



RESEARCH ARTICLE

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Key Points:

- Serpentinization and its products may be used to create a Mg-based concrete for building structures and protection from radiation on Mars
- Serpentinization produces molecular hydrogen (H_2) which would potentially serve as a feasible energy resource on Mars
- Mg-based cement in this study achieved a compressive strength similar to Portland cement

Supporting Information:

- Supporting Information S1

Correspondence to:

C. Oze,
coze@oxy.edu

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Constructing Mars: Concrete and Energy Production From Serpentinization Products

Allan N. Scott¹ and Christopher Oze² 

¹Department of Civil Engineering, University of Canterbury, Christchurch, New Zealand, ²Geology Department, Occidental College, Los Angeles, CA, USA

Abstract In situ building materials and energy resources are necessary for the long-term human habitation of Mars. Olivine ($(Mg,Fe)_2SiO_4$) is a regionally abundant mineral on Mars and may potentially serve both purposes. Here we assess olivine hydrolysis (i.e., serpentinization) and related products including magnesite ($MgCO_3$) as a means to produce a Mg-based cement and molecular hydrogen (H_2) as an energy resource. Magnesium oxide (MgO) may be derived from $Mg(OH)_2$ dehydration or calcination (i.e., thermal decomposition) of $MgCO_3$. In laboratory experiments, Mg-based concrete using MgO achieved a compressive strength in excess of 20 MPa. Serpentinization-related H_2 gas concentrations increased with increasing temperature (20 to 50 °C) and decreasing olivine particle size reaching a maximum H_2 production rate of $4.73 \mu mol g^{-1} day^{-1}$, 3 orders of magnitude greater than previously observed. Results from this study indicate that serpentinization and related products serve as a viable pathway for both concrete and fuel production on Mars with minimal energy expenditure and primarily constrained to the availability of water. Moreover, this process would work equally as well on Earth and has significant potential to decrease global industrial CO_2 emissions.

Plain Language Summary Our results demonstrate that concrete and fuel (H_2) can be produced on Mars using in situ resources.

1. Introduction

Long-term human habitation on the surface of Mars is not feasible without mitigating the high levels of radiation which are detrimental to human health (Bodiford et al., 2005; Hassler et al., 2013; Kerr, 2013). Radiation protection depends on thicker and/or more resistant barriers (i.e., higher atomic weight metals such as lead) than Earth-delivered human shelters are feasibly capable of providing (Chevrier & Mathe, 2007). To solve this problem, in situ resources available on the surface, such as regolith, must be appropriated. In order to create increased protection, a binder system such as cement is required which may serve to ensure the stability and cohesiveness of surface and/or subterranean structures. A range of potential binder options for in situ resource utilization of regolith have been investigated including sulfur-based binder systems (Wan et al., 2016), biological protein composites using bovine serum albumin (Roedel et al., 2014), and possible lime production from anorthite using a recycled fluorine process (Burt, 1991). However, these options have a variety of limitations regarding energy (i.e., solar or nuclear power), water availability and consumption, and material processing. Ideally, an efficient low-energy means of processing of in situ resources would be preferable for the long-term settlement of Mars.

Cement on Earth has been primarily based on the processing of Ca-containing minerals, such as Ca-carbonate, to produce lime (CaO). However, this Ca-centric approach is not applicable for the Mg-rich chemistry of Mars or the energy required for calcination ($>1,000$ °C). Olivine ($(Mg,Fe)_2SiO_4$) is a regionally abundant mineral on the surface of Mars (Hoefen et al., 2003; Mustard et al., 2005; Ody et al., 2013). In water and at relatively low temperatures (<300 °C), olivine undergoes hydrolysis in a process referred to as serpentinization. This process has occurred in Mars' geologic past and may be currently occurring in the Martian subsurface (Ehlmann et al., 2010; Viviano et al., 2013). Products of serpentinization include serpentine group minerals ($Mg_2Si_2O_5(OH)_4$), Mg hydroxide or brucite ($Mg(OH)_2$), and molecular hydrogen (H_2 ; Oze & Sharma, 2007). Additionally, this process results in highly alkaline fluids ($pH > 10$) which aids in secondary carbonate formation such as Mg-carbonate, also regionally present on Mars (Ehlmann et al., 2008; Morris et al., 2010). The primary and secondary Mg-related products of serpentinization potentially provide a means for a Mg-binder system via the formation of MgO (i.e., similar to lime), whether utilizing $Mg(OH)_2$ or Mg-carbonate,

