

Research Article

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Experimental investigation of the synthesis gas purification using the PSA method to isolate hydrogen

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Highlights

- Adsorption capacity increases with higher pressures, reaching an optimum at 15 bar.
- The best adsorption durations are 3 minutes at 5 bar and 9 minutes at 15 bar.
- p/f ratio and pressurization direction significantly impact hydrogen purity.

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ABSTRACT

This study experimentally investigates the purification of synthesis gas using the pressure swing adsorption (PSA) method to isolate H₂. The effects of different pressures and flow rates on adsorption durations has been examined. The lowest adsorption capacity has been observed at 5 bar, while the highest has been recorded at 15 bar. It has been observed that the column adsorption capacity increases with increasing pressure. The results indicate an increase in column retention capacity with pressure. The optimum adsorption duration has been found to be 3 minutes at 5 bar and 9 minutes at 15 bar. Additionally, significant effects of p/f ratio and pressurization direction on H₂ purity has been observed. These findings are consistent with the study's objective which is to provide insights on optimization of PSA parameters and enhancing hydrogen purity. The results support a broader understanding of PSA technology and potential applications of hydrogen purification in industrial areas and paves the way for further progress. Future research suggests that modeling studies could provide further insights and the use of different adsorbents may enhance H₂ purity.

Keywords: Hydrogen purification, Pressure swing adsorption, Active carbon

1. INTRODUCTION

Hydrogen has gained a significant importance as a clean and sustainable energy carrier in fields such as transportation, energy storage and industrial applications. However, due to its wide flammability range and low ignition energy properties, hydrogen requires safety precautions especially in high-pressure systems. Explosion resistant structures play a vital role for lowering risks which can be occurred during hydrogen storage and transportation. Preventing catastrophic failures in hydrogen fueling stations, hydrogen pipelines and industrial facilities rely on advanced safety measures and robust storage systems. Highlighting these critical implementations will underscore the need for continuous progress in hydrogen industry and safe handling technologies.

In recent years, there has been a notable surge in interest in hydrogen as a potential alternative energy source, driven by the imperative for clean energy solutions. This trend is underscored by a thorough examination of research conducted over the past three decades, revealing a substantial corpus of scholarly articles and patent applications. Hydrogen finds extensive application across diverse sectors including petrochemistry, fuel cells, and the metallurgical industry. Hydrogen production primarily depends on technologies like steam reforming, but ensuring high purity is crucial, especially for applications such as fuel cells. In this regard, Pressure Swing Adsorption (PSA) plays a vital role as a key technology for achieving the required hydrogen purity. [1-3].

PSA systems function on the principle of channeling mixed gas under high pressure into adsorbents, where impurities are adsorbed, and subsequently expelling these impurities (desorption) at low pressure. Adsorbents possessing the ability to capture undesired gas components are packed into the PSA column, culminating in the generation of pure H₂ at the PSA outlet. Typically, the operational cycle of PSA systems encompasses pressurization, adsorption, desorption, and purge stages. Research has evidenced the feasibility of attaining high-grade hydrogen, typically falling within the 3-4 purity range, utilizing PSA technology. Additionally, while attaining higher purity levels through parameter optimization is feasible, it often entails heightened complexity and costs. A notable advantage of PSA technology is its low energy consumption. Given these factors, the significance of PSA in the realm of renewable and sustainable energy becomes evident. However, despite its reliance on seemingly straightforward components, the effectiveness of PSA hinges upon numerous parameters, each exerting a direct influence on its performance. These parameters encompass ambient temperature, system pressure,

flow rate, flow direction, the quantity and nature of adsorbents, column dimensions, and the number of columns employed [4-6].

Anna et al. [7] mentioned the mathematical and experimental studies using parameters such as adsorption pressure, P/F ratio, production flow rate, and production time, discussing their compatible and consistent results with each other. In another study, Yang et al. [8] created a dynamic model incorporating mass, energy, and momentum balances to obtain process optimization showing hydrogen recovery and conditions providing maximum hydrogen purity. Choi et al. [9], while examining CO₂ separation with PSA, developed a model that observes the effects of adsorption time and reflux ratio to achieve process optimization. Witte et al. [10], PSA performance can be analyzed by considering Key Performance Indicators (KPIs) such as productivity, recovery, and purity. In the study examining the effect of adsorption time and purge flow rate on PSA, it was concluded that total productivity increases recovery but reduces product purity. Among the many parameters affecting PSA performance, column dimensions are also crucial. Findings from Ankit et al. [11] showed the impact of column length/column diameter (L/D) ratio on PSA performance. There are findings indicating that adsorbent selection is also a critical step. Sarkera et al. [12], when the effect of different types of activated carbon and zeolites on CO₂ adsorption was examined, it was observed that activated carbon provides the highest performance while 4A zeolite provides the lowest.

Several studies have demonstrated the efficiency of PSA technology by focusing on optimizing operational parameters such as pressure, flow rates and adsorbent selection. This study aims providing experimental insights into single-column PSA systems by using these parameters. In this experimental study sought to examine the fundamental dynamics of PSA at room temperature, employing activated carbon within a single column. This setup afforded a detailed investigation into the influence of adsorbent capacity on removing impurities from synthesis gas within the Single-Column PSA system. Consequently, the study aimed to gather data on the requisite amount of adsorbent for each impurity and the optimal operating pressures of the system, facilitating system refinement and optimization. Ultimately, the objective was to generate findings that could serve as a benchmark for scale-up endeavors in the future.

2. EXPERIMENTAL SETUP

The experimental setup, as outlined in Figure 1, consists of input ports, a single column, and an output line. The setup includes two separate inlets (1-2), one for the supply of synthesis gas, composed of 2% CO, 4% CH₄, 26% CO₂, and 68% H₂, used in the experiment and another for the purge step. Check valves are placed immediately after both inlets to prevent backflow. To control the direction of flow, which is a critical parameter in PSA systems, manual valves are used in the setup. There are two outlet ports in the system. One outlet is used for collecting the purified product. This line is equipped with a back pressure regulator (BPR) and a needle valve to maintain the system at the desired pressure and to control the flow rate of the product gas.

