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Heating-Rate-Coupled Model for Hydrogen Reduction of JSC–1A

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Abstract

A previously developed and validated model for hydrogen reduction of JSC-1A for a constant reaction-bed temperature is extended to account for reaction during the bed heat-up period. A quasi-steady approximation is used wherein an expression is derived for a single average temperature of reaction during the heat-up process by employing an Arrhenius expression for regolith conversion. Subsequently, the regolith conversion during the heat-up period is obtained by using this representative temperature. Accounting for the reaction during heat-up provides a better estimate of the reaction time needed at the desired regolith-bed operating temperature. Implications for the efficiency of the process, as measured by the energy required per unit mass of oxygen produced, are also indicated.

Introduction

A major objective of lunar in-situ resource utilization (ISRU) (Refs. 1 to 3) is production of oxygen from lunar regolith. Mare basalts containing oxygen include olivine, ilmenite, and pyroxene (Ref. 4). Energy has to be supplied to remove the oxygen, i.e., the associated chemical reactions are endothermic. Free energy considerations show that the iron containing minerals require the least energy input for this purpose (Ref. 5). Various technologies have been proposed for the chemical conversion. These include ilmenite reduction by hydrogen, molten electrolysis, and carbothermal reduction using methane (Refs. 6 and 7). The reduction processes involve gas-solid reactions. Such systems are well developed for earth applications but mainly for the purpose of metal extraction, as opposed to water vapor and oxygen extraction for lunar applications. In addition, in the lunar context, the reactor must interface with the other sub-system processes such as upstream regolith extraction and beneficiation and downstream electrolysis and phase separation in a way that establishes the most favorable balance between efficiency, robustness, and equivalent system mass. A need exists, therefore, to establish an analytical framework to couple these sub-systems together. The building blocks of this framework are the individual sub-system models. The development of a generalized analytical model of the chemical reactor sub-system has been discussed in an earlier paper and the model has been validated against experimental data from reduction of JSC-1A, a lunar regolith simulant (Ref. 8).

The model is formulated in terms of the following overall steps: (i) inflow of pure hydrogen into the regolith containing reactor, (ii) reaction of hydrogen with the iron oxide portion of the regolith, and (iii) outflow of left-over hydrogen and produced water vapor. Step (ii) is the gas-solid reaction step which is the key to the conversion. Results of regolith particulate analysis conducted on Apollo-returned samples (Ref. 6) suggest that the particles are not completely solid but have some porosity that would enable gases to diffuse into the particles, i.e., the iron oxide portion can be essentially completely converted with the reduced iron remaining behind in the particulates. It may also be noted that JSC-1A has significant glass content with iron oxide dispersed in the glass phase. The diffusion of gases in the glass phase is expected to be the controlling feature for the reduction process. These observations support utilization of a

"shrinking core" diffusion-reaction model (Ref. 9) for the gas-solid reduction process. This model is then coupled with the inflow/outflow of gases to complete the reactor sub-system model.

Two key parameters appear in the model: (i) a diffusion time related to the diffusion of gases in the interior of the regolith particles, and (ii) a residence time of the gases in the reactor. It is found that for high levels of conversion or for a relatively small ratio of residence time to diffusion time, the governing parameter is the diffusion time. Validation of the model was conducted by comparison to experiments carried out at Pacific Northwest National Laboratory (PNNL) (Ref. 10) and at Lockheed Martin (Ref. 11). A factor *F*, which is related to the diffusion times for different particle sizes, is derived from the model formulation and is defined as follows:

$$F = 6\frac{c_0}{\rho_a}\frac{k}{(1+k)}\frac{D}{r_p^2} \tag{1}$$

Using the PNNL data for JSC-1A in conjunction with the model, the factor *F*- is correlated to the temperature by an Arrhenius expression:

$$\log(F) = -\frac{10761}{T} - 2.8808 \tag{2}$$

The above expression appears to work well in the temperature range of 873 to 1273 K, i.e., in between 600 and 1000 °C.

