Design and Construction of a Helium Purification System Using Cryogenic Adsorption Process

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ARTICLE INFO	ABSTRACT
	One of the most appropriate methods for elimination of trace impurities in helium is cryogenic adsorption process. Therefore, in this study, design and construction of cryogenic adsorption helium purification system (3 $Nm^3.h^{-1}$, 80 bar) was carried out using activated carbon as an adsorbent at 77 K. For evaluation of adsorption dynamics and effect of pressure on elimination of trace impurities, helium purification was performed from 10 to 70 bar. Results showed that helium can be purified from 99.95 % to 99.99999 % at pressure of 60 bar using a cryogenic adsorption process. Effect of regeneration condition on helium purity and recovery was investigated by three different regeneration methods: regeneration by helium at 40 °C and at 180 °C as purge gas, countercurrent to feed direction, and regeneration via vacuum. Results showed that regeneration of bed by vacuum could be proposed as the most appropriate method because of maximum helium purity and high recovery of helium (98 %).

1. Introduction

Helium is one of the most widely used gases due to its extraordinary properties. It has a wide variety of important applications, especially in the aerospace industry, and defence. It also plays a vital role in the heat treatment of optical fibres. It is used for deep-sea divers (as a breathing mixture) to purge and pressurize liquid hydrogen rocket propulsion system due to its low liquefaction temperature and to cool superconducting magnets [1]. The worldwide consumption of pure helium has increased by between 5 and 10 percent a year in the past decade, with the biggest growth in its use as a coolant for the superconducting magnets in magnetic resonance imaging (MRI) body scanners.

Helium can be produced from air or natural gas [2, 3]. Generally, natural gas containing more than 0.3 percent helium is considered economic for helium extraction. A two-stage pressure swing adsorption (PSA) process was earlier developed by Knaebel and Rein-hold, and D'Amico et al. [4]. Their feed gas contained about 2-4 vol. % helium and 70 vol. % nitrogen and concentrated helium to a level of greater than 98 vol. % from the feed. The process involved two stages of PSA used in series that sequentially undergo a seven-step cyclic separation process. This helium is

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almost impure and should be purified to purity higher than 99.99 % [5, 6]. The choice of which purification method to use depends on the purity requirements of the particular process and the type of contamination in the waste stream. Typical purification techniques include pressure swing adsorption, membrane, and cryogenic technologies [7]. Final purification of helium, for removal of trace impurities, is typically done using either (a) an absorber at liquid nitrogen (LN_2) temperature and high pressure or (b) pressure-swing adsorption (PSA) processes [5]. Low-temperature adsorption can yield helium puritiy of 99.9999 % or higher purities, while PSA processes recover helium at better than 99.99 % purity [8]. In a cryogenic adsorption process, helium purifier works on high pressure and low temperature [1]. Low temperature of process is obtained by immersing the adsorbent column in LN_2 vessel. Adsorption of gases increses with a decrease in adsorption temperature, because adsorption is an exothermic process [9]. Impure helium gas passes through heat exchangers and enters adsorbent column containing an adsorbent such as silica gel, activated carbon, or zeolites. Numerous experimental and theoretical studies on helium purification by cryogenic adsorption process can be found in the literatures. Hsiao et al. [10] constructed a helium purification system for purification of helium to 99.9995 % by means of cryogenic adsorption process. The purifier removes impurities with 95 $Nm^3.h^{-1}$ feed flow rate on activated carbon bed at LN_2 temperature.

Stoll et al. [11] built a fully automatic large capacity helium purifier by means of cryogenic adsorption process with 135 Nm^3/h feed flow rate and 150 bar operating pressure. They cited that this system can decrease output air impurity level to less than 10 ppm, with cycle time of 24 hours which included operation, regeneration, precooling, and pressurization time. The adsorption bed was silica gel immersed in LN_2. Two heat exchangers, one immersed in the LN_2 bath and the other operating at 65 K existed before adsorber bed. Regeneration of the adsorbent bed was carried out by heating the adsorbent to 150 K, pumping out desorbed impurities, back filling with helium, re-pumping and, finally, backfilling with pure helium. Maiti et al. [12] used a helium purification system to purify impure helium (99 %, containing about 1 % air and moisture) at pressure of 20 bar and flow rate of 1.6 $Nm^3.h^{-1}$ by means of cryogenic adsorption process. They cited that by passing helium through activated charcoal bed at LN_2 temperature, helium was purified to 99.99 %. Thingstad [13] studied purification of neon-helium mixture with nitrogen, oxygen and moisture impurities by means of cryogenic adsorption process. In this purifier, helium-neon mixture was purified from oxygen and moisture by passing the feed from three heat exchangers and adsorber column containing linde-type 13X molecular sieve at LN_2 temperature. The heat exchangers were used as a dryer to remove moisture. Molecular sive was used for removing oxygen, nitrogen, and trace amounts of moisture. Regeneration of dryers and adsorber column performed by heating the purifier till 260 °C for two hours and evacuation of system for half-hour under vacuum.