including the processing of Mg-carbonate deposits already present on the Martian surface. Calcination of Mg-carbonate (magnesite) likely requires more energy (higher temperatures) compared to the dehydration of $\text{Mg}(\text{OH})_2$ to form MgO; however, how well MgO will serve as a replacement for lime and being constrained to geologic materials must first be assessed. Additionally, how H_2 produced during serpentinization may be accrued to serve as an energy resource requires further investigation. If these first steps show promise, this nonconventional serpentinization-based approach has the potential to both lower the energy requirements for making a cement as well as providing a fuel source.

Here we assess the novel application of serpentinization and related Mg-carbonate to produce a Mg-binder system and H_2 for energy/fuel on Mars. Ultimately, this process is dependent on (a) water availability/collection, (b) energy requirements for maintaining temperatures above Martian freezing and processing serpentinization materials, (c) the strength/performance of the Mg-binder system, and (d) H_2 collection and storage. Given these obstacles, the Mg-binder system and its feasibility for use in a Martian concrete will first be addressed.

2. Materials and Methods

2.1. Martian Concrete Simulant Production

Cementitious components of the concrete mix design used in this investigation were selected to be representative of those potentially available on Mars. A light burnt (670 °C) MgO, derived from magnesium carbonate, was combined with a natural amorphous silica (i.e., volcanic glass) sourced from the Taupo Volcanic Zone, New Zealand. The quantities of the constituents used in concrete mix design are summarized as follows: water 200 kg/m³, MgO 200 kg/m³, amorphous silica 300 kg/m³, sand 558 kg/m³, stone 1,000 kg/m³, and superplasticizer admixture 20 L.

A wide variety of regoliths with varying gradations are present on Mars. One of the key parameters in the selection of a fine aggregate for use in concrete is the fineness modulus. A sand with a fineness modulus (FM) greater than 3 is considered too coarse while a FM below 2 indicates too many fines which increases water demand. The FM of the Colombia hills sol 499 sample in the Gusev crater was estimated to be 2.53 (Scott et al., 2017). A local 13-mm greywacke coarse aggregate and river sand with a FM of 2.77 were selected for the use in the mix. While the material properties of the aggregate fillers were not identical to those generally available on Mars they did provide a reasonable approximation for the present trials involving short-term strength and general mix characteristics. Estimates of long-term strength, durability and rheology of the concrete, where high-fidelity simulants should be used, were not part of the present investigation.

The magnesium silicate hydrate (MSH) concrete was produced in 90-L pan mixer. The MgO, amorphous silica, and sand were initially dry mixed for approximately 1 min until homogenous followed by the addition of water and super plasticizer and mixed for a further 2 min. The coarse aggregate was added next and all the material mixed for another 2 min.

Paste containing MgO and amorphous silica and a control of general purpose Portland cement with water/binder ratios of 0.4 were cast in sealed plastic bottles for X-ray diffraction (XRD) analysis at 1 and 28 days. Concrete cubes with a dimension of 100 × 100 × 100 mm were cast in two layers, whereby each layer was vibrated for up to 60 s to remove any entrapped air. One day after casting the samples were removed from their molds and cured in water at 20 °C for 7 days. Once the samples were removed from the water curing they were stored in air under ambient conditions of approximately 20 °C and 60% relative humidity until testing at 28 and 90 days. The concrete cube samples were tested in a load-controlled compression machine at a rate of 2 kN/s.