It may be noted that the model assumes a constant operating temperature during the reduction process. This implies that it considers a situation where the regolith is brought up to the desired operating temperature prior to initiating the hydrogen flow and the reduction process. In order to extend the applicability of the model, it is useful to incorporate reaction during the regolith heat-up stage as well. In fact, current configurations being tested by NASA envision hydrogen flow through the reactor and associated reduction during heat-up. If significant reduction takes place during the heat-up then it may be possible to increase the energy efficiency of the process, i.e., the amount of water (or oxygen) produced per unit energy input.

In the next section, the methodology to incorporate reaction during heat-up is described. The Results and Discussion section first presents comparison of the model with experimental results provided by NASA Johnson Space Center (JSC). Then, the model is exercised to present the effects of heat-up time and operating temperature of the regolith bed on the water production. Finally, theoretical results on impact of reduction during heat-up on the energy efficiency of oxygen production in the reactor are described in the Results and Discussion section.

Nomenclature

c_0	molar concentration of hydrogen at inlet to regolith bed (moles/m^3)
D	effective intra-particle gas diffusion coefficient (m^2/s)
k	equilibrium constant
L	length of regolith bed (m)
r	regolith particle radial coordinate (m)
r_p	regolith particle radius (m)
t	time (s)
t_D	intra-particle diffusion time (s)
t_R	reactor residence time (s)
T	temperature (K)
T_h	heater temperature (K)
T_i	minimum temperature for reduction (K)

T_r final operating temperature (K)
$< T >$ average temperature during heat-up (K)
u velocity through reactor (m/s)
α particle conversion fraction
ε void fraction in regolith bed
ρ_a molar concentration of iron oxide in regolith (mol/m^3)
τ time constant for regolith-bed heat-up (s)

Model Description

The shrinking core formalism utilized in the model is schematically depicted in Figure 1. Conversion proceeds from the exterior particle surface inward. As the reaction progresses into the interior of the particle, the reactant and product gases must diffuse through the particle matrix structure formed after the conversion.

The equation for the conversion fraction α (related to the fraction of iron oxide converted) at a given time, t, is given by (Ref. 8)

$$3[1-\alpha]^{2/3} - 2[1-\alpha] - 1 = -\frac{Ft}{\left[1 + 3(1-\varepsilon)\left(\frac{D}{r_p^2}\right)\frac{[1-\alpha]^{1/3}}{[1-(1-\alpha)^{1/3}]}\frac{L}{u}\right]} \tag{3}$$

where the factor F is given by Equation (1). For the case when the residence time, L/u, of the gases is small compared to the diffusion time, r_p^2/D, of gases in the particle matrix the equation for α becomes (Ref. 8)

$$3[1-\alpha]^{2/3} - 2[1-\alpha] - 1 = -Ft \tag{4}$$

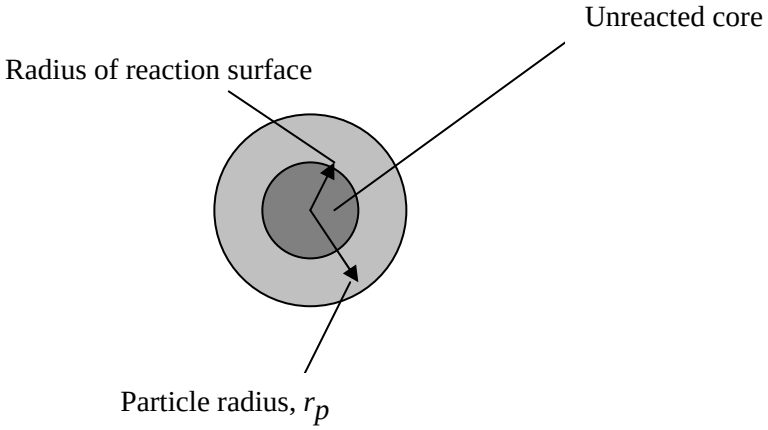


Figure 1.—Shrinking core model configuration.

As noted earlier, the model assumes a fixed operating temperature. In order to account for reaction during heat- up, it may be assumed that the conversion during the heat-up process is the summation of a sequence of quasi-steady reduction steps with increasing temperature. This is valid as long as the particle is assumed to be at a uniform temperature at each sequential step as far as the gas diffusion process is considered, i.e., the intra-particle thermal conductivity is much larger than the intra-particle gas diffusivity, D, which appears to be the case. Equation (4) indicates that during each of these sequential steps the rate of conversion, $d\alpha/dt$, is proportional to the F corresponding to the sequential temperature. An average rate of conversion during the heat-up period may then be defined by using an appropriate average for F that may be utilized in Equation (4).