In the present work, design and construction of a helium purification system by a cryogenic adsorption process has been evaluated. The first part of the paper deals with design of adsorber column in the cryogenic adsorption process. In this part,

BET eqution was used for estimation of the required absorbent mass, and accuracy of this eqution was validated against experimental data by purification of impure helium. Knowledge of adsorption dynamics in cryogenic adsorption process provides a valuable guide for design of the cyclic process. Thus, the second part of the paper is focused on the effect of adsorption pressure on product purity in a cryogenic adsorption process. After purification step, it is necessary to regenerate the adsorption columns and whole piping networks. Efficiency of the purifier system depends on the effectiveness of the regeneration process. The efficiency of this process can be improved by modifying the regeneration condition. Many studies have been performed on the regeneration condition of adsorber column in cryogenic adsorption process; however, almost no paper has been published on the effect of adsorber regeneration condition on performance of purifier. Therefore, in last part of the paper, three methods for bed regeneration were used, and effect of regeneration condition on helium recovery and purity was investigated.

2. Design of activated carbon adsorber column

Adsorption bed in cryogenic adsorption process removes all traces of impurities from impure helium at LN_2 temperature by cryoadsorption. Granular activated carbon is one of the best adsorbents in cryogenic adsorption process due to its maximum BET surface area and less bulk density as compared with the other adsorbents, which are commonly available in adsorption process such as activated alumina, molecular sieve, or zeolite, and silica gel. More BET surface area means more micropores for multi-layered gas adsorption. The less bulk density of activated carbon reduces the adsorber bed weight. Its regeneration temperature is also lower than other adsorbents, i.e., it is much easier and quicker to reactivate the saturated carbon. Nitrogen adsorption capacity [14, 15] of activated carbon is higher than molecular sieve which are commercially available, like silica gel and activated alumina at cryogenic temperature and higher pressure.

2.1. Adsorbent mass requirement

Quantity of adsorbents required to adsorb impurities in helium purification system, by cryogenic adsorption method, can be calculated with BET equation [16]. BET theory aims to explain the physical adsorption of gas molecules on a solid surface and serve as the basis for an important analysis technique for the measurement of the specific surface area of material. The BET equation of monomolecular layer formed over an adsorbent surface (the case in which only n layers of the gas can be adsorbed) is expressed as follows:

$$\frac{V}{m_a} = \frac{V_m Z x}{1-x} \cdot \left[\frac{1-(n+1)x^n + nx^{n+1}}{1+(Z-1)x - Zx^{n+1}} \right] \quad (1)$$

where V is the volume of gas adsorbed at 101.325 kpa and 273.2 K, m_a is the mass of adsorbent, V_m is the volume of gas per unit mass of adsorbent required to form a monomolecular layer over the entire adsorbent surface, x is $\frac{P}{P_{sat}}$, P is partial pressure of the gas being adsorbed, P_{sat} is saturation pressure of the gas being adsorbed at the temperature of the adsorbent [16]. Parameter Z is a function of adsorption energy and the temperature of the adsorbent:

$$Z = e^{\frac{\theta}{T}} \quad (2)$$

$$\theta(K) = \frac{\Delta H_{ads}\left(\frac{J}{mole}\right) - \Delta H_{con}\left(\frac{J}{mole}\right)}{R\left(\frac{J}{K.mole}\right)} \quad (3)$$

where ΔH_{ads} is adsorption heat of the first layer, and ΔH_{con} is condensation heat of adsorbed gase on adsorbent surface. For monomolecular adsorption n=1, the equation can be written as:

$$\frac{V}{m_a} = \frac{V_m Z\left(\frac{P}{P_0}\right)}{1 + Z\left(\frac{P}{P_0}\right)} \rightarrow m_a = \frac{V\left(1 + Z\left(\frac{P}{P_0}\right)\right)}{V_m Z\left(\frac{P}{P_0}\right)} \quad (4)$$

As shown in the equation, m_a is the adsorbent mass required for adsorption of one layer of gases on the adsorbent. Basic asumptions for design of helium purification system are shown in table 1.

As shown in the table, total amount of impurities in helium are asummed to be about 0.1 %. Therefor, quantity of impurities which enters the adsorber columns within 10 hours (V) can be calculated as follows:

$$V = (0.001) \times \left(3\ \frac{Nm^3}{h}\right) \times 10\ (h) = 0.03\ Nm^3 = 30000\ cc$$

Adsorption parameters of nitrogen and oxygen at 77 K are compared in Table 2.

Table 1
Basic assumptions for design of helium purification system.

