 74 wt% in the base case PFD (Figure 4), confirming the effectiveness of this process-intensification strategy.

4. Conclusion

This study investigated process intensification of glucose hydrogenation to sorbitol by combining excess hydrogen feed with a hydrogen recovery–recycle loop in Aspen HYSYS V11. The base-case flowsheet, consisting of a mixer, a fixed-conversion reactor, and a single distillation column, produced sorbitol with a purity of 74 wt% when operated at an approximately stoichiometric hydrogen-to-glucose molar ratio and without hydrogen recycle. Under these conditions, a substantial amount of unreacted hydrogen exited the system, indicating inefficient hydrogen utilization and limited product quality. The flowsheet was systematically

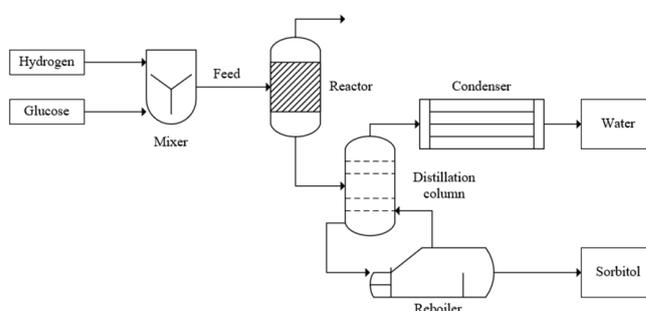


Figure 3. Process Flow Diagram (PFD) of the sorbitol production process before modification / intensification.

Table 1. Comparison of sorbitol purity before and after the modification process.

Parameter	Base case (unmodified)	Intensified case (modified)
Specified H ₂ :glucose molar ratio	1:1	4:1
Fresh H ₂ feed (kmol.h ⁻¹)	7.996	31.75
H ₂ recycle flow (kmol.h ⁻¹)	–	2.857
H ₂ leaving system / purge (kmol.h ⁻¹)	40.01	2.857
Glucose feed (kmol.h ⁻¹)	67.04	67.04
Sorbitol product flow (kmol.h ⁻¹)	19.31	5.877
Sorbitol purity (wt%)	74.00	99.37

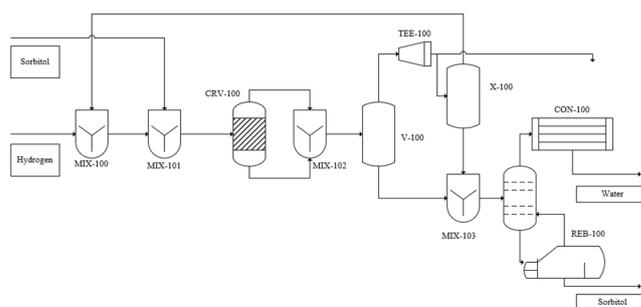


Figure 4. Process Flow Diagram (PFD) of the sorbitol production process after modification / intensification.

intensified by increasing the hydrogen-to-glucose molar ratio to 4:1 and integrating a flash-based hydrogen separation step followed by a recycle loop with purge control. This configuration reduced hydrogen losses by recovering and recycling a portion of the unreacted hydrogen while maintaining the same glucose feed rate. As a result, the sorbitol product stream became more concentrated, and its final purity increased to 99.37 wt%. The combined effect of higher hydrogen partial pressure in the reactor and improved upstream phase separation significantly enhanced hydrogenation selectivity and facilitated downstream distillation.

Overall, the results confirm that excess hydrogen feeding combined with a hydrogen recovery-recycle loop is an effective process-intensification strategy for producing high-purity sorbitol. Beyond increasing product purity from 74 wt% to 99.37 wt%, the intensified flowsheet also improves hydrogen utilization, delivering benefits from both energy and economic perspectives. Future work could extend this approach by incorporating detailed energy integration, evaluating catalyst deactivation effects, and performing comprehensive economic analysis to further assess the industrial feasibility of the intensified process.

CRedit Author Statement

Author Contributions: Y.D. Martela: Conceptualization, Methodology, Investigation, Software (Aspen HYSYS simulation), Formal Analysis, Writing, Review & Editing, Project Administration, Supervision; N.N. Putri: Conceptualization, Methodology, Investigation, Software (Aspen HYSYS simulation), Formal Analysis, Writing, Review & Editing, Project Administration, Supervision; N.E. Putri: Methodology, Investigation, Writing, Review & Editing; E.A. Aidina: Visualization, Data Curation; R.F.A. Putri: Writing (Abstract and Conclusion), Review & Editing; N.E.A. Anindya: Methodology & Editing. All authors have read and agreed to the published version of the manuscript.

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