Let the heat-up begin at time t =0. Experimentally, it has been found that there is no discernible reduction until the regolith (simulant) temperature reaches a certain value. For JSC-1A this temperature is in the range 873 to 923 K (i.e., 600 to 650 °C). Let, in general, this minimum temperature for reduction be denoted as T_i and the corresponding time for the regolith to reach this temperature be t_i. The reduction period is then given by $(t–t_i)$.

In previous work it was shown that, for a temperature T_h of a coaxial heater, the time needed to raise the regolith bed from a temperature T_i to T is given by (Ref. 12)

$$t/\tau = -\log\left(\frac{T_h - T}{T_h - T_i}\right) \tag{5}$$

where the time constant τ depends, among other quantities, on the mass and specific heat of the regolith. When the heater temperature is raised in steps between the initial and final temperatures, as is generally the case, the regolith- bed temperature rise is almost linear in time as shown in an example scenario in Figure 2 in non-dimensional form.

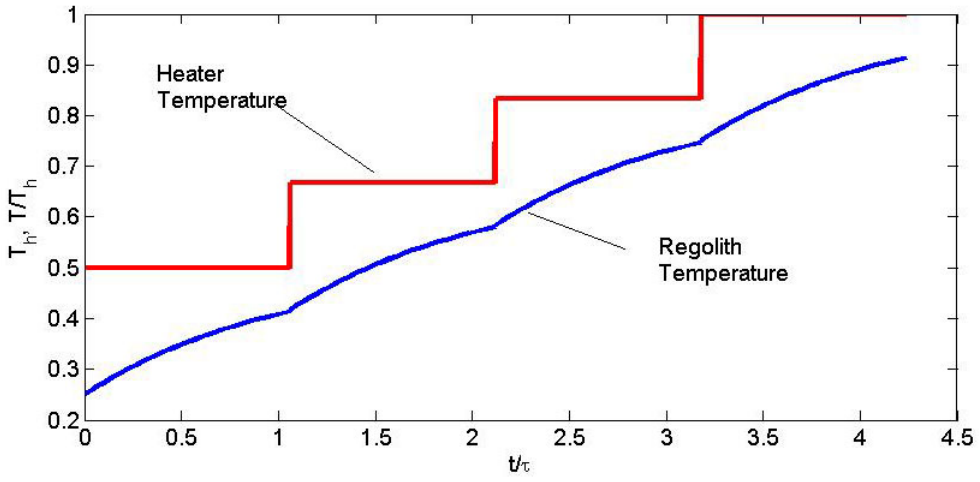


Figure 2.—Heater temperature profile and corresponding calculated regolith bed temperature.

The discussion above indicates that the time-average value of F or log(F) may be obtained by simply averaging it over the temperature range T_i to T_r. The average for log(F) becomes

$$< \log(F) >= -\frac{10761}{(T_r - T_i)} \int_{Ti}^{Tr} \frac{1}{T} dT - 2.8808 \tag{6}$$

so that

$$< \log(F) >= -\frac{10761\left[\log\left(\frac{T_r}{T_i} \right) \right]}{(T_r - T_i)} - 2.8808 \tag{7}$$

Comparing Equations (2) and (7) yields an average temperature for log(F) during the heat-up period

$$< T >= \frac{T_r - T_i}{\log\left(\frac{T_r}{T_i} \right)} \tag{8}$$

With the approximation that log<F> is roughly equal to <log(F)> in the temperature range of interest, the same value of the average temperature may be utilized for F.

Figure 3 plots < T > versus T_r for JSC-1A. It is seen that the variation of < T > in the temperature range of interest is essentially linear. For cases where the residence time is comparable to the diffusion time, Equation (1) may be first used to obtain the appropriate average value of the diffusion coefficient D from the averaged value of F with the equilibrium constant, k, being evaluated at < T >. Then, Equation (3), which incorporates the ratio of the residence and diffusion times, may be utilized.