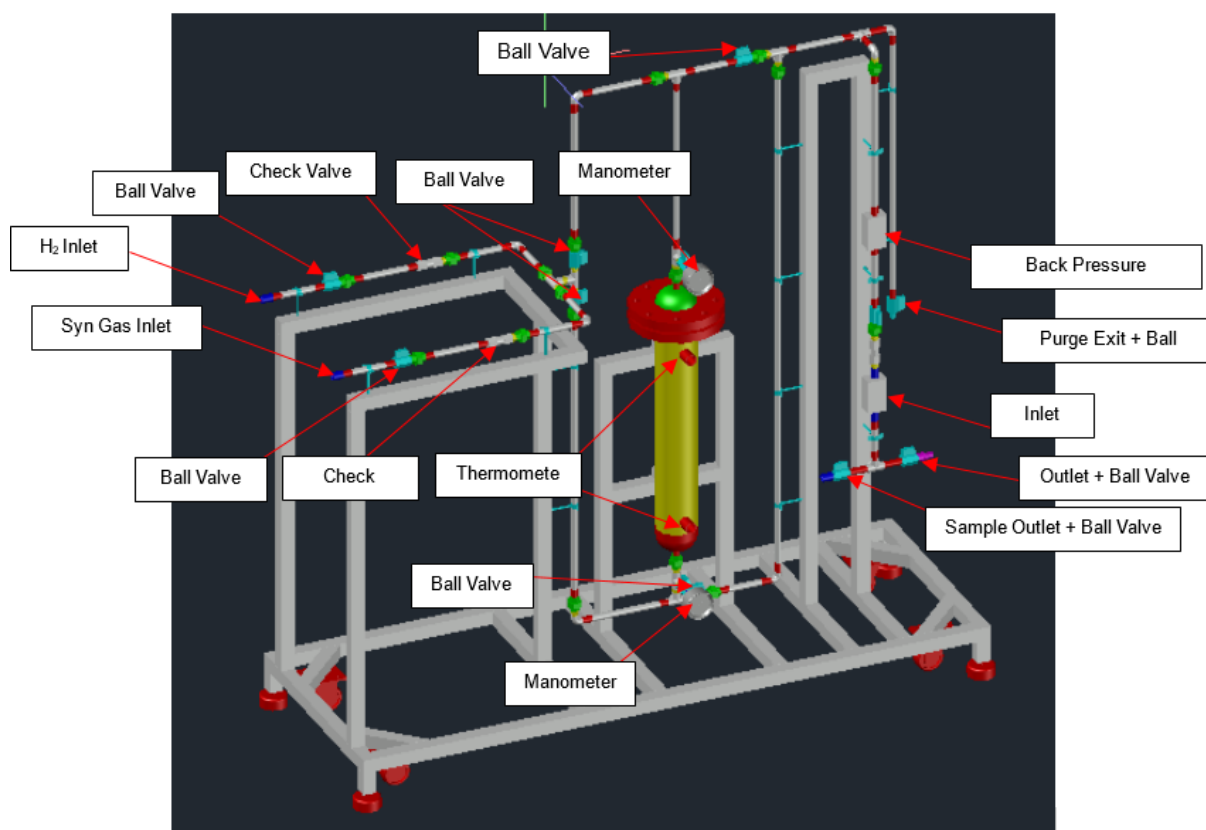


Figure 1. 3D view of lab scale PSA experimental setup

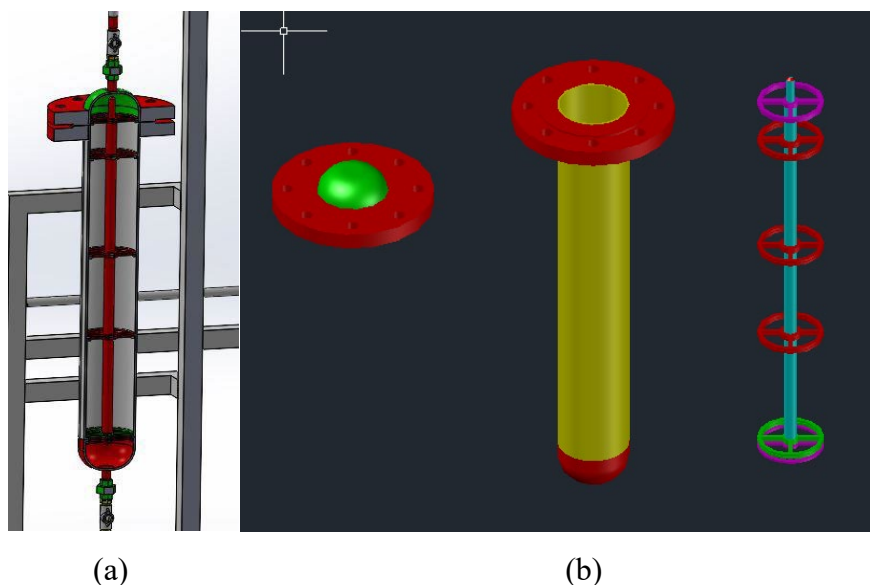
To ensure continuous flow, a second outlet is used for gas discharge when no samples are taken. For the purge process, flow meters are installed at the hydrogen gas inlet and the product sample outlet, while pressure and temperature gauges are placed at the column's inlet and outlet for parameter control. Column material selection is very important for manufacturing the experimental setup of PSA. As can be seen in Figure 2, the experimental setup is made from 304 stainless steel

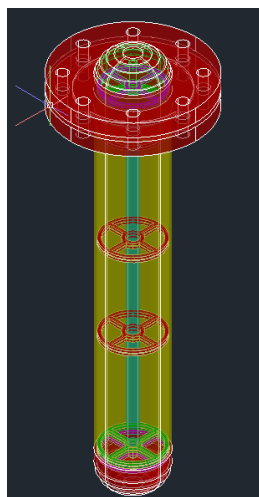
and assembled using threaded connections. Loctite is used to prevent leakage in the system. Stainless steel 300 series (e.g., 304 and 316) typically perform better and exhibit less hydrogen embrittlement when used with hydrogen [13-14].

Synthesis gas from the reformer and hydrogen gas for the purge process are supplied in cylinders. The adsorbents used were kept at 200°C for 2 hours to remove moisture and impurities. The experimental setup is designed to test three different adsorbents—Zeolite 13X, 5A, and Activated Carbon—placed in different layers within a single column. However, in this experiment, only the activated carbon studies are presented.



Figure 2. The production version of lab scale PSA experimental setup





(c)

Figure 3. The internal structure of a single PSA column (a), a PSA column with cap and layer separator (b), PSA column assembly (c)

The internal structure of the column is designed to fill different adsorbents separately (Figure 3a). The PSA column body consists of a cap, column body, rod, and layer separator disks (Figure 3b). The stem bar is positioned in the middle of the column and by doing so, the height of each adsorbent level can be adjusted. The principle is, placing nuts to a predetermined height and placing separation disc on. In Figure 3c, a transparent view of the PSA column can be seen. The cap of the column has a slot for sealant to prevent leakage.