He purity	Operation time	Total impurity	Operation pressure	Feed flow rate
99.9 %	10 (h)	Nitrogen	80 (bar)	3 Nm^3/hr

Table 2
Adsorption parameters of oxygen and nitrogen on activated carbon at 77 K.

Z	$V_m\left(\frac{cc}{gr}\right)$	$\theta\,(K)$	$P_0\,(atm)$	Gas type
49.34	181.5	300.2	1	Nitrogen
12.18	240	192.5	0.2	Oxygen

As shown in the table, nitrogen is harder to omit than oxygen and other impurities due to its higher P_0 and lower θ and Z quantites. Therefore, to simplify the calculations, total amounts of impurities in feed were assumed as nitrogen.

Therefore, based on this assumption and equation (4), recuired mass of activated carbon for adsorption of impurities in feed is equal to 165 gram. Since helium purification system should be regenerated after saturaton of activated carbon by impurities, estimation of adsorbent requirement is based on 70 % saturation of adsorbent column as suggested by Haselden [17]. Hence, quantity of activated carbon requirement is:

$$m_a = \frac{165\ gr}{0.7} = 235\ gr$$

2.2. Dimension of adsorber column

Velocity of helium stream through the adsorbent bed should have an optimum value, as high value of approach velocity through adsorbent bed results in bed fluidization, powdering of carbon particles, and insufficient amount of time for proper adsorption of impurities on the adsorbent surface. Appropriate adsorption of impurities on adsorbent almost ocuurs between 10^{-2} to 1

m.S^{-1} of the feed gas velocity ranges [18]. Dimension of adsorber column can be determined by placing the lower and upper limits of velocity in Equation (5).

$$Q\left(\frac{m^3}{s}\right) = A\,(m^2) \times v\left(\frac{m}{s}\right), A = \pi\frac{D^2}{4} \quad (5)$$

In this eqution, Q, A, D, and v are feed flow rate, cross section area of the column, internal diameter of column, and velocity of gas through fixed adsorber column, respectively. Inlet feed flow rate to adsorber column, at 77 K and 80 bar, is equal to $2.69 \times 10^{-6}\,\frac{m^3}{s}$. The range of adsorber column internal diameters, calculated based on the gas velocity upper and lower limits, is shown in Table 3.

Table 3
Velocity of the feed gas within the adsorber column according to the internal diameter (ID) of the column.

V (m/S)	ID of column (mm)
10 -2	18.51
1	1.85

As shown in the Table, adsorber column can be made of a tube with ID between 1.85-18.5 mm. It contains one eighth to three quarters inch tubes. One eighth inch tube has very small ID and can not be selected as adsorber column due to its high pressure drop, difficulty in adsorber loading, and the large acquired tube length. Length of the adsorber column can be calculated by equation (6) as follows:

$$L(m) = \frac{4\,m_a(kg)}{\rho\left(\frac{kg}{m^3}\right)\pi D^2(m)} \quad (6)$$

where ρ and L are apparent density of activated carbon used in adsorber column and length of the adsorber column, respectively. Acquired lengths of the adsorber column are shown in Table 4 based on equation (6) and internal diameter of the column.

Table 4
Acquired length of adsorber column according to the column ID.

Nominal diameter of the column (inch)	Wall thickness (mm)	ID (mm)	L (m)	Gas velocity (m/S)
$\frac{1}{4}$	0.762	4.826	26.03	0.147
$\frac{3}{8}$	0.762	7.966	9.48	0.054
$\frac{1}{2}$	0.762	11.176	4.85	0.027
$\frac{3}{4}$	0.762	17.520	1.97	0.010

Larger diameter columns require higher flow rates, hence larger volumes of feed gas. Therefore, at low feed flow rates, large internal diameter of the column can result in gas channeling and a decrease in feed gas velocity within the column. On the other hand, pressure drop increases in a packed column with a small internal diameter. To

reach the desired feed gas velocity, minimum pressure drop, and appropriated adsorption of impurities in a adsorber column, the internal diameter of bed should be balanced. In this study, a column with $\frac{1}{2}$ inch nominal diameter was selected as an adsorber column. In this column, the gas velocity is near to 10^{-2} m.S^{-1}, the pressure drop is low, and the probability of gas chanelling is almost low.

In this process, the adsorbent column should be immersed in LN$_2$ bath, and the space is limited. Therefore, an adsorbent column can not be constructed using straight pipes. A helical coiled form might be the most appropriate choice for construction of adsorber column. The number of turns of helicall coiled column (N) can be calculated using equation (7) [19].

$$N = \frac{L}{\sqrt{(2\pi r)^2 + p^2}} \qquad (7)$$

where L is lengh of pipe used for construction of helical coiled column, p is the spacing between consecutive coil turns (measured from center to center), and r is the radius of helix. The height of helical coiled adsorber column (H) can be calculated by equation (8).

$$H = N p + d_0 \qquad (8)$$

Adsorber column was made based on calculations. Charactristics of adsorber column are shown in Table 5. Austenitic stainless steel of specification 304 L was selected as dsorption column pipe material.

