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GAS COMPRESSION USING TEMPERATURE SWING ADSORPTION

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ABSTRACT

For a manned mission to Mars, NASA is interested in providing pure carbon dioxide at close to 1 bar to react with hydrogen to form methane and water. The Martian atmosphere is approximately 95% carbon dioxide, 2.7% nitrogen, 1.6% argon, and 0.7% other compounds at a pressure of about 0.008 bar. If the carbon dioxide is adsorbed at a low temperature and then the system is closed and heated, the pressure will increase because of both desorption and expansion of the gas. Usually the pressure increase due to desorption will be significantly larger than the gas expansion. By operating adsorption systems in series, significantly higher pressures can be obtained. Several systems were designed at a conceptual level for use on Mars as reliable alternatives to a mechanical compressor. Solar energy will be used for heating. Methods to remove most of the impurities during the compression process are explored.

Key Words: Compression; Mars; Temperature swing adsorption

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INTRODUCTION

A NASA problem on Mars focused our attention to develop methods to simultaneously compress and purify a gas using temperature swing adsorption. Although the initial application is on Mars, the approach is general and there are potential earth-based applications. The modeling done here is conceptual using idealized models.

A manned mission to Mars would require use of Martian resources to sustain life. The Martian atmosphere is approximately 95% carbon dioxide, 2.7% nitrogen, 1.6% argon, and 0.7% other gases including water and oxygen. Nominal nighttime ambient conditions at the Martian equator are about 0.008 atm and 200K. The carbon dioxide can be used as feed for the Sabatier reaction,

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$$

Electrolysis of the water will produce oxygen for human use and oxidizer for rocket fuel.

$$2H_2O \rightarrow 2H_2 + O_2$$

Hydrogen would be recycled to the Sabatier reaction. The net reaction after hydrogen recycle is,

$$CO_2 + 2H_2 \rightarrow CH_4 + O_2$$

Liquid hydrogen would be shipped from earth. The net result is that 48 kg of useful products (methane and oxygen) are produced on Mars for every 4 kg of hydrogen shipped to Mars. Since weight is a major concern, this is very useful for a long-term mission.

The separation challenge is to compress and purify the carbon dioxide to about 1 atm, 450K, and 99.7% CO_2 purity for the Sabatier reaction. These conditions represent a nominal starting point for design since optimum conditions for the reaction may be somewhat different. Since the station would probably be unmanned initially, it is desirable to have a very simple and robust method for compressing the gas. NASA wants to avoid use of a mechanical compressor on Mars because of concerns about reliability when operated for long periods at an unmanned station. One alternative NASA is interested in is adsorption compression. In adsorption compression, a gas is initially adsorbed at a cold temperature. The closed adsorption chamber is then heated, which in Mars will be done with solar energy. (This choice probably fixes the cycle time to a Martian day of 24 hr, 37 min, 23 sec.) The pressure of the chamber can increase markedly upon heating because of both the expansion of the gas and the desorption of gas from the adsorbent.

PROCESS CYCLES

Although this research was *not* supported by NASA, our curiosity was raised by the problems NASA will encounter on Mars. At the start, we were naïve and reinvented parts of the adsorption compression system, which was first proposed in 1978 and seriously studied for NASA.[1–3] However, being naïve has advantages since we also invented new cycles for adsorption compression and improved impurity removal methods. At the conceptual, exploratory level of this research, we assume that all steps occur at equilibrium and that the chambers are well mixed. Thus, the expected complications that arise because of finite mass and heat transfer rates are conveniently ignored. However, since the period of a solar-heated cycle will be one Martian day, the process may be reasonably close to equilibrium.