2.1. Experiment Steps

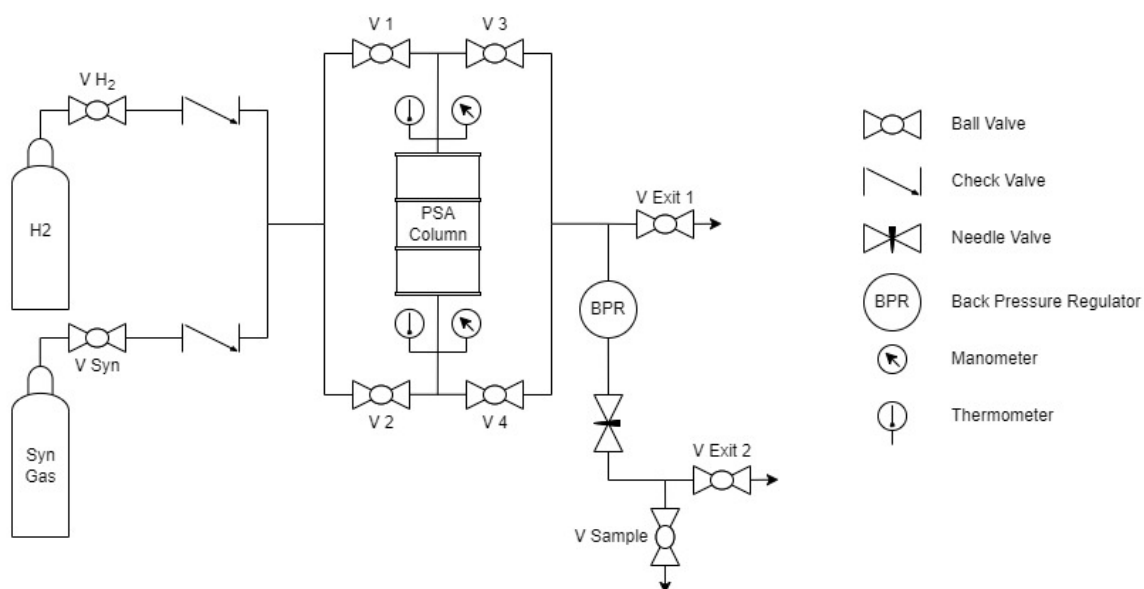


Figure 4. Schematic Layout of the PSA experimental setup

Figure 4 presents a schematic view of the system. The purification process consists of pressurization, adsorption, desorption, and purge steps. Detailed information for each step is provided below.

Step 1 Pressurization: The column is pressurized to the operating pressure with synthesis gas. Internal pressurization is achieved using a pre-set BPR (back pressure regulator), while the flow is adjusted according to the desired flow rate for adsorption using a flow meter.

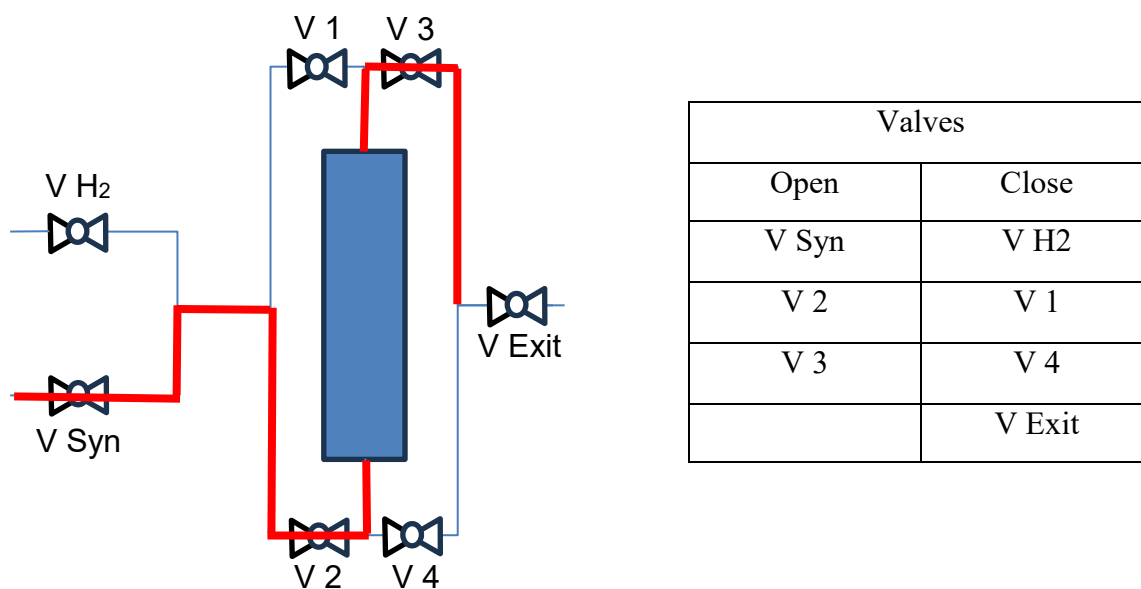


Figure 5. Simplified Schematic of Pressurization Step of PSA

As seen in Figure 5, Syn Gas Valve (V Syn), V2 and V3 are set as open to obtain flow from bottom to top, and the column is pressurized.

Step 2 Adsorption: Flow is maintained over the pressurized column at a constant outlet flow rate for the desired duration of adsorption.

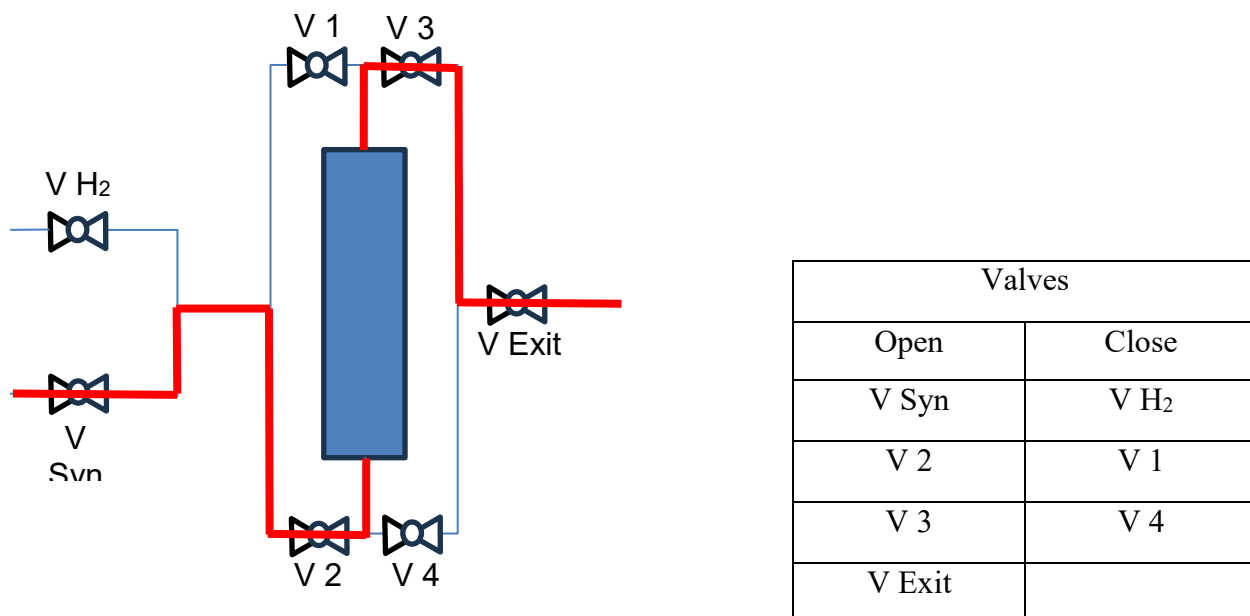


Figure 6. Simplified Schematic of Adsorption Step of PSA

After pressurization of the column, the outlet valve (V Exit) is set to open as in Figure 6, and with the help of BPR and needle valve, a constant flow rate is obtained.