Table 5
Characteristics of adsorber column.

Parameters	Value (m)
do	0.0127
r	0.07
p	0.025
N	11
H	0.29

3. Experimental section
3.1. Apparatus and procedures
A schematic diagram of the apparatus used in cryogenic adsorption process is shown in Figure 1. The helix of adsorption column (A-01) was $\frac{1}{2}$ inch stainless steel (type 304) tube with 4.86 m length and 29 cm height. The column was packed with a granular local activated carbon, 1-3 mm. To prevent the carryover of adsorbent particles, microwire mesh was placed at the ends of the bed and stone wool was compressed on both sides of the bed (top and bottom of the bed). In-line filter was used to restrain entrance of fine particles to the gas lines. All lines were quarter-inch (6.4 mm) stainless steel tubes. Helium (99.95 %,) was used as feed gas and nitrogen (99.9 %,) was used to prepare gas mixtures. Characteristics of helium gas are shown in Table 6. The gas mixture (5 % N$_2$ in He) was prepared using two MFCs (mass flow controller, Alicat, USA). Check valves (CV) and microfilters were installed after mass flow controllers to prevent reverse flow and contamination. A gas mixer (GM) was installed after helium and nitrogen streams to mix the two gases better. Gas mixture composition and the outlet gas stream from adsorption bed were analyzed by an online gas chromatograph (TG2552, TGF Co Ltd, Iran) equipped with gas sampling valve (10 port, VICI), molecularsieve 5A and propack Q columns, TCD and FID detectors, preconcentration column (11 cm quarter inch copper tube containing 1 gr silica gel). Trace amount of impurities in ultra pure helium was analysed with the preconcentration technique using online GC [20]. A stainless steel helical coiled tube heat exchanger (E-02) was used to decrease the feed gas temperature till 77 K before adsorption column (5.5 m, quarter inch

tube). E-02 heat exchanger was immersed in LN$_2$ bath to decrease the feed temperature till LN$_2$ temperature. A needle valve (V-04) was installed after adsorption column to maintain constant pressure inside the adsorption bed by controlling the flow rate of the outlet stream. A pressure gauge (PI) was placed at the top of the bed to pressure reading. Needle valves were used to alternately direct the flow in and out of the column for adsorption and regeneration steps. An electrical furnace (E-01) was used to increase purge gas temperature (He) in the regeneration step. A vacuum pump (5 Nm^3 h^{-1}, Woosung rotary) was used for regeneration of bed by vacuum. Activated carbon was used as an adsorbent. It was activated at 473 K for 3 hr in a furnace before packing the bed.

Table 6
Characteristics of helium gas.

Impurity type	Concentration (ppm)
Nitrogen	120
Oxygen	20
Carbon dioxide	8
Carbon monoxide	0.5
Methane	1

3.2. Cryogenic adsorption process description

The cryogenic adsorption experiments for helium purification were performed using two types of feed: helium (99.95 %) and He/N$_2$ mixture (5 % N$_2$ in He).

Operation of purifier undergoes the following modes:

(1) Cooling down of purifier
(2) Bed feed pressurization (PR)
(3) High pressure adsorption (AD)
(4) Cocurrent depressurizing (CD)
(5) Regeneration

In cryogenic adsorption process, it is necessary to maintain the column temperature at LN$_2$ temperature. Therefore, LN$_2$ bath was filled with LN$_2$ in the pressure of 0.2 bar using pressurization system of Dewar before all experiment run. For purification of the helium feed, bed was pressurized to the desired pressure by the feed stream (PR step), while the valves of helium cylinder, V-01, V-03, V-04, and V-05 were opend and other valves were closed. Feed flow rate was controlled by MFC and was set at desired flow rate. For purification of the He/N$_2$ mixture, the gas mixture was made, while the valve of He and N2 cylinders was opened, and two MFCs were set at desired flow rate to prepare the gas mixture; after that, the gas mixture was analysed by onlie GC to confirm the gas mixture composition. Purification of gas mixture was done, while V-01, V-03, V-04, and V-05 valves were opend and other valves were closed. In all experiment runs, the desired column pressure was controlled by the pressure regulator connected to the gas cylinder. In AD step, the high pressure feed flows through E-02 heat exchanger and A-01 adsorption bed. After that, the product gas was analysed by online GC. CD step was performed by closing V-03 valve to decrease the bed pressure till atmospheric pressure. Before the regeneration step, liquid nitrogen was removed from LN$_2$ vessel. Regeneration of adsorption bed was performed by three methods: regeneration by vacuum, regeneration by hot helium stream, and helium at ambient temperature countercurrently to feed direction. Regeneration of bed by hot helium stream was performed, while the MFC set at desired flow rate and helium entered electrical furnace (E-01) and its temperature increased to a desired level. The hot helium stream entered the bed countercurrently to feed