Carbon dioxide is adsorbed by both zeolite molecular sieves and activated carbon. The capacity of the zeolite adsorbent for carbon dioxide appears to be larger at low pressures, but activated carbon has a higher capacity at high pressures. At the delivery pressure chosen for the Sabatier reaction (1.07 atm) zeolites have a higher capacity for CO_2. Zeolites adsorb comparable amounts of argon and nitrogen although nitrogen is more strongly adsorbed.[4] Activated carbon adsorbs little argon, but adsorbs nitrogen in appreciable amounts. It appears that activated carbon will have a higher selectivity for carbon dioxide compared to argon and nitrogen than zeolites. Thus, the choice of the adsorbent is not obvious. NASA used zeolite 13X[2] while our calculations are based on activated carbon.

We will start by considering cycles for compressing a pure gas since these cycles are simple, help to explain the basic process, and may have applications on earth. However, these cycles are not satisfactory for compressing the atmosphere on Mars since the argon and nitrogen interfere with the operation of the cycle, particularly the vacuum driven capture of the carbon dioxide. After the introductory presentation, processes for multicomponent mixtures, which would be applicable on Mars are explored. These processes need to include the removal of the unwanted components.

Cycles for Pure Gas

Basic Cycle

Step 1. Open the adsorber and let it equilibrate with the Martian atmosphere (assumed to be pure carbon dioxide) at night. As adsorption occurs, the moles in the gas phase and the pressure in the adsorber decrease. Since this partial vacuum pulls in more gas from outside, this step can be done without a blower or compressor. During adsorption, the vessel can be either cooled (which will maximize adsorption) or be adiabatic (adsorber will heat up).

Step 2. Close the vessel and heat it externally (direct mode) to about 450K using solar energy as the heat source. This can be done with a heating fluid, by heating fins, electronically or with microwaves. As the temperature increases the pressure increases because of the thermal expansion of the gas and the increase in moles in the gas from desorption.

Step 3. Partially unload high pressure, hot gas as source of "compressed" gas. The gas can be discharged at high pressure or through a pressure control valve at constant pressure. Due to desorption, the system will cool during this step if heating is not continued.

Step 4. Depressurize and cool the system.

Repeat Cycle.

The carbon dioxide (species A) was assumed to be an ideal gas and adsorption was assumed to follow a Langmuir form with an Arrhenius temperature dependence for K and linear temperature dependence for $q_{\max}$,

$$q = \frac{q_{\max}(T)K_A(T)P}{1 + K_A(T)P}, \quad K_A = K_{A,O}e^{(-Q/RT)}, \quad q_{\max} = q_{\max,o} + aT \qquad (1)$$

The initial moles of gas in the tank after equilibration with the atmosphere are,

$$n_i = \frac{\varepsilon V P_{atm}}{RT_C} + \rho_{bulk} V \frac{q_{\max}(T_C)K_A(T_C)P_{atm}}{1 + K_A(T_C)P_{atm}} \qquad (2)$$

The first term on the right hand side of this equation is probably unimportant on Mars, but may be important for applications on earth. After heating, the moles of gas in the tank are unchanged, but the temperature and pressure change.

$$n_i = \frac{\varepsilon V P_{OH}}{RT_H} + \rho_{bulk} V \frac{q_{\max}(T_H)K_A(T_H)P_{OH}}{1 + K_A(T_H)P_{OH}} \qquad (3)$$

Solve for P_{OH}. After discharge of gas at the designer-chosen supply pressure, P_{supply}, the moles remaining are,

$$n_f = \frac{\varepsilon V P_{supply}}{RT_H} + \rho_{bulk} V \frac{q_{\max}(T_H)K_A(T_H)P_{supply}}{1 + K_A(T_H)P_{supply}} \qquad (4)$$

For simplicity, we assumed that $T = T_H$ although it will actually be cooler if delivery is done adiabatically. The amount of gas delivered per cycle is $n_i - n_f$.