Step 3 Desorption: The pressure in the column is released to atmospheric pressure.

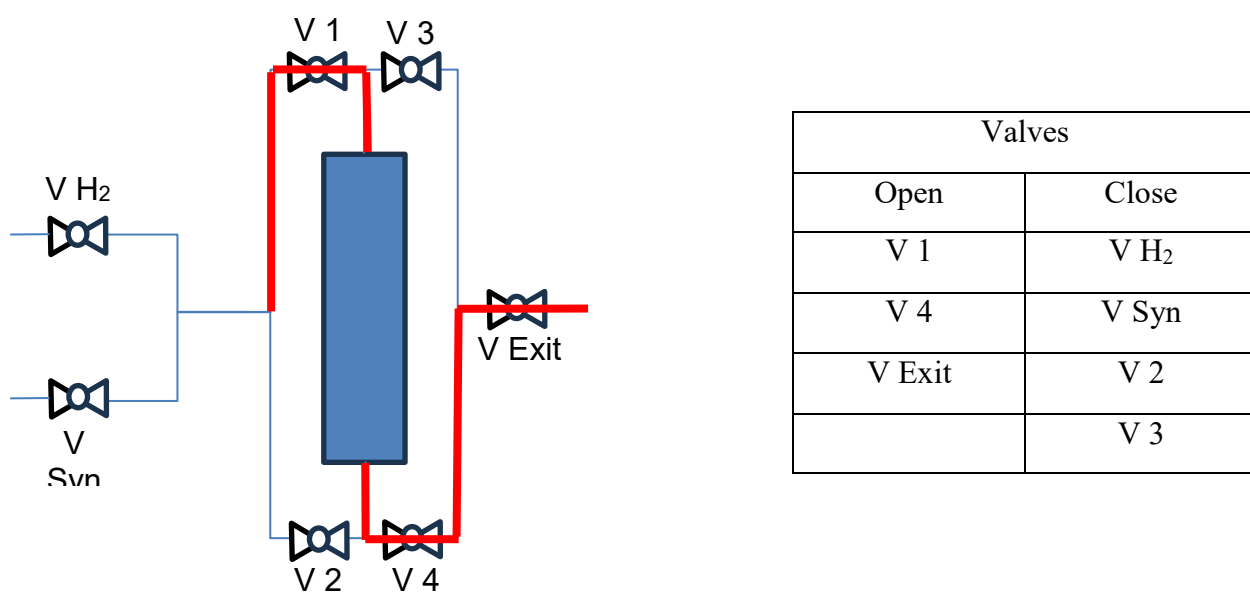


Figure 7. Simplified Schematic of Desorption Step of PSA

In the desorption step, valve orientation is changed (Figure 7) to obtain reverse flow in the column with the direction of top to bottom.

Step 4 Purge: The adsorbents are cleaned for the next cycle with a predetermined constant flow of H₂.

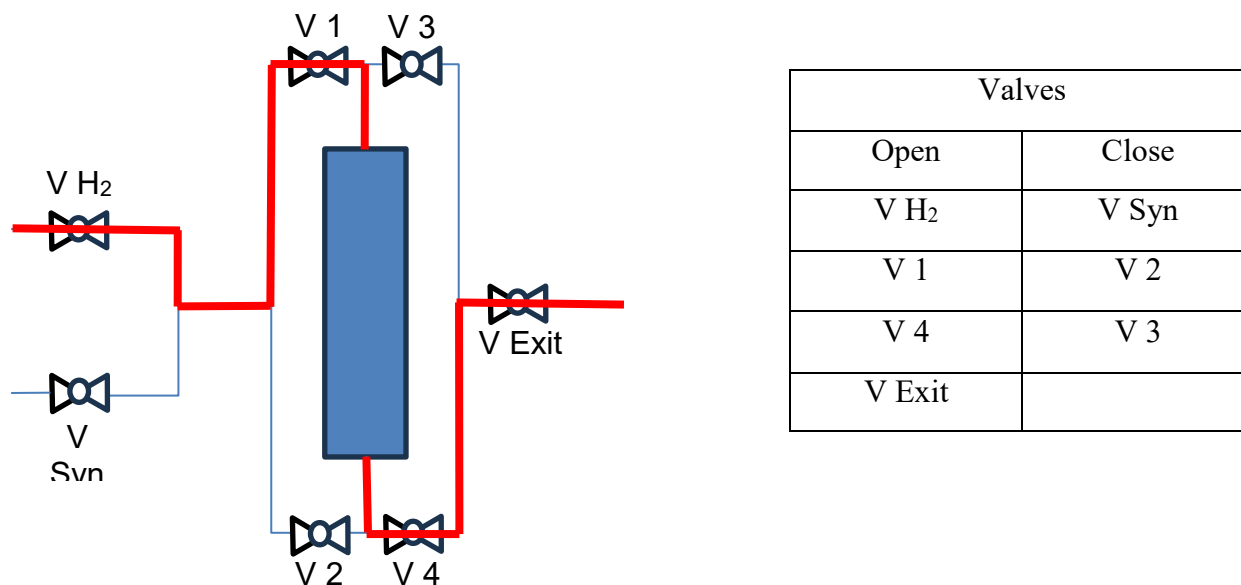


Figure 8. Simplified Schematic of Purge Step of PSA

In the purge step, the system is cleaned by constant flow of H₂ in the direction of top to bottom (Figure 8). By doing so, the PSA system is prepared for the next cycles.

2.2. Design Calculations

In the first phase of the column design study, the amount of hydrogen planned to be purified was determined considering the hydrogen consumption of approximately 10 L/min for a 1 kW PEM fuel cell. The PSA efficiency was assumed to be 50%. It was assumed that the required hydrogen would be supplied by a non-commercial Steam Methanol Reformer (SMR) system. As a result of the research conducted, the average composition of an SMR product gas mixture was determined. The synthesis gas mixture, composed of 2% CO, 4% CH₄, 26% CO₂, and 68% H₂, was supplied in cylinders. Subsequently, several different studies from the literature were considered to determine the column diameter and length.