direction by opening V-07, V-04, V-03, V-02 valves and closing V-01, V-05, and V-06 valves. By entering the hot gas to bed, the impurities desorbed from the bed and composition of purge gas were anlysed by

online GC. Vacuum regeneration of bed was performed by opening V-06, V-04 valves and closing V-05, V-07, and V-03 valves to desorbed/adsorbed impurities from bed by a rotary vacuum pump.
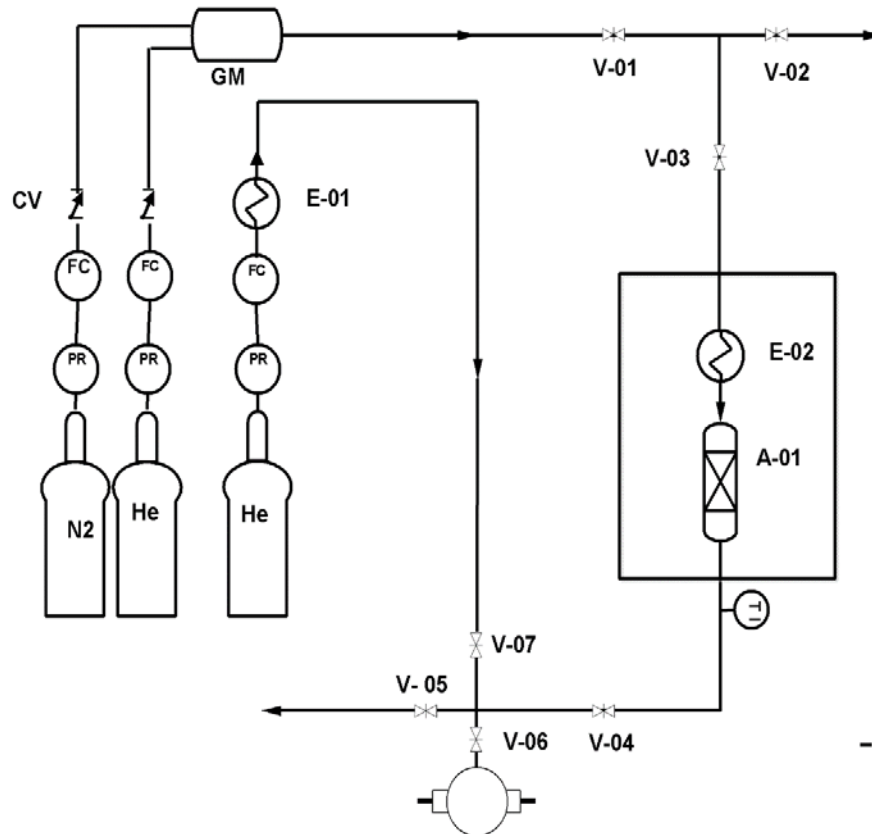
Figure 1. Schematic diagram of apparatus for a cryogenic adsorption process. (PR: pressure regulator, FC: mass flow controller, CV: check valve, GM: gas mixer, TI: temperature indicator, A-01: adsorbent column, E-01, electrical heater, and E-02, heat exchanger).

4. Result and discussion

4.1. Design validation using experimental data

The ability of the BET equation to calculate the required adsorbent mass was validated against experimental data by means of He/N_2 mixture (5 % N_2 in He). The experiments were performed at pressure of 8 bar and 1 slpm feed flow rate. In these conditions, the BET equation (equation 4) estimates V value (the amount of nitrogen that can be adsorbed on 235 gr activated carbon) equal to 42544.7 cc. Since the feed consists of 50 ml nitrogen

per minute, the adsorbent column should purify the feed gas mixture for about 851 min based on BET equation. Therefore, the experiments were carried out for 900 min. The results are shown in figure 2. As shown in the figure, the purity of helium increases from 95 % in the feed to about 99.97 % by crogenic adsorption (77 K and 8 bar) of nitrogen on activated carbon. As can be seen, in the figure, the adsorbent is not saturated by impurities after about 900 min. In addition, helium purity does not decrease during gas mixture purification. The obtained results