2.2. Serpentinization Experiments

The serpentinization process and reaction products were evaluate by combining 3 g of olivine from Dunn Mountain, New Zealand, with 6 mL DI water in a 12-mL glass vessel with a septum lid to allow for extraction and measurement of the gas in the head space. The effects of finesses on the reaction rates of the olivine were assessed for samples with average particle sizes of 41 and 152 μm. The reaction products were evaluated at two temperatures of 20 and 50 °C after 3, 24, and 72 hr. Triplicate samples were used for each fineness, temperature, and exposure period. The Mg concentration of the reaction products in solution was determined by inductively coupled plasma mass spectrometry at the University of Canterbury after filtering

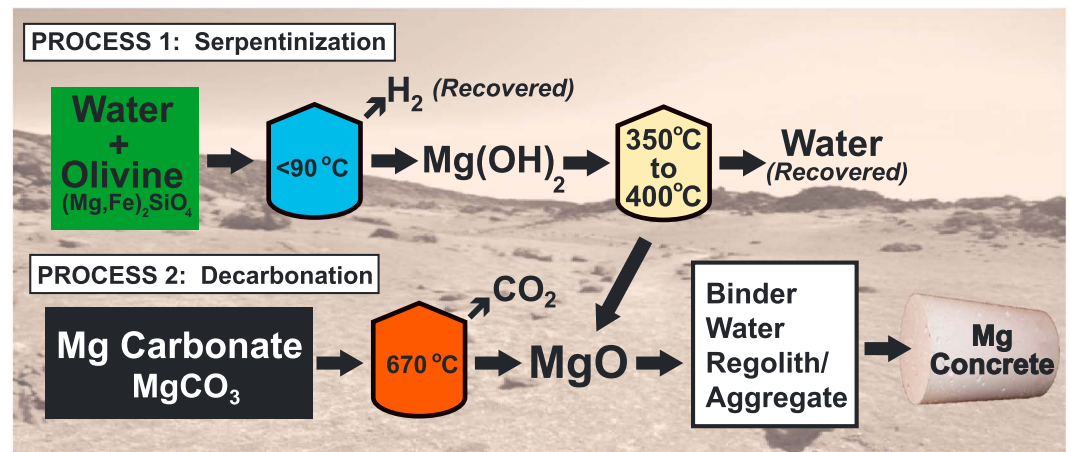


Figure 1. A schematic demonstrating serpentinization (*process 1*) and decarbonation or calcination of MgCO_3 (*process 2*) related to the production of Martian concrete is shown.

through a 0.2- μm membrane. The gas composition in the head space of control samples containing only DI water at 20 and 50 °C was tested to verify that any measured hydrogen in the olivine test samples was a result of the serpentinization process and not from any off gassing of the septum lids or from dissolved gases in the DI water. The reacted olivine was assessed for $\text{Mg}(\text{OH})_2$ formation as well as conversion to MgO following heating of the $\text{Mg}(\text{OH})_2$ to 375 °C, while the Mg-carbonate was assessed for conversion to MgO by heating the material to 670 °C.

2.3. Material Analysis

X-ray diffraction (XRD) analyses of the paste samples were conducted at 1 and 28 days using a Philips PW1729 X-ray diffractometer at the University of Canterbury. XRD patterns were collected between 2θ values of 3–70°, with a generator potential and current of 50 kV and 40 mA, respectively, using $\text{CuK}\alpha$ radiation. SEM analysis was performed on the hydrate paste portion of the samples after 28 days using a JEOL 6400 scanning electron microscope. Additionally, X-ray diffraction (XRD) analyses of magnesite, magnesite heated to 670 °C, MgO , olivine, olivine reacted with water, and reacted olivine heated to 375 °C were made using a PANalytical X'Pert Pro (Caltech) and using a scan speed of 0.5°/min.