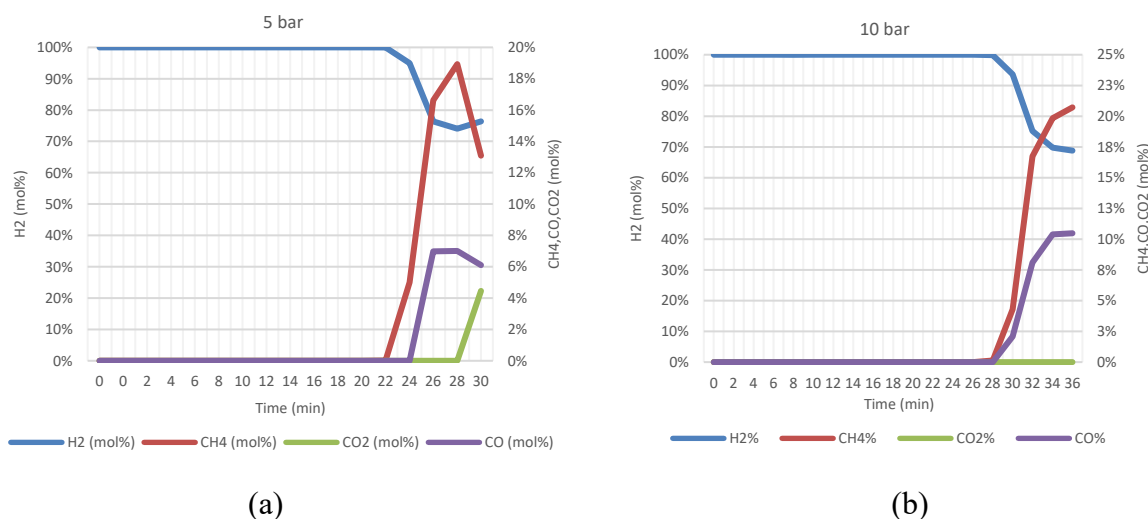
According to the study by Xiao et al. [15], the breakthrough time, defined as the time when CO exits ($t \approx 250$ s), was accepted. Additionally, according to Sarker et al. [16] the isotherm curve in Figure 10, the adsorption capacity of Zeolite 5A at 10 atm pressure and 293°K temperature is approximately 4.5 mol/kg adsorbent. In this direction, the CO₂ adsorption capacity of Zeolite 5A was calculated as approximately 4.5 mol CO₂/kg adsorbent over 1250 s at 9.12 bar and 16°C (Fig. 9 and 10). Based on this value, the amount of adsorbent was calculated through the amount of CO and CO₂ retained during the 250 s cycle period, and the bed volume was determined using the bulk density value. The Purge/Feed ratio, 0.13, is used in the calculations as stated in the study Abdeljaoued et al. [17]. Based on the values obtained from the literature, the column diameter was determined to be 82 mm and the length 540 mm for the system that was manufactured.

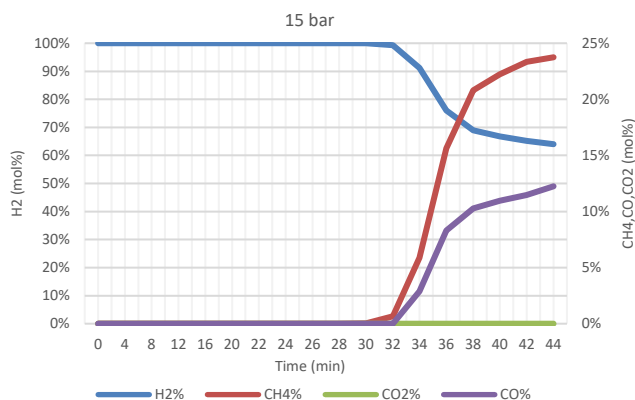
3. RESULTS and DISCUSSIONS

In this study, adsorption capacity experiments, adsorption time, flow rate and cycle experiments were performed under various pressures. Studies were carried out using activated carbon, which is the most preferred adsorbent for purifying synthesis gas.

3.1. Adsorption capacity of the column

To determine the adsorption capacity of the column under different pressures, breakthrough curves were generated at room temperature for pressures of 5, 10 and 15 bar. A constant sample flow rate of 5 SLPM was established for each pressure value. The percentages of H₂, CO, CO₂, and CH₄ were determined using gas chromatography for samples taken at regular intervals. The system was shut down approximately 10 minutes after observing impurities in the sample flow. Figure 1 presents the breakthrough curves at different pressures.





(c)

Figure 9. Breakthrough curves for 5 SLPM constant product flow rate at 5 bar (a), 10 bar (b), and 15 bar (c)

According to the graphs presented in Figure 9, the adsorption time of the column for pressures of 5, 10, and 15 bars were observed to be 22, 28, and 30 minutes, respectively. Each 5-bar increase increased by 4 minutes in the column's adsorption capacity. These results indicate a notable increase in adsorption capacity with increasing pressure. As pressure increases at a constant temperature, gas molecules interact more with the surface, leading to an increase in the amount of adsorption due to changes in thermodynamic equilibrium conditions. This phenomenon is explained by Le Chatelier's principle. The increase in system pressure is balanced by the adsorption of more molecules [18-20]. It was observed that at all three pressure values, the order of impurity emergence was CH₄, CO, and CO₂. Additionally, while CO₂ was seen at the 28th minute at 5 bar, CO₂ adsorption continued at 10 and 15 bar. This is due to the high selectivity of activated carbon for CO₂. The polarity of CO₂ molecules and the surface properties of activated carbon enable effective adsorption of this gas.

Based on the results obtained from the breakthrough curves, parametric studies were conducted at the lowest and highest retention capacities of 5 bar and 15 bar, respectively. Initially, at the working pressure of 5 bar, parameters such as adsorption duration, pressurization direction, purge-to-feed ratio, and product flow rate were examined. Considering the results obtained from these investigations, adsorption duration, flow rate, and cycle experiments were completed for 15 bar. In each experiment, the adsorption and purge durations were kept equal, as commonly done in the literature. For the examination of the adsorption duration effect at 5 bar, a comparison was made using the commonly employed 0.4 p/f ratio in the literature. The pressurization direction was

chosen to be from bottom to top, referencing the column orientation. After completing these experiments, the optimum value was selected and used for subsequent experiments. The molar percentages of the gases provided in the experiments were obtained from measurements taken immediately after the purge process was completed. The third stage of the PSA cycle, desorption, was completed for each working pressure by reducing the pressure to atmospheric levels within approximately 30 seconds. Additionally, after each parameter test, the column was fully regenerated with pure hydrogen to prepare it for the next test. To ensure the reliability of the results, given the manual control of the system, each experiment was repeated three times. The results presented below are the averages of these three values.

3.2. Operating Pressure Experiments

3.2.1. Experiments for 5 bar

Adsorption Duration:

To determine the appropriate adsorption duration, experiments were conducted with the operating pressure fixed at 5 bar and the product flow rate at 5 SLPM, testing adsorption durations of 3, 6, and 9 minutes.