show that the BET equation is reliable and can be used to design an adsorber column in cryogenic adsorption systems.
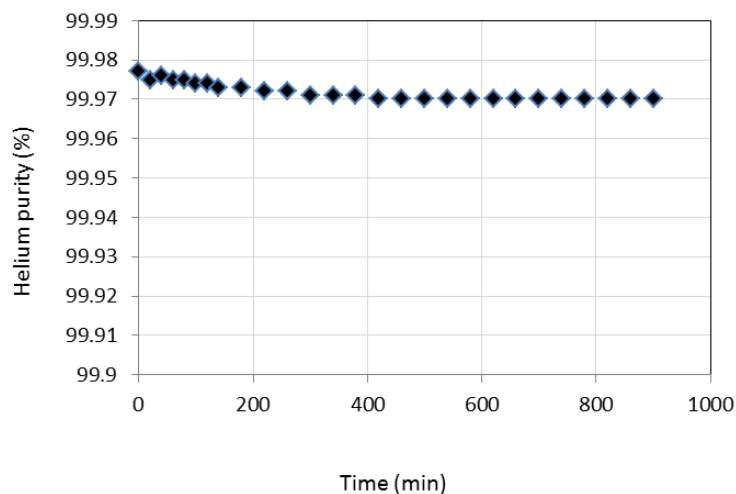

Figure 2. The change of helium purity after about 900 min passing of 5 % N_2 in helium from adsorbent column.

4.2. Effect of pressure on helium purity

Figure 3 shows the effect of pressure on helium purity. As shown in the figure, increase of feed pressure leads to an increase in helium purity, and maximum purity of helium (99.99999 %) is achived at 60 bar pressure.

Figures 4 and 5 show the effect of pressure on concentration of impurities in helium feed. As can be seen in the figures, concentation of impurities decreases with an increase in pressure. This phenomenon can be attributed to the accessible adsorbent cites and rate of the adsorbate striks on the adsorbent surface.

On the solid adsorbents, there are many cites for adsorption of gas molecules, and total porosity of adsorbent is usually classified into three groups: micropore, mesopore, and macropore [9]. As pressure increases, more sites in the adsorbent are accessible due to diffusion of gas molecules to micropores; therefore, greater amounts of impurities can be adsorbed. On the other hand, when the pressure is increased, adsorption of impurities increases due to an increase in the gas molecules striking on the accessible adsorbent surface, and more strikes result in more adsorption probability.
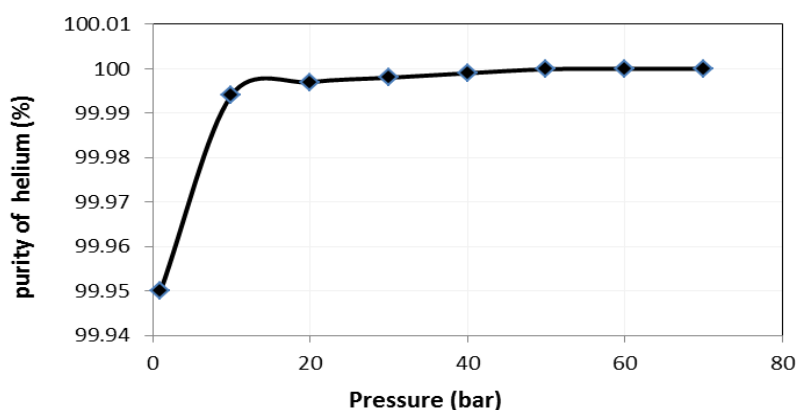
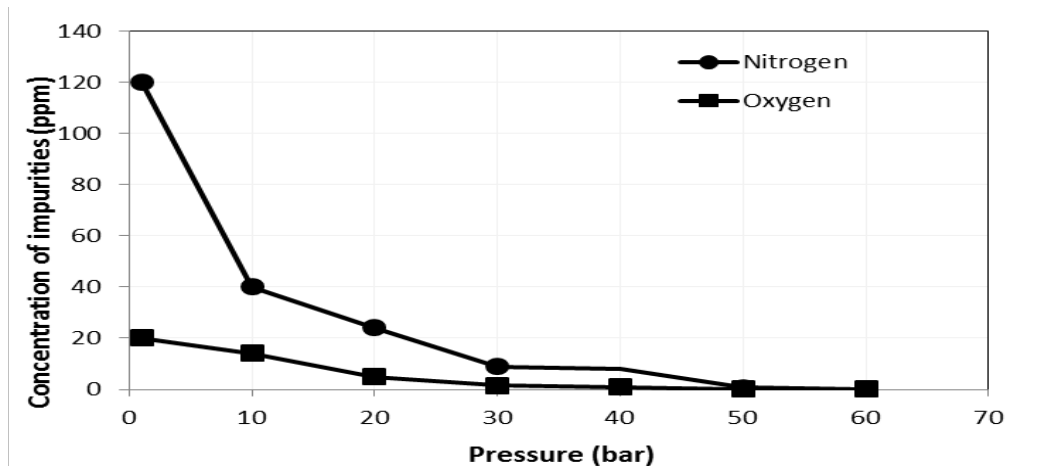**Figure 3.** Effect of pressure on helium purity.