Calculations were done for the adsorption of carbon dioxide on (Pittsburgh Chemical Co.) BPL activated carbon. Equilibrium data were from Ref. [5] and the properties of activated carbon were estimated from Ref. [6]. The range of the temperature cycle is from 200 to 450K. Since equilibrium data were available at 212.7, 260.2, and 301.4K, extrapolation of the data was required. The initial moles at 200K was $n_i = 0.469\,\mathrm{g\,mol/L}$. The pressure of the tank while hot was 10.03 atm. For a supply pressure of 1.07 atm the estimated delivery of carbon dioxide was 0.04 g CO_2 per gram adsorbent, which represents delivery of 86% of the initial moles of carbon dioxide in the tank. This prediction compares to the experimental value of 0.11 g CO_2 per gram 13X zeolite reported by Rapp.[2]

Staged Systems: Two-Tank Temperature Swing

Higher delivery pressures can be achieved with a staged system. In the simplest two-level system, two tanks containing adsorbent are loaded with gas at the cold temperature. Tank A is heated. The valve between the two tanks is opened, tank A discharges into B and the pressures equilibrate. Tank B will also heat up and tank A cools down, but temperatures equilibrate at a much slower rate than pressures because of the heat capacities of the adsorbent and tanks. After pressure equilibration but before temperature equilibration, the tanks are again isolated. The result has been a net transfer of gas from tank A to tank B. The pressure in B has increased. Next, both the tanks are cooled. Tank A is again opened to the atmosphere and equilibrated with atmospheric gas. Tank B remains closed. Tank A is then closed and heated, following which the valve between tanks A and B is again opened and gas transfers to tank B. As this is continued for multiple steps, the pressure in tank B will increase. Alternatively, a single large A tank can be used to charge a smaller tank B. More complex processes with additional B tanks or a third level (C tanks) have been developed.

The initial moles of gas in tanks A and B are given by Eq. (2) with the individual tank volumes V_A and V_B substituted for V, respectively. We will make the simplifying assumption that tanks A and B do not change temperature during the discharge and pressure equilibration steps. Then the moles of gas in each tank after discharge and pressure equilibration ($P_{A,i} = P_{B,i}$) is,

$$n_{A,i} = \frac{\varepsilon V_A P_{B,i}}{RT_H} + \rho_{\mathrm{bulk}} V_A \frac{q_{\max}(T_H) K_A(T_H) P_{B,i}}{1 + K_A(T_H) P_{B,i}} \tag{5a}$$

$$n_{B,i} = \frac{\varepsilon V_B P_{B,i}}{RT_C} + \rho_{\mathrm{bulk}} V_B \frac{q_{\max}(T_C) K_A(T_C) P_{B,i}}{1 + K_A(T_C) P_{B,i}} \tag{5b}$$

Since the pressure in tank B builds up over a number of cycles, the first term on the right-hand side of these equations will eventually become important. During pressure equilibration, the number of moles of carbon dioxide in the two-tank system is constant.

$$n_{A,0} + n_{B,i-1} = n_{A,i} + n_{B,i} \tag{6}$$

Combining Eqs. (5) and (6) results in a recursion relationship with the equilibration pressure $P_{B,i}$ as the unknown. Upon rearrangement, the result is a cubic equation for $P_{B,i}$ that can be solved algebraically.[7] The results in all of our simulations have consisted of two negative roots and one (physically realistic) positive root.

Results for a two level system cycling between 200 and 450K are shown in Figs. 1 and 2. The calculations were done for a pseudo-Martian atmosphere that was pure carbon dioxide. Figure 1 shows the pressure of tank B while cold and the moles of carbon dioxide in tank B for two tanks of equal size. Although the data points at each cycle are connected for visual enhancement, the increases occur as discrete jumps. At first, the adsorbent has a very high capacity for carbon dioxide and the pressure of the cold tank increases rather slowly. Then at about nine cycles, the pressure increases rapidly until saturation occurs. The number of moles in tank B first increases linearly and then levels off. To supply high-pressure gas, tank B would be heated. Its pressure will then increase markedly, and can be calculated by determining $n_{B,i}$ from $P_{B,i}$ and Eq. (5b), setting $n_f = n_{B,i}$ and solving Eq. (4) for P_{supply}. For example, after eight cycles the pressure in tank B is 0.491 atm (point 1) when the tank is at 200K. After heating tank B to 450K, its pressure is predicted to jump to 131 atm. Tank B does not have to be discharged completely when supplying high-pressure gas. If we cycle between points 1 and 2 while tank B is cold (eight and five cycles) gas can be delivered at