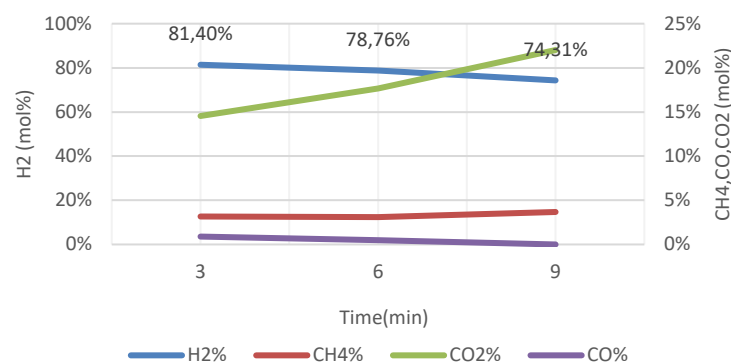


Figure 10. Comparison of different adsorption durations at 5 bar and 5 SLPM constant product flow rate

Figure 10 shows the molar percentages of H₂, CO, CO₂, and CH₄ in the product gas for different adsorption durations. A decrease in the H₂ percentage with increasing adsorption duration is noteworthy. The H₂ percentages for adsorption durations of 3, 6, and 9 minutes are 81.40%, 78.76%, and 73.31%, respectively. It is observed that the purge process is less effective for adsorption durations of 6 and 9 minutes compared to 3 minutes. The 3-minute adsorption duration,

which provided the highest H₂ percentage after purging, was selected, and subsequent studies at 5 bar were conducted using this value.

Purge to Feed:

For the p/f ratio, which is one of the most critical parameters in PSA systems, three different ratios were examined. As clearly shown in the graph provided in Figure 11, the 0.3 ratio was less effective for the column purge process compared to the other two ratios. For the 0.4 and 0.5 p/f ratios, the molar percentage of hydrogen measured after the purge process was approximately 77%. There is no significant difference between these two values. Therefore, the 0.4 ratio, which provides a better purge process with less hydrogen usage, was selected as the optimal value.

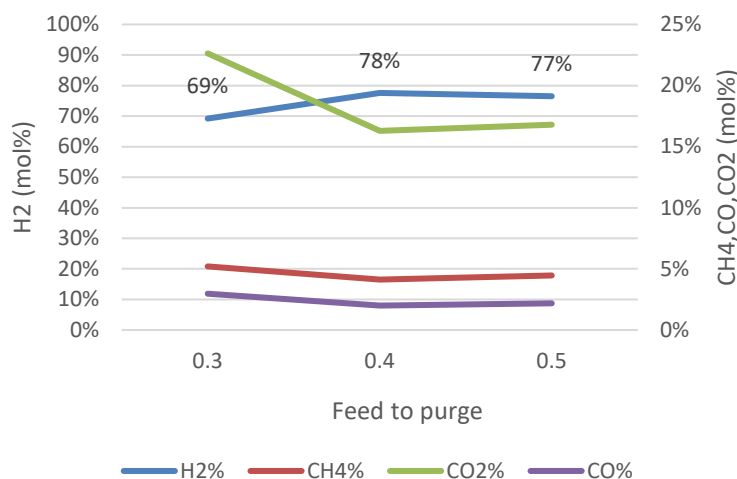


Figure 11. Comparison of purge-to-feed ratio

Direction of Pressurization:

In the breakthrough, adsorption duration, and p/f ratio experiments, the synthetic gas fed into the system for pressurization was directed from the bottom to the top of the column. In the experiment where the pressurization direction was changed, impurities were observed even during the adsorption step. While the molar percentage of hydrogen during the adsorption step was measured as 100% in all other experiments, this value dropped to around 87% when the direction was changed. This indicates that having the pressurization direction the same as the adsorption direction, and opposite to the purge and desorption steps, provides an advantage.

Different Flow Rates:

For the 3-minute duration, which provided the highest % H₂ value after the purge process, flow rates of 1, 2, 3, 4, 5, and 10 SLPM were examined.

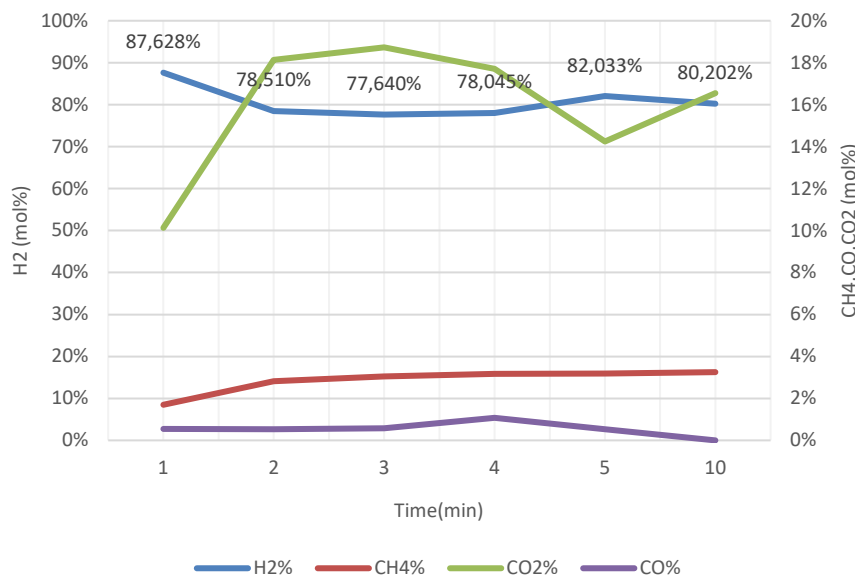


Figure 12. Comparison of different product flow rates using 3-minute adsorption/purge duration at 5 bar

In Figure 12, the results for 1, 2, 3, 4, 5, and 10 SLPM flow rates are compared. During sample collection, low flow rates of 1 and 2 SLPM caused difficulties in sampling. This explains the high H₂ percentage (87%) obtained at 1 SLPM. When examining the H₂ percentages obtained from all flow rates, the outlet H₂ percentage was measured to be approximately 78% on average, regardless of the outlet flow rate. This is due to maintaining a constant purge/feed ratio of 0.4 across all flow rates, resulting in a consistent percentage increase. The 5 SLPM flow rate, with an H₂ percentage of 82%, provided relatively better results.

Cycle Trials:

Another factor examined in PSA systems is determining the number of cycles required to reach cyclic steady-state conditions. For this purpose, adsorption and purge duration of 18 minutes, close to the total column adsorption capacity, were selected. In Figure 13, gas molar percentages are provided based on the cycle. For each cycle, pressurization, adsorption, desorption, and purge steps were completed and immediately moved to the next cycle.

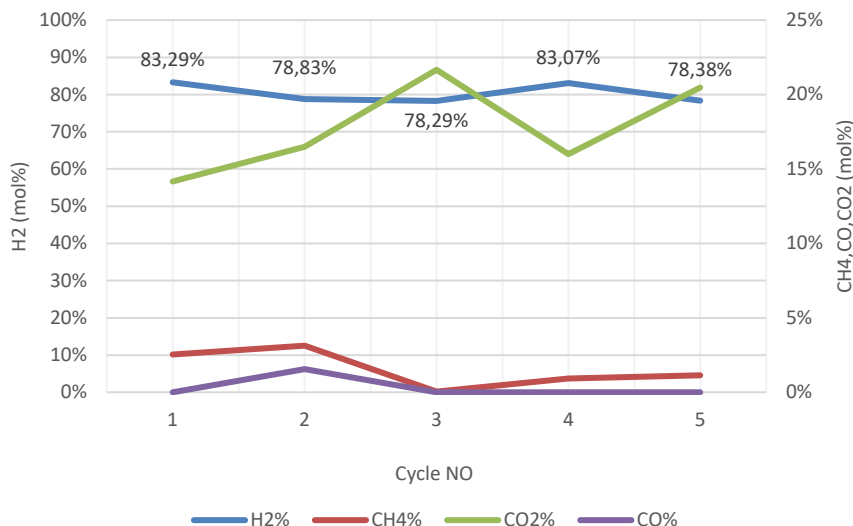


Figure 13. Comparison of cycles at 5 bar and 5 SLPM

It was observed that the product H₂ percentage, starting at 83% in the first cycle, decreased to 78% by the 5th cycle. An experimental margin of error is considered for the 83% observed in Cycle 4. Although more stable measurements were obtained after the first cycle, increasing the number of cycles is necessary to observe cyclic stability.