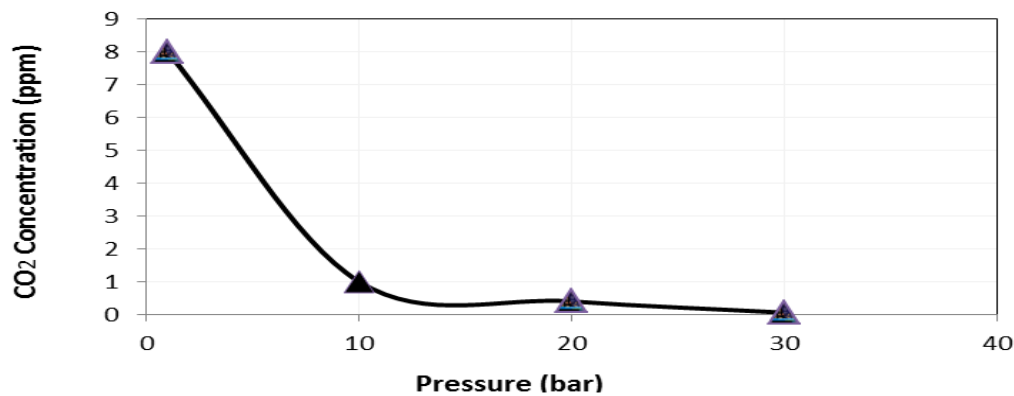**Figure 4.** Effect of pressure on nitrogen and oxygen concentration.

Figure 5. Effect of pressure on carbon dioxide concentration.

A comparison between figures 4 and 5 shows that removal of nitrogen and oxygen is more difficult compared with that of carbon dioxide. As shown in the figures, for removal of oxygen and nitrogen pressure of purification, unit should be increased till 60 bar, while carbon dioxide can be removed at pressure of 30 bar. This phenomenon can be attributed to boiling point of gases. Carbon dioxide has low boiling point (194.7 K); therefore, its molecules have low mobility at liquid nitrogen temperature (77 K) and gas molecules strike on the adsorbent surface result in more adsorption probability.

4.3. Regeneration of bed by helium as purge gas
After purification of feed (5 % N_2/He) for 15 hours, the adsorption bed was regenerated

using countercorent helium to feed direction as purge gas. Fig. 6 shows the effect of purge gas temperature on desorption time of nitrogen from adsorption bed which is saturated by 5 % N_2 in He at 8 bar. In this figure, the change of N_2 concentration to its initial concentration ($\frac{C}{C_0}$), desorbed from bed, is plotted versus desorption time at 40 °C and 180 °C purging gas temperatures. 1 slpm pure helium (99.95 %) was used as purge gas. As illustrated in the figure, desorption rate of N_2 using helium at 40 °C is slow and increases by increasing the temperature of the helium. This phenomenon can be attributed to the nature of desorption process. Because desorption is an endothermic process and increases with an increase in purge gas temperature, concentration of adsorbate (N_2)

decreases immediately with the introduction of the purge gas at 180 °C. As illustrated in the figure, a complete regeneration of the bed cannot be performed by helim stream at 40 °C, after 300 min, and about 30 % of N_2 remained in the activated carbon, while, by

the introduction of helium at 180 °C, complete regeneration of bed occurred after about 170 min. After this time, almost all the adsorbed N_2 was desorbed from activated carbon.
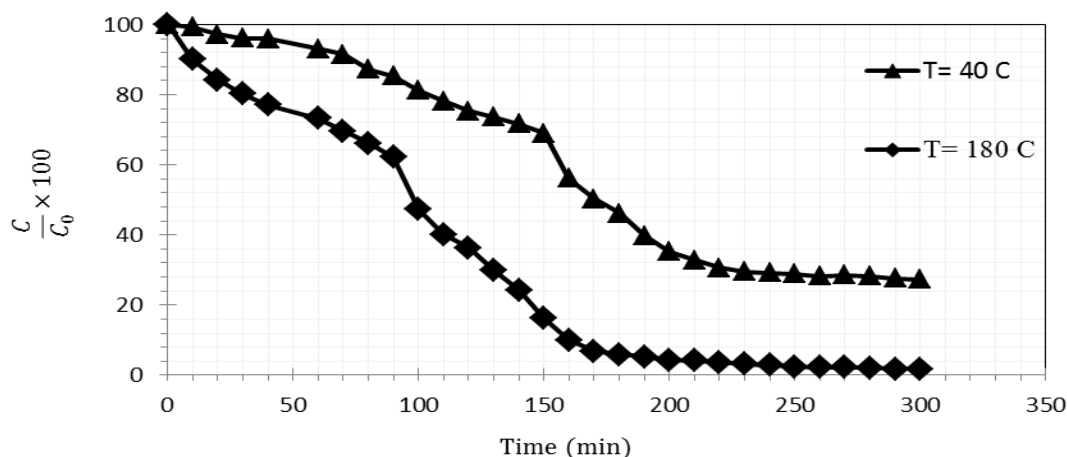

Figure 6. Effect of purge gas (helium) temperature on regeneration of adsorption bed.