Particle size distribution (PSD) for 300- μm subsamples was determined with a HORIBA LA950 particle analyzer using a refractive index of 1.60–0.00i. The FM modulus (ASTM, 1997) and PSD for particles greater than 300 μm were determined by sieve analysis. The gas composition in the headspace of the reaction vessels was analyzed with an Agilent 3000A microgas chromatography using an argon zero grade carrier gas with a purity greater than 99.998%. An Agilent 7500cx ICPMS with argon carrier gas was used to measure the concentration of the dissolved reaction products.

3. Results and Discussion

3.1. Mg-Binder System

The proposed and tested production process for a Martian Mg-binder concrete from both serpentinization and decarbonation of Mg-carbonate to produce MgO and Mg concrete is provided in Figure 1. Regionally abundant olivine on Mars has the potential to react with water to form $\text{Mg}(\text{OH})_2$ at low temperatures (<50 °C; see section 3.2) where it can be dehydrated at temperatures <350 °C to form MgO (Figure 2; e.g., Hamilton & Christensen, 2005; Hoefen et al., 2003; Mustard et al., 2005). $\text{Mg}(\text{OH})_2$ was not detected in olivine reacted with water using XRD (see section 3.2); however, a more sensitive technique, especially when $\text{Mg}(\text{OH})_2$ is likely less than 1 wt.%, may be able to confirm the presence of $\text{Mg}(\text{OH})_2$ and its conversion to MgO . This is a matter of further investigation. A Mg-carbonate derived MgO was used to approximate the material which could be obtained from processing of the Mg-carbonates on Mars such as the Mg-Fe-carbonates found in the Comanche outcrops in the Gusev crater (Morris et al., 2010), though at a lower Fe concentration (Figure 1). Magnesium oxide, whether it is sourced from Mg-carbonates or Mg-hydroxide, as an intermediary in the processing of MgO , can provide similar levels of reactivity depending on the thermal processing

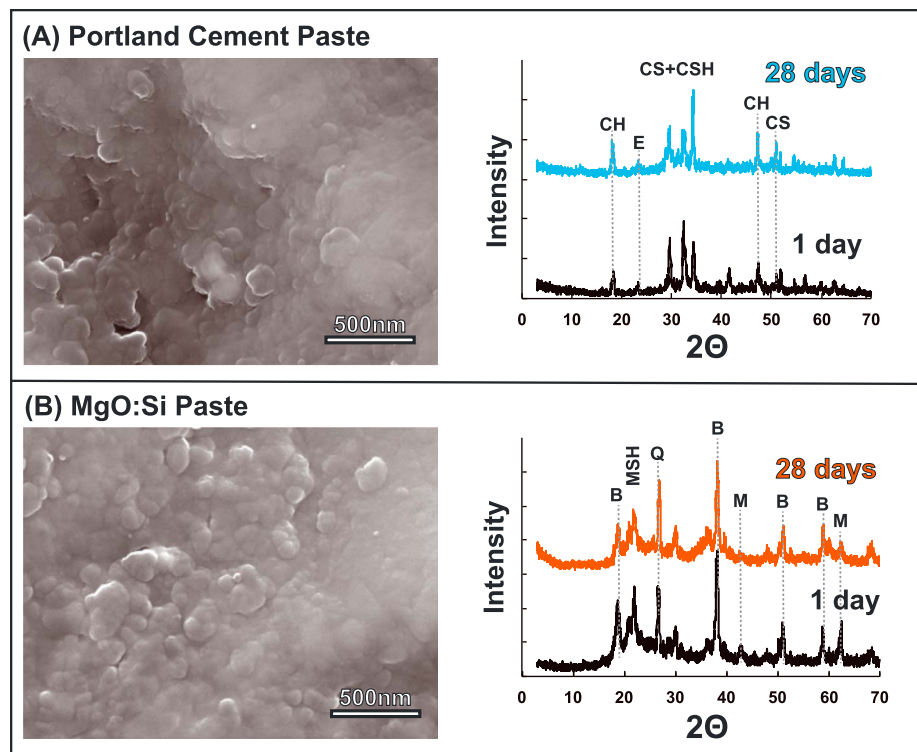


Figure 2. Scanning electron microscope (SEM) images and XRD spectra of (a) Portland cement paste—calcium silicate hydrate and (b) MgO:Si paste—magnesium silicate hydrate. XRD peaks are noted for several phases including B—brucite, CH—calcium hydroxide, CS—calcium silicate, CSH—calcium silicate hydrate, E—ettringite, M—MgO, MSH—magnesium silicate hydrate, and Q—quartz.