3.2.2. Experiments for 15 bar

Adsorption Duration:

For the working pressure of 15 bar, experiments began with adsorption/purge durations of 3, 6, and 9 minutes.

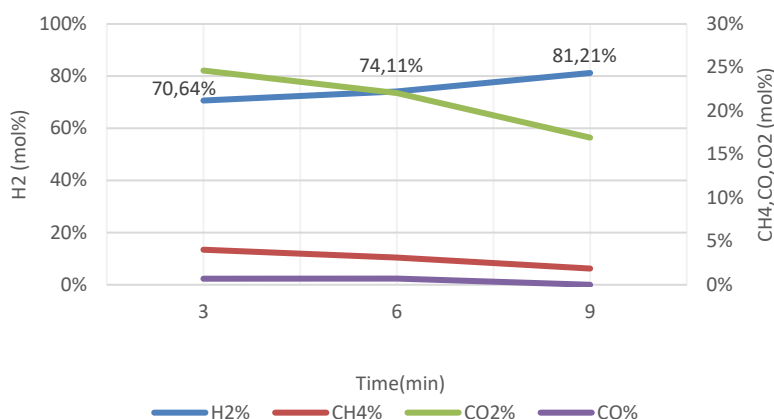


Figure 14. Comparison of different adsorption/purge durations at 15 bar and 5 SLPM

In Figure 14, the gas molar percentages measured after purging are provided for the three different duration experiments. For adsorption/purge durations of 3, 6, and 9 minutes, the H₂ percentages were measured as 70.64%, 74.11%, and 81.21%, respectively. In 5 bar experiments, the amount of H₂ for the same adsorption times changed in the opposite direction, as mentioned above. It is evident that with increasing column pressure, adsorption durations can be longer. This is explained by the fact that at higher pressures, more impurities can be removed from the column during desorption, resulting in fewer impurities needing to be removed from the column before the purge step [21-22]. Thus, a more effective impurity removal process is achieved for the column.

Different Flow Rates:

Due to the negligible difference in H₂% caused by flow rates of 1, 2, 3, 4, 5, and 10 SLPM in the 5 bar experiments, and the known higher adsorption capacity of the column at 15 bar from previous studies, a comparison of adsorption/purge durations of 3, 6, and 9 minutes was conducted at a flow rate of 10 SLPM.

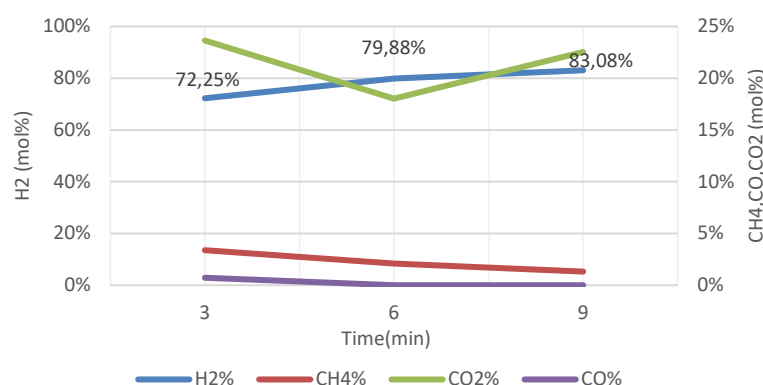


Figure 15. Comparison of different adsorption durations at 15 bar and 10 SLPM

The results obtained in Figure 15 confirm the findings in Figure 6, showing an increase in H₂% with increasing adsorption duration. The use of a 10 SLPM flow rate results in a proportional increase in the amount of H₂ used for purging due to the 0.4 p/f ratio. In the experiments with different flow rates in the 5 bar trials, this difference is observed to continue more steadily. At 15 bar, the increase in H₂ percentage is relatively higher with the effect of increasing pressure, and for each adsorption duration, this change averages about 3%. Therefore, it was decided to conduct the cycle experiments at a flow rate of 10 SLPM.

Cycle Trials:

The final experiment at 15 bar involves observing the change in H₂ quantity after 5 cycles. Until this parameter, all examinations were continued by selecting the experimental conditions that provided the highest value in H₂ measurement after purging. However, for this parameter, separate cycle experiments were conducted for adsorption durations of 3, 6, and 9 minutes.