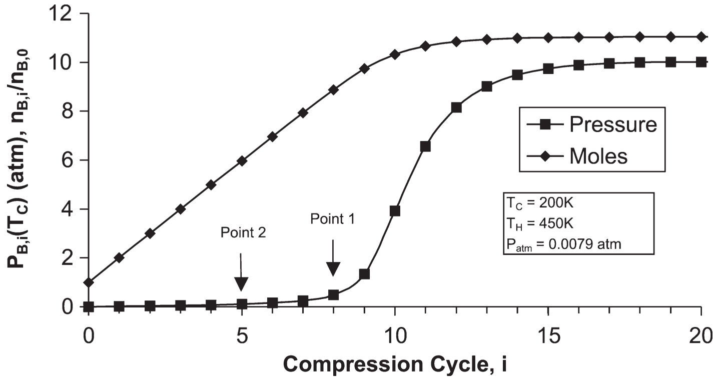

Figure 1. Compression cycles for two-level TSA.

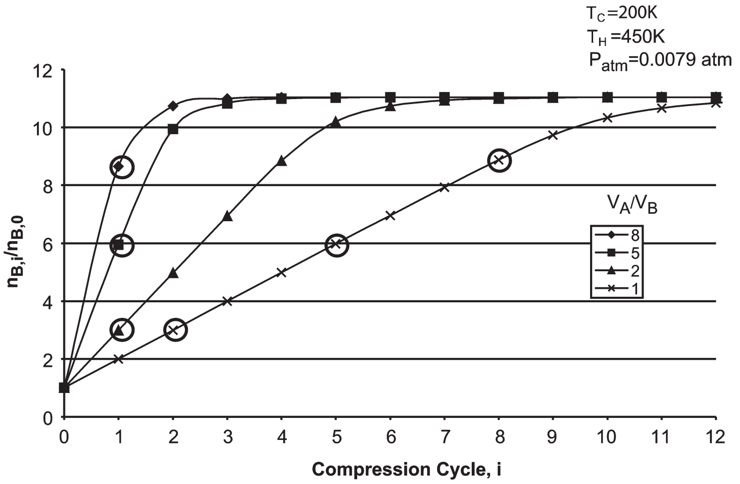

Figure 2. Compression cycles for various volume ratios of tank A to tank B.

any desired pressure up to 79.3 atm until tank B (while hot) has dropped to 79.3 atm. Cooling tank B while closed to 200K reduces the pressure to 0.103 atm (point 2). If the loading cycle is continued, only three steps are needed to return tank B to point 1.

Figure 2 shows the compression cycles for cases where tank A is larger than tank B. Comparing the curve for equal tank sizes ($V_A/V_B = 1$), we see that two cycles for equal size tanks produces only slightly more moles of gas than one cycle when $V_A/V_B = 2$. The same is true for four cycles of the equal sized tank compared to one cycle when $V_A/V_B = 4$, and eight cycles of the equal sized tank compared to one cycle when $V_A/V_B = 8$. The maximum moles of carbon dioxide are the same, but fewer cycles are required when a larger A tank is employed. Thus, in the previous example only one step would be needed to return from point 2 to point 1 if tank A were slightly larger than three times bigger than tank B.

The optimum tank sizes and number of tanks depend upon the adsorbent, the values of T_C and T_H, and the required supply pressure. In the linear range for the number of moles (see Fig. 1) the two-tank system will deliver almost the same number of moles as are input into tank A if we cycle between any two adjacent steps. For example, we could initially pressurize tank B with four steps and then cycle between steps 4 and 5.