It may be noted that while the analysis herein has been carried out for JSC-1A, a similar methodology may be employed for other simulants whose reduction is described by the shrinking core model with an Arrhenius expression for F.

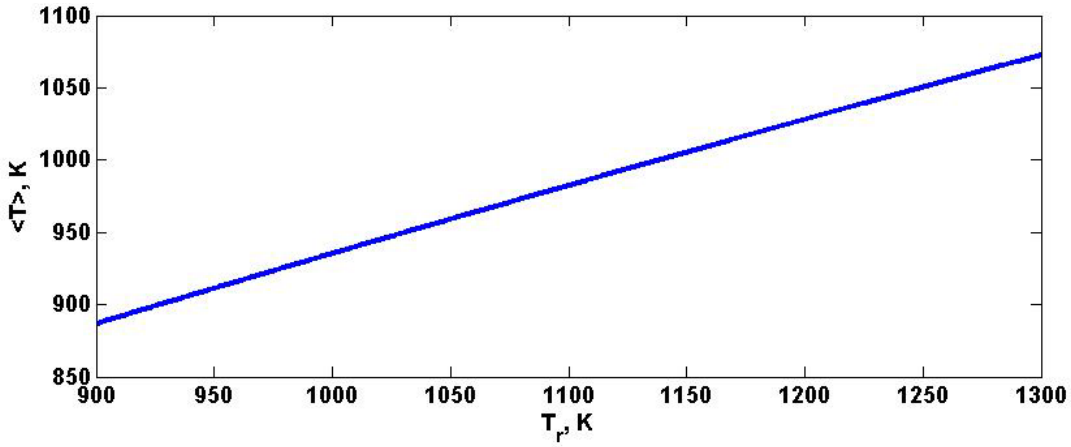


Figure 3.—Average value of temperature during heat-up as a function of the final operating temperature.

Results and Discussion

As noted earlier, the constant-temperature reduction model (i.e., not including reaction during heat-up) has been validated against experiments. In these experiments, physically bound water was first baked off at high temperatures (above 600 °C) in the presence of an inert gas (e.g., argon or helium) until the detectable humidity at the reactor outlet was negligible. Subsequently, the simulant was brought up to the operating temperature in the presence of the inert gas by raising the heater temperature. The time taken for this step also helped assure that the simulant bed was close to the heater temperature. At this point, the hydrogen flow was initiated. In this manner, the water production due to hydrogen reduction at the operating temperature was unambiguously determined. It would be useful to have similar validation experiments for reaction during heat-up where all of the bound water is baked off prior to initiating the hydrogen flow and the simulant temperature is well characterized. Because of schedule constraints in hardware development, these tests have not been conducted. However, some test results are available where hydrogen flow is present during the bound water bake-off and simulant-bed heat-up process. First, the model results will be compared to this data to assess the model applicability.

The experiment was conducted at NASA JSC in a cylindrical laboratory reactor of inner diameter 0.021 m with a JSC-1A batch size of 10 grams. Based upon non-reacting tests with JSC-1A in the same reactor, it is estimated that the bound water amount for this sample size is about 0.04 grams. By the time, the heater reached the 650 °C mark, measurements showed that about 0.02 grams of bound water had been removed (ignoring the small amount of water that would be produced by hydrogen reduction in this lower temperature regime) leaving an estimated 0.02 grams of bound water to come out during the hydrogen reduction process in addition to that formed by reaction. The heater profile for this test run with the final operating temperature of 1080 K (approximately 807 °C) is plotted in Figure 4 (only the portion beyond 650 C is shown which is considered here). The experimental values of the water out, expressed as a conversion fraction are shown in Figure 5. Two curves are shown: one (red), where the bound water is included in the conversion fraction, and the other (green), where the bound water is subtracted out from the measured water in a linear fashion with time over the duration of the experiment. Thus, at the end of the experiment, the two experimental curves differ by the 0.02 grams of estimated bound water which translates to a difference of approximately 0.07 in the conversion fraction.