4.4. Regeneration of bed by vacuum

Regeneration of bed by vacuum was performed after 15 hours purification of 5 % N_2 in helium. After purification step, LN_2 was removed from LN_2 bath and temperature of bed increased to ambient temperature by 180 °C helium stream. After that, evacuation was done via a vacuum pump for desorption of adsorbed nitrogen from activated carbon. To determine the remaining adsorbate (N_2) on activated carbon, the slow reverse stream of 180 °C pure helium (99.95 %) flowed through adsorbent bed, and composition of outlet helium was analysed after each 30 min evacuation of bed by online GC. Results showed that, after about 180 min of continuous evacuation of bed (at 10^{-3} torr pressure), almost no adsorbed nitrogen remained on activated carbon. Therefore, it can be concluded that the bed can be

regenerated by evacuation of impurities for about 180 min, and after this time the bed is cleaned for the next cycle helium purification.

4.5. Effect of bed regeneration condition on efficiency of helium purifier

The performance of helium purification process depends on helium purity and recovery. Energy requirement of process is usually proportional to the helium recovery. Helium purity in the maximum possible value is often the most desirable case for helium purification process in industry. So, the effect of regeneration condition on helium purity and recovery was investigated as two targeted dependent variables in helium purification process. Recovery of the cryogenic helium purification process was evaluated via equation (9) [21].

$$\text{Recovery} = \frac{(\text{product eluted from AD step} - \text{product used in PG step}) \times \text{product purity}}{\text{feed from PR and AD step} \times \text{composition of Helium in feed}} \quad (9)$$

For evaluation of purifier efficiency, purification of He/N_2 mixture (5 % N_2 in He)

is performed after each regeneration type of the bed, and the results are shown in table 7.

Table 7
Effect of regeneration condition on helium purity and recovery.

Regeneration type	Recovery (%)	Purity (%)
Helium (180 °C)	78.8	99.97
Helium (40° C)	74.2	99.34
Vacuum	98.2	99.97

As shown in the table, maximum helium purity is achieved when complete regeneration of bed occurs in regeneration step (regeneration by vacuum and hot helium stream). This phenomenon occurs due to compelete regeneration of bed from impurities in this step. As can be seen in the table, by vacuum regeneration of bed, helium recovery is in the maxium value because of low helium consumption in the regeneration step. Therefore, vacuum regeneration of bed can be proposed as the most appropriate bed regeneration method in helium purification system due to lower energy requirements and higher helium recovery (98 %).

5. Conclusions

In the present study, design and construction of a helium purification system was performed to purify 3 Nm^3/hr helium at 80 bar pressure. Cryogenic adsorption method was used for purification of helium using activated carbon at LN_2 temperature. Required mass of activated carbon for purification of helium was assumed using BET model. Accuracy of the model was confirmed by exprerimental data. Experimental results showed that an increase in pressure elimination of impurities increases. Purification of helium was performed in 10-70 bar pressure range and helium was purified from 99.95 % in feed to 99.99999 % at 60

bar. Regeneration of adsorber bed was performed by helium at two different temperatures (40 °C and 180 °C) countercurrent to feed direction. Results show that regeneration of activated carbon performs faster than helium at 40 °C and almost complete regeneration of bed occurs by helium stream at 180 °C after about 170 min. Effect of vacuum on desorption of impurities from bed was investigated using a vacuum rotary pump. Results show that, after about 180 min, desorption of impurities from bed occurs compeletly. In addition to selecting an optimum connditon, effect of regeneration condition on helium purity and recovery was investigated. Based on results, regeneration of bed by both vacuum and hot helium stream resulted in maximum helium purity; however, regeneration of bed by vacuum can be proposed as an appropriate condition in helium purification system due to higher helium recovery (98 %).

Nomenclature

V	volume of adsorbed gas [ ].
V_m	volume of monolayer adsorbed gas per unit mass of adsorbent [ ].
P_{sat}	saturation pressure [ ].
m_a	adsorbent mass [ ].
Q	feed flow rate [ ].
A	cross-section area [ ].
V	velocity of gas [ ].
ρ	apparent density [ ].
L	length of the adsorber column [ ].
Θ	adsorption energy [ ].
H	height of adsorber column [ ].
N	number of turns of a helical coiled column [ ].
p	spacing between consecutive coil turns [ ].
r	the radius of helix [ ].

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