It is interesting to compare the two-tank system with equal-sized tanks to the one-tank system for the set conditions (supply pressure = 1.07 atm, T_C = 200K, and T_H = 450K). The two-tank system can deliver almost 100% of the moles charged to tank A at this supply pressure vs. 86% for the one-tank system; however, since the two-tank system weighs twice as much, the efficiency per unit weight is approximately 50% compared to 86% for the one-tank system. Thus, the one-tank system is preferable to the two-tank system with equal-sized tanks. Using multiple tanks allows one to consider operation of the entire system at a much broader range of pressures and temperatures. If the Sabatier reaction was run at higher pressure or lower temperature, or the cold temperature was somewhat above 200K, the efficiency of the one-tank system would drop while (in the linear range) the efficiency of the two-tank system would remain constant. At some point, the two-tank process with equal-sized tanks will become preferable to the one-tank system. Using tanks of unequal size increases the efficiency per unit weight of the two-tank system. At a value of $V_A/V_B \sim 10$ its efficiency per unit weight matches that of the one-tank system for the current conditions of the Sabatier reaction.

If tank B remains at 200K during discharge of tank A and pressure equalization, carbon dioxide will start to condense at pressures of about 1.5 atm. In actual practice, tank B will heat up, but condensation still limits the pressures that can be achieved. However, condensation provides an additional method for storing carbon dioxide that can be delivered at high pressure by heating.

Cycles for Multicomponent Gas Mixtures

Since the atmosphere of Mars is not pure carbon dioxide, the presence of other gases affects the operation of the adsorber and of the Sabatier reaction. The presence of inerts in the adsorber interferes with the vacuum driven capture of pure carbon dioxide that allowed the previous cycles to operate without a fan or blower. In addition, the product carbon dioxide will not be pure. Pure carbon dioxide is desirable in the reactor since inerts can greatly decrease the reaction rate. We are also interested in studying the separation of carbon dioxide from the inerts since there are probably uses for recovered inerts on Mars.

First, we will assume that the other components are nonadsorbed inerts. If we operate without a fan or blower, there will be flow of gas into the tank, but no flow out. The nonadsorbed inert components that enter the tank will stay in the gas phase while carbon dioxide is absorbed and the concentration of inerts will increase. We can calculate the total moles of all gases in the tank

$$n_i = \frac{P_{atm}\varepsilon V}{RT_C} + \rho_{bulk} V \frac{q_{max}(T_C)K_A(T_C)y_0 P_{atm}}{1 + K_A(T_C)y_0 P_{atm}} \quad (7)$$

The moles of carbon dioxide can be calculated by multiplying the gas phase term by the carbon dioxide mole fraction y_0. Since everything that enters the tank stays there, the net mole fraction of carbon dioxide will be the same as in the atmosphere,

$$z_0 = \frac{n_{\mathrm{CO_2},i}}{n_i} \tag{8}$$

where z_0 is the carbon dioxide mole fraction in the atmosphere (0.95). Substituting in the equations for moles of carbon dioxide and total moles we obtain,

$$\begin{aligned} &z_0 \frac{P_{\mathrm{atm}} \varepsilon V}{R T_{\mathrm{C}}} + z_0 \rho_{\mathrm{bulk}} V \frac{q_{\max}(T_{\mathrm{C}}) K_{\mathrm{A}}(T_{\mathrm{C}}) y_0 P_{\mathrm{atm}}}{1 + K_{\mathrm{A}}(T_{\mathrm{C}}) y_0 P_{\mathrm{atm}}} \\ &\quad = \frac{y_0 P_{\mathrm{atm}} \varepsilon V}{R T_{\mathrm{C}}} + \rho_{\mathrm{bulk}} V \frac{q_{\max}(T_{\mathrm{C}}) K_{\mathrm{A}}(T_{\mathrm{C}}) y_0 P_{\mathrm{atm}}}{1 + K_{\mathrm{A}}(T_{\mathrm{C}}) y_0 P_{\mathrm{atm}}} \end{aligned} \tag{9}$$

This equation can be solved for y_0, the mole fraction of carbon dioxide in the gas after equilibration. Substitution of y_0 into Eq. (7) gives the value of n_{i}. If there are inerts present, the carbon dioxide mole fraction, y_0, will be smaller which will make n_{i} smaller.