The model prediction is also shown in Figure 5. In general, the model prediction is between the two data curves with and without the estimated bound water removal. There is some overprediction of the water out at short times, i.e., at lower temperatures during the heat-up process which may be due to $< T >$ being higher than the initial temperature. However, it appears that the cumulative water out, i.e., the conversion fraction, at the end of the heat-up period is reasonably well represented by the model.

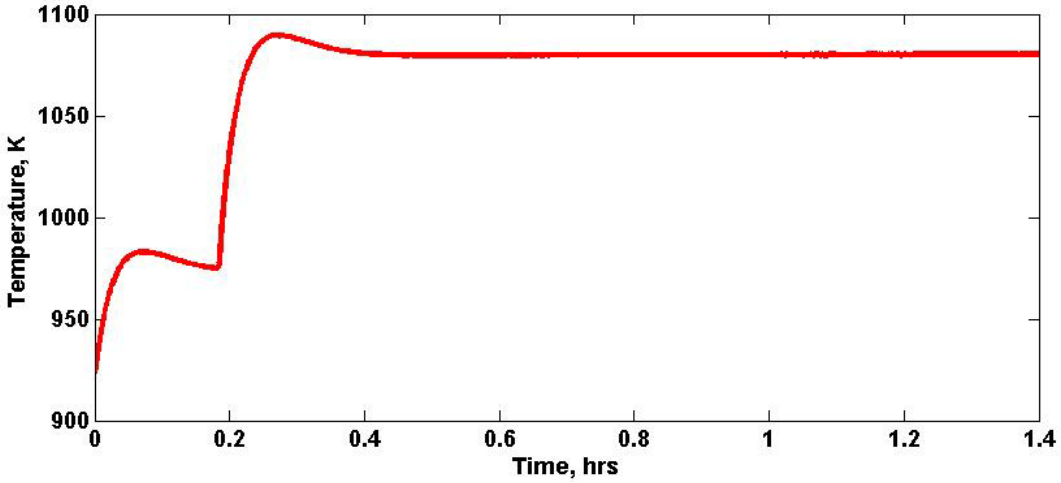


Figure 4.—Time profile of heater temperature from experiment.

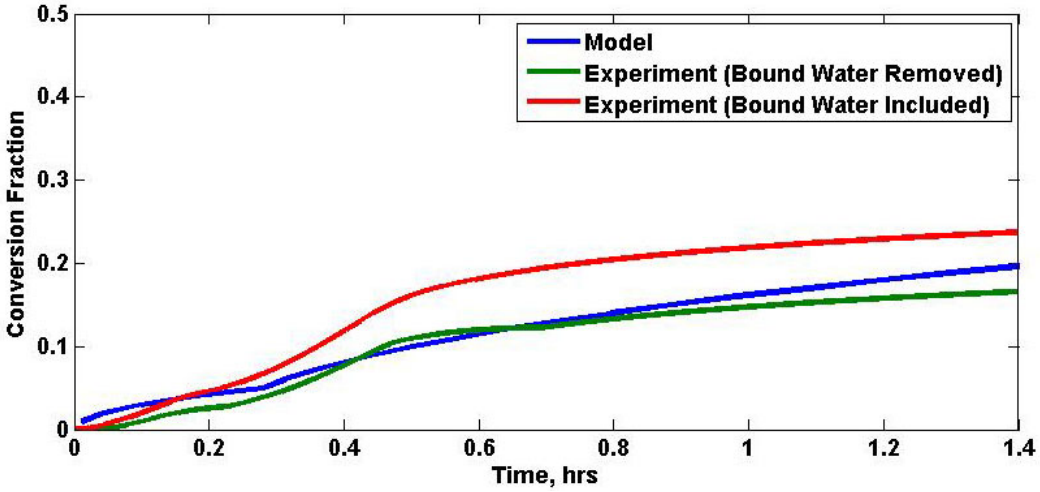


Figure 5.—Experimental and predicted values of conversion fraction for the heater temperature profile of Figure 4.