(Zhang et al., 2011). The Mg-carbonate produced MgO, while not identical to the proposed serpentinization Mg-hydroxide derived MgO, is sufficiently similar to provide a reasonable indication of the potential performance of the Mg-binder system on Mars using either olivine as the feedstock or Mg-rich carbonates. As significant quantities of MgO could be quickly processed from calcination of magnesite at 670 °C (see Figure S1), we chose to use this route to test our proof of concept of whether MgO could replace lime. In the end, both routes are capable of forming MgO (Figure 1); however, more investigations are needed to increase the yield of serpentinization-related $\text{Mg}(\text{OH})_2$ for conversion to MgO.

Magnesium oxide (MgO) can be combined with amorphous silica to produce a magnesium silicate hydrate (MSH) binder with similar properties to conventional Portland cement (PC). Previous work on MSH binders has focused on combining MgO with silica fume, an extremely fine industrial by-product, to produce mortars with compressive strengths up to nearly 90 MPa (Tran & Scott, 2017; Wei et al., 2006). While silica fume is not available on Mars, naturally occurring amorphous silica is present globally in the regolith at low concentrations and regionally at concentrations greater than 80% (Ruff et al., 2007). The high concentrations of amorphous silica found in the Hellas Basin and the Gusev crater on Mars are considered to be the result of aqueous alterations similar to those which occur near hydrothermal springs on Earth (Bandfield, 2008; Bandfield et al., 2013; Ruff et al., 2007). In the present investigation, a naturally occurring amorphous silica from the Taupo Volcanic Zone, New Zealand, was used. This amorphous silica was formed as a result of hydrothermal alterations of ignimbrite and pumice breccia (Chisholm, 1997), providing a reasonable analog for Martian silica.

The nature of the MSH hydration product of the Mg binder is compared with calcium silicate hydrate (CSH) of the PC binder in Figure 2. The physical features including the size and morphology of the amorphous silicate hydrate component of the two systems, as shown in the SEM images, are similar. Based on XRD results, neither the PC binder nor the Mg binder reached full hydration by 28 days as significant peaks of calcium silicate (CS—the main clinker component) in the PC-binder system and MgO and brucite, an intermediary reaction product, in the Mg-binder system were identified. The greatest difference between the two systems is

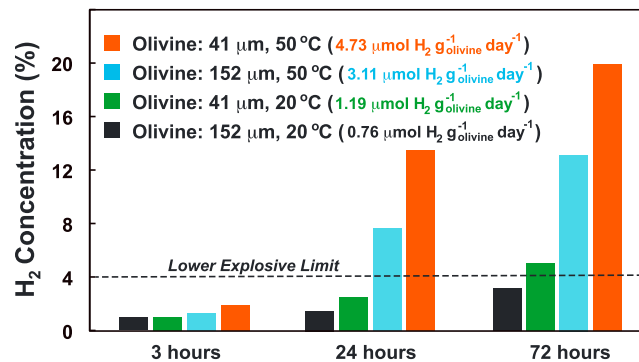


Figure 3. Effects of temperature and particle size on H₂ production (%) over time (hr) with the rates of H₂ production for each particle size and temperature ($\mu\text{mol H}_2/\text{g}$ of olivine/day) are shown. The lower explosive limit (4%) of H₂ in air S(nitrogen, oxygen, CO₂) is included.

related to the chemistry of the pore solution. The pH of the pore solution of MgO–silica fume binder system is approximately 10.5, which corresponds with Mg(OH)₂ equilibrium (Zhang et al., 2011), compared with ~12.5 associated with Ca(OH)₂ and other alkalis found in the PC-based systems.