When the tank is heated to T_{H}, the moles of inert in the gas phase and the total moles of gas in the tank are unchanged. After some manipulations, the total moles in the tank after heating is,

$$\begin{aligned} n_{\mathrm{i}} = {}& \frac{P_{\mathrm{OH}} \varepsilon V}{R T_{\mathrm{H}}} \\ & + \rho_{\mathrm{bulk}} V \frac{q_{\max}(T_{\mathrm{H}}) K_{\mathrm{A}}(T_{\mathrm{H}})(P_{\mathrm{OH}} - (1 - y_0) P_{\mathrm{atm}}(T_{\mathrm{H}}/T_{\mathrm{C}}))}{1 + K_{\mathrm{A}}(T_{\mathrm{H}})(P_{\mathrm{OH}} - (1 - y_0) P_{\mathrm{atm}}(T_{\mathrm{H}}/T_{\mathrm{C}}))} \end{aligned} \tag{10}$$

Only P_{OH} is unknown and can be solved for. This pressure is increased at higher temperatures since $q_{\max}$ and K_{A} are both smaller. The presence of inerts will limit the pressure that can be achieved by heating since less gas, n_{i}, is available to be desorbed.

If a fan is placed at the input and the external gas is blown through the tank, the exiting gas will be concentrated in nonadsorbed inerts. When the adsorbent becomes saturated, the exit gas and the gas in the void spaces will approach the concentration of the external atmosphere, $y_0 = z_0$, instead of being concentrated in nonadsorbed inerts. Thus, the mole fraction of carbon dioxide y_0 in the cold tank is known and n_{i} can be determined directly from Eq. (7). After the loading step, we would close and heat the tank. Equation (10) can be used to determine P_{OH}. Since the inerts remain in the gas, we can easily calculate the inert mole fraction in the gas as,

$$y_{\mathrm{inert}} = (1 - y_0) = (1 - z_0)(P_{\mathrm{atm}}/P_0)(T_{\mathrm{C}}/T_{\mathrm{H}}) \tag{11}$$

The product gas will be concentrated in carbon dioxide and diluted in nonadsorbed inerts.

If adsorption is strong these concentration and adsorption effects for inerts can be marked. After loading with the fan, $y_0 = z_0 = 0.95$. If the adsorbent has the same properties as activated carbon but does not adsorb the inerts, the pressure of the tank after heating would be 9.55 atm. The presence of inerts in the hot gas leaving the tank would decrease from an average of 5 to 0.0019% when loading with a fan. Since argon and particularly nitrogen adsorb, the actual separation will be less dramatic.

Further purification of gas (beyond that obtained during the loading step) can be obtained by doing a partial internal purge after tank B is fully pressurized and heated.[8] Before supplying gas at P_{supply}, partially open the valve and allow hot gas to escape and the pressure to drop. The first gas to escape will be most concentrated in nonadsorbing inerts and weakly adsorbed impurities. Some carbon dioxide will be used to purge the inerts and impurities. The price for producing a purer carbon dioxide is fewer moles of gas will be available for delivery at the supply pressure. Since calculations are similar to pressure swing adsorption calculations,[6] they are straightforward if one assumes that the inerts do not adsorb and that adsorption of the carbon dioxide is linear. If the linear isotherm is,

$$q \text{ (moles CO}_2\text{/kg adsorbent)} = A(T)yP \tag{12}$$

then the solution is,

$$P_{\text{f}} = P_{\text{OH}} \left(\frac{y_{\text{OH}}}{y_{\text{f}}}\right)^{(1+(\gamma/\alpha))} \left(\frac{1 - y_{\text{f}}}{1 - y_{\text{OH}}}\right)^{(\gamma/\alpha)} \tag{13}$$

where

$$\gamma = \varepsilon V/RT_{\text{H}}, \quad \alpha = \rho_{\text{bulk}} V_{\text{A}}(T_{\text{H}}) \tag{14}$$