Within the restrictions of the model, the conversion fraction during heat-up depends upon the heat-up time and the final steady-state operating temperature. The effects of both of these factors are considered next using predictions from the model. Figure 6 plots the conversion fraction as a function of heat-up time to a final operating temperature of 1273 K (i.e., 1000 °C). In order to make the discussion general, it is assumed that the reactions are not constrained by hydrogen availability; this allows the use of Eq. (4) without detailed knowledge of the reactor geometry and simulant batch size with results being presented in terms of conversion fraction. The heat-up time here is measured from the stage where the regolith temperature reaches 873 K which, as previously noted, is close to the temperature where measurable reduction begins to occur. As may be expected, as the heat-up time increases, the conversion fraction also increases, although the correspondence is not linear. Thus, at shorter heat-up times, the rate of conversion, given by the slope of the curve, is faster. This occurs because during the initial stage of conversion the outer layers of the regolith particles are being reduced so that the effects of slower intra-particle diffusion are limited. However, once these outer layers are reduced, intra-particle diffusion effects become more important and the rate of conversion is reduced.

The effect of the final operating temperature on the conversion fraction during heat-up is shown in Figure 7 for a fixed heat-up time of two hours. As before, the heat-up time is measured after the initial reduction temperature of 873 K is reached. In contrast to the non-linear dependence on heat-up time, the dependence on the final operating temperature is nearly linear. Thus, Figures 6 and 7 together show that the conversion fraction during heat-up may be increased by both increasing the heat-up time as well as by increasing the final operating temperature.

If heat losses from the reactor are well-controlled, then the energy required to bring the regolith to the operating temperature depends mainly on the mass of regolith and its specific heat. If these factors are fixed, then increasing the heat-up time reduces the power requirement for the fixed total energy so that if power is a constraint, then increasing the heat-up time may be a viable option. However, an influencing factor is the amount of oxygen that needs to be produced in a given time. As Figure 6 shows there is a diminishing rate of conversion with increase in heat- up time which would prohibit arbitrary increase in the heat-up time. On the other hand, while the conversion fraction for a fixed heat-up time may be increased by increasing the final operating temperature, as shown in Figure 7, this increases the overall energy and power requirements.

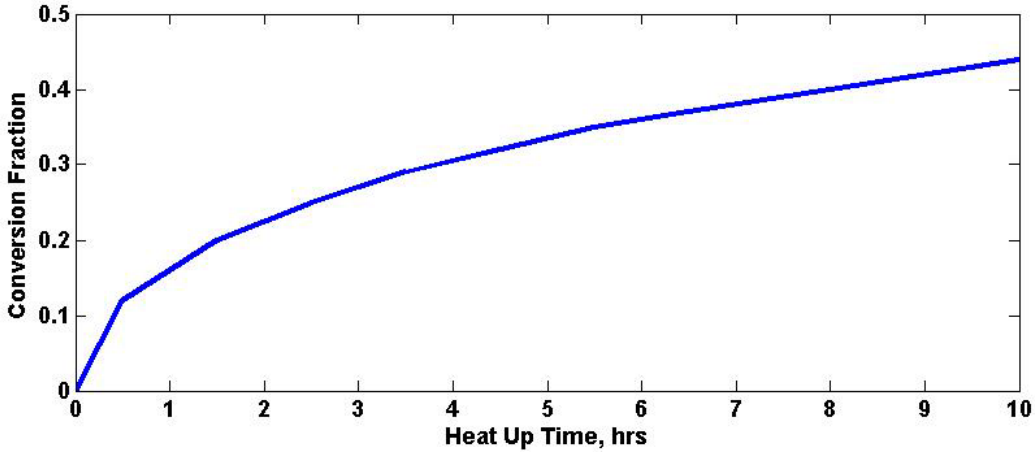


Figure 6.—Conversion fraction as a function of heat-up time for a final operating temperature of 1273 K.