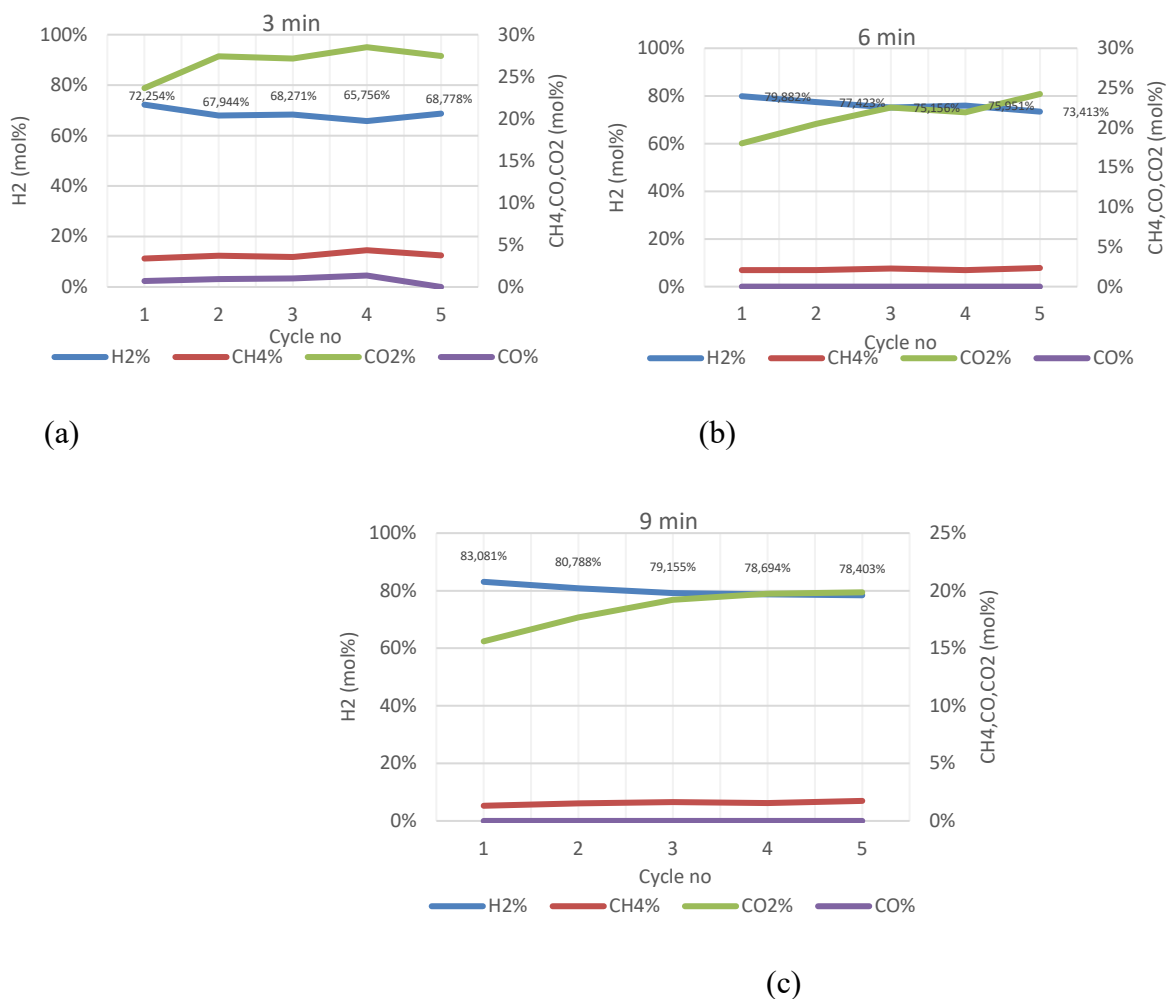


Figure 16. Comparison of cycles for adsorption durations of 3 minutes (a), 6 minutes (b), and 9 minutes (c) at 15 bar and 10 SLPM flow rate

In Figure 16, comparison graphs for 5 cycles are provided. According to the results obtained, for an adsorption duration of 3 minutes, the H₂ percentage decreased from 72% in the 1st cycle to 68% in the 5th cycle. The H₂ percentages decreased from 79% to 75% for 6 minutes and from 83% to 78% for 9 minutes. Similarly, the percentages of CO, CO₂, and CH₄ gases decreased proportionally. The best result in cycle experiments is obtained with an adsorption duration of 9

minutes. Therefore, the optimum adsorption duration for a working pressure of 15 bar and a flow rate of 10 SLPM is determined to be 9 minutes. The decrease in H₂ percentage is approximately 4% for 3, 6, and 9 minutes. It should be noted that a steady H₂ percentage was not achieved for all three adsorption durations, and more cycle experiments should be conducted to reach a cyclic steady state.

4. CONCLUSIONS

In this study, the focus was on purifying H₂ using the PSA method for synthesis gas with proportions of 2% CO, 4% CH₄, 26% CO₂, and 68% H₂. Active carbon was chosen as the adsorbent for purifying the synthesis gas. The experiments were performed on the most crucial parameters for PSA systems, including operating pressure, adsorption/purge duration, p/f ratio, column pressurization direction, various flow rates, and cycle experiments.

Breakthrough curves were generated at constant flow rates to determine the column adsorption capacity at 5, 10, and 15 bars. The lowest adsorption capacity was observed at 5 bar, while the highest was at 15 bar. It was observed that the column adsorption capacity increased with increasing pressure. In experiments with adsorption duration trials at 5 bar, a decrease in the H₂ ratio was observed with an increase in duration. Conversely, at 15 bar, an increase in adsorption duration led to an increase in the H₂ ratio. This is because more impurities are removed from the column at high pressure during desorption. While the optimum adsorption duration was determined to be 3 minutes at 5 bar, it was 9 minutes at 15 bar. Therefore, experiments at 5 bar were continued with this adsorption duration. Trials were conducted at 5 bar working pressure for p/f ratios of 0.3, 0.4, and 0.5. It was observed that the 0.3 ratio provided lower purification compared to the other two ratios. While the 0.5 ratio provided approximately the same purification level as the 0.4 ratio, it consumed less hydrogen, making 0.4 p/f the optimal value. Another noteworthy result is the effect of pressurization direction. When trials were conducted with the pressurization direction, the adsorption direction from bottom to top, and the desorption and purge direction from top to bottom, 100% H₂ was obtained in the adsorption step for all parameters. However, when the pressurization direction was opposite to the adsorption direction, the H₂ ratio decreased to 87% in the adsorption step. In experiments with different flow rates at 5 bar, no significant change in H₂ ratio was observed with an increase in flow rate. Due to the difficulty in sampling at low flow rates, 5 SLPM flow was determined to be optimal, and 5 cycles were conducted with each cycle lasting 18 minutes. A decrease in the H₂ ratio was observed after 5

cycles. Due to the issues encountered with low flow rates at 5 bar, flow rates of 5 and 10 SLPM were tested for adsorption durations of 3, 6, and 9 minutes at 15 bar. An increase in H₂ ratio was observed with an increase in adsorption duration for both flow rates. Transitioning from 5 SLPM to 10 SLPM for each adsorption duration resulted in a 3% increase. The optimum adsorption duration of 9 minutes was determined for 15 bar, 10 SLPM, and 9 minutes, resulting in the highest H₂ ratio of 83%. The 5-cycle experiment was completed with these optimum values. The highest H₂ ratio was obtained within 9 minutes after 5 cycles. More cycle experiments may be conducted to achieve CSS. As a result of the study, the optimum working pressure was determined to be 15 bar, with an adsorption/purge duration of 9 minutes, flow rate of 10 SLPM, p/f of 0.4, and the pressurization direction from bottom to top.

This study provides a comprehensive assessment of the effects of operating pressures, adsorption times, and purge/feed ratios on hydrogen purification using PSA technology, providing valuable insights for both academic research and industrial applications. By systematically examining the relationship between pressure and adsorption capacity, this study aligns with and extends previous research, reinforcing the practical importance of PSA optimization in high-purity hydrogen production.

Both in terms of time and cost, it is not feasible to conduct all experiments with pressurized synthesis gas cylinders. Based on the information obtained, future modeling studies could provide further insights. Additionally, studies using different adsorbents in a single column to obtain higher H₂ purity are another situation that should be examined.

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DECLARATION OF ETHICAL STANDARDS

The authors of the paper submitted declare that nothing which is necessary for achieving the paper requires ethical committee and/or legal-special permissions.

CONTRIBUTION OF THE AUTHORS

Kemalcan Sevuk: Writing, Methodology, Experiment, Visualization.

Berre Kumuk: Writing, Methodology, Visualization, Review & Editing.

Mustafa Ilbas: Writing, Methodology, Review & Editing, Supervision.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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