The designer can pick the desired final pressure P_{f} and solve for the final mole fraction of carbon dioxide y_{f} after the partial purge. Very high purity gas can be produced. Figure 3 shows the resulting mole fraction of carbon dioxide in the waste discharge of the tank for both the linear solution and for a stepwise numerical integration with the Langmuir isotherm. The tank starts at the lower right in Fig. 3 and moves toward the upper left. When the discharge is pure enough, the purge can be stopped and product withdrawal started. A fan was used for the loading step and all of the inerts were assumed not to adsorb.

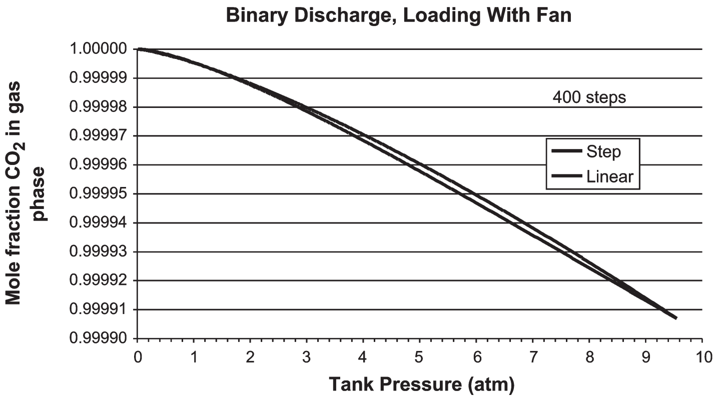

Figure 3. Discharge of a tank at 450K for a binary system in which only CO_2 adsorbs. The linear curve is above the step curve.

DISCUSSION

The presence of a fan introduces moving parts, which may be undesirable in remote locations such as Mars. The two major advantages of using a fan are it speeds up the loading step considerably and it results in significant removal of inerts. If the cycle is fixed at the Martian day, then speed is not important. Another option for obtaining higher carbon dioxide purities, which has not been studied in detail, is to use a two-stage system without a fan during the loading of the first stages. During the pressure equalization, (loading of the second stage) adjust the outlet valve so that there is a continuous small withdrawal of cold gas from the tank. This is equivalent to using a fan during this step. The concentration of inerts (adsorbing and nonadsorbing) will decrease, but at the price of losing some of the pressurized carbon dioxide from the first stage.

The calculations in this research were done for the nominal conditions of the Sabatier process. An alternative is to do CO_2 electrolysis to produce CO and H_2O. This process will also benefit from pure, pressurized CO_2.

Although the original design was made for applications on Mars, there may be applications of gas compression using temperature swing adsorption on earth. For example, at remote locations one could adsorb nitrogen from air using a zeolite molecular sieve. Upon heating the closed system the gas pressure will increase. The pressurized gas could be used for pumping water, operation of pneumatic controllers, inflating tires, providing concentrated nitrogen or oxygen, and so forth. Since the starting pressure is much higher than on Mars, the saturation limits of the adsorbent become important much earlier. However, heating and cooling may be much simpler on earth since solar ponds can be used for the heat source and ambient air or ground water as the cooling source.

This conceptual research ignored heat and mass transfer rates and the adsorption of inerts that will undoubtedly be important in practice. Temperature will not be constant during the loading and desorption steps. In practice, the tanks will not be well mixed and there will be concentration and temperature waves during all steps. At least some of the inerts such as nitrogen will adsorb and will compete for sites with carbon dioxide. Additional research is needed to include these effects. The next step for evaluating the merits of the design ideas presented here would be to simulate the processes on simulation software without these limiting assumptions. Detailed simulations will also allow one to explore various partial purge options.

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