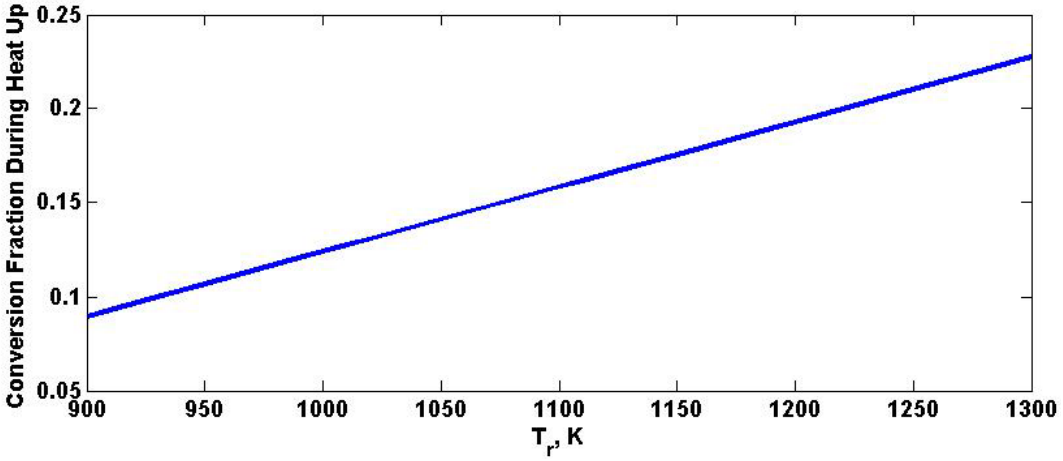


Figure 7.—Conversion fraction during heat-up as a function of final operating temperature. The heat-up time is 2 hr.

The manner in which reaction during heat-up impacts the overall conversion is shown in Figure 8. The figure plots conversion fraction as a function of reaction time at the operating temperature (1273 K) for three cases: with no reaction during heat-up, one-hour reaction during heat-up, and two-hour reaction during heat-up. For the case with no reaction during heat-up, the conversion fraction starts at zero at time zero and then increases as shown in previous work. Initially, the conversion rate is high as the outer layers of the particles are reduced and then the conversion rate drops as intra-particle diffusion becomes important. For the one-hour and two-hour reaction times during heat-up, a finite conversion has taken place by the time the operating temperature is reached. However, by this time, the conversion rate has reduced since reduction is now taking place in the interior of the particles. As a result, after one to 2 hr of conversion at the operating temperature, the conversion fractions for all three cases are very close to each other.

It has been proposed to utilize the energy efficiency of the process, defined as the cumulative amount of oxygen produced per unit energy input, as a tool for evaluating the performance of a given reactor configuration and operation timeline (Ref. 13). The cumulative amount of oxygen produced may be calculated from the conversion fraction; for JSC-1A, a conversion fraction of unity implies production of 2.8 percent water by weight, or equivalently, 2.49 percent oxygen by weight. The cumulative energy input is given by the sum of the energy needed to heat-up the regolith and the heat losses. An example set of calculations for the ratio of oxygen produced to energy input as a function of reaction time are presented

in Figure 9. Since the heat loss depends upon the reactor configuration and insulation characteristics, a specific reactor configuration and batch size needs to be chosen for the calculations. In the example here, the inner diameter of the reactor is 0.13 m and its external heat-loss surface area is approximately 0.4 m^2. The heat loss to the surroundings is assumed to be by radiation, which is appropriate for a lunar configuration.

In Figure 9, the time origin refers to the time when the reactor reaches the operating temperature (1273 K in this case). For the case of reaction during heat-up, some oxygen has already been produced by this time (see also Fig. 8). Thus, in this case, the energy efficiency is positive at time zero, in contrast to the case with no reaction during heat- up for which no oxygen has been produced. The difference in the energy efficiencies for the two cases decreases with increase in reaction time at the operating temperature. This suggests that the increase in energy efficiency with reaction during heat-up needs to be balanced against associated additional complexity of the reactor design and operational procedures.

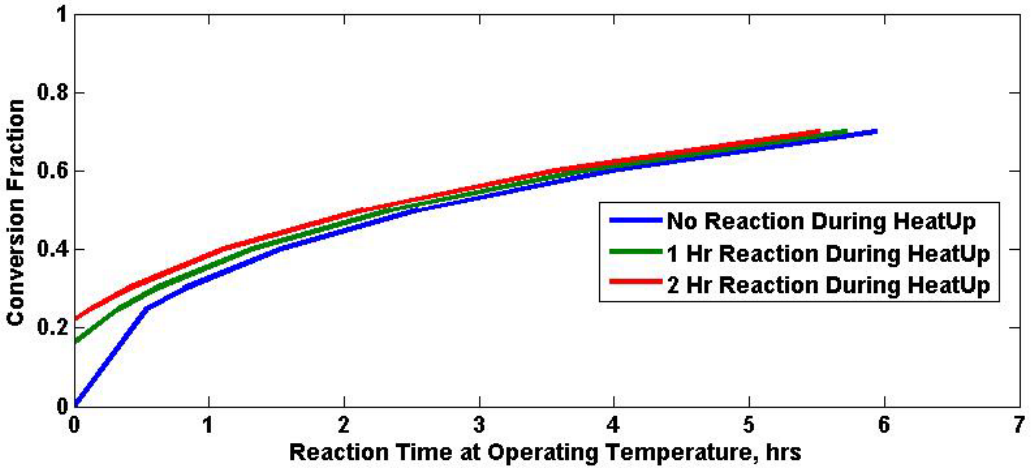


Figure 8.—Conversion fraction during reaction at an operating temperature of 1273 K for different heat-up times.

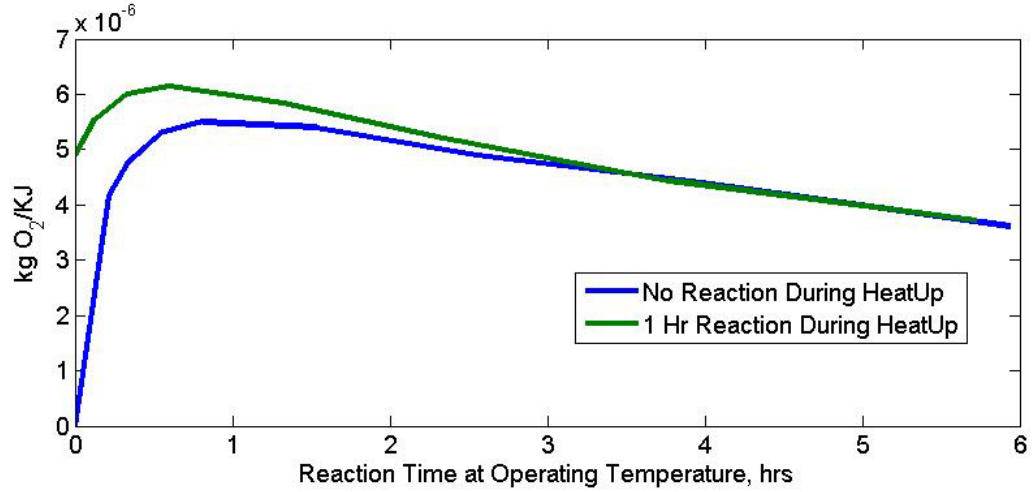


Figure 9.—Oxygen produced per unit energy input as a function of reaction time at the final operating temperature.

Conclusion

A previously developed model for hydrogen reduction of JSC-1A has been enhanced by adding the capability to account for conversion during the simulant heat-up period. The predicted value of the conversion at the end of the heat-up period, utilizing a suitably derived average temperature, is in satisfactory agreement with available data. The conversion fraction during heat-up increases with the heat-up time and final steady-state operating temperature. However, there is a diminishing rate of conversion with increase in heat-up time, as well as in overall energy and power requirements, with increase in the final temperature. The energy efficiency of the process, defined as the cumulative amount of oxygen produced per unit energy input, is generally higher with reaction than with no reaction during heat-up although the difference becomes small for longer reaction times. The model finds application in overall reactor system analysis studies.

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14. ABSTRACT
A previously developed and validated model for hydrogen reduction of JSC-1A for a constant reaction-bed temperature is extended to account for reaction during the bed heat-up period. A quasi-steady approximation is used wherein an expression is derived for a single average temperature of reaction during the heat-up process by employing an Arrhenius expression for regolith conversion. Subsequently, the regolith conversion during the heat-up period is obtained by using this representative temperature. Accounting for the reaction during heat-up provides a better estimate of the reaction time needed at the desired regolith-bed operating temperature. Implications for the efficiency of the process, as measured by the energy required per unit mass of oxygen produced, are also indicated.

15. SUBJECT TERMS
Hydrogen reduction; ISRU; Lunar regolith simulant; Lunar regolith; Lunar simulant

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