The Mg-binder concrete achieved a 28-day compressive strength of ~18 MPa, which is comparable to typical residential quality PC concrete on Earth where the gravity and the resulting force acting on a structure is 2.6 times greater than on Mars. The Mg binder showed significant continued hydration and development with 90-day strengths in excess of 28 MPa. Opportunities to significantly increase the compressive strength of the Mg-binder system beyond 28 MPa by decreasing the water/binder ratio of the concrete mixture are possible. The structural capacity of the Mg-binder concrete, therefore, should be more than adequate to meet the Martian loading conditions. Precast sections for permanent habitation modules could be cast in temporary pressurized facilities using the Mg-binder concrete and transported to site for rapid assembly. An alternative construction method for the early stages of Martian settlement would involve spraying a Mg-binder mortar system on the interior of the inflatable structures, similar to conventional shotcrete. This approach will provide greater structural stability as well as increased radiation and micrometeorite protection.

3.2. Hydrogen Production

Serpentinization and H₂ production may occur over a wide variety of pressures and temperatures, even at temperatures <100 °C (Holm et al., 2015; Morrill et al., 2013; Neubeck et al., 2014; Oze & Sharma, 2007). Elevated H₂ concentrations have been noted in both low-temperature field observations (Morrill et al., 2013) and laboratory experiments (Holm et al., 2015; Neubeck et al., 2014; Oze & Sharma, 2007), where Fe²⁺ oxidation in olivine (mainly forsteritic in character) during hydrolysis governs H₂ production. Molecular hydrogen (H₂) measurements are commonly conducted on serpentinization fluids; however, H₂ gas (i.e., headspace gas) was measured in our serpentinization experiments at 20 and 50 °C in order to estimate the extent that H₂ could be collected and processed as an energy resource. Additionally, the relationship of the particle size of olivine and H₂ production was investigated.

Hydrogen gas production related to our low-temperature serpentinization experiments are presented in Figure 3. With a decrease in particle size (i.e., increasing the surface area per mass of olivine) H₂ production increased by 50% at the same temperature. More importantly, an increase in temperature from 20 to 50 °C resulted in a H₂ production increase of 4 times for olivine of the same particle size. The fastest rate of H₂ production was approximately 4.73 $\mu\text{mol/g}$ of olivine/day at the highest surface area and temperature which was at least 3 orders of magnitude greater than that previously measured by Neubeck et al. (2014) over almost one year. To increase the efficiency of H₂ production, higher temperatures would likely be necessary. Please note that Mg concentrations were in excess of 0.23 mM with a corresponding measured pH of approximately 10.2 after only 3 hr. This supports that simultaneous Mg(OH)₂ and H₂ production (Figures 1 and 2) are feasible.

An approximation of the resource requirements for H₂ production can be developed based on the geology and mineralogy of Mars. The composition of olivine across the surface of Mars varies widely from predominantly Mg-rich forsterite to Fe-rich fayalite. The olivine composition in Hellas Basin for instance is

estimated to be Fo₇₄ (Ody et al., 2013), which is within the overall general range of Fo_{68–91} for olivine suggested by Hamilton and Christensen (2005). For the purposes of the following estimate, an olivine concentration in the regolith of 25% is assumed which is within the range of 20 to 30% provided by Edwards and Ehlmann (2015). Using regolith containing 25% olivine and assuming a H₂ production rate of 4.73 μmol/g of olivine/day, as measured in the investigation, 1,000 t of regolith could produce 105 kg of H₂ in approximately 45 days. Assuming that 20% of the olivine undergo serpentinization, four 1,000-t regolith processing units could produce 5,000 kg of H₂ from 48,000 t of regolith, and a similar quantity of water in 18 months. Combined with liquid oxygen or potentially perchlorate (i.e., abundant in Martian regolith (Hecht et al., 2009)), the hydrogen mixture could provide an effective rocket fuel to launch a Mars Ascent Vehicle or for use as a propellant for the return to Earth.

While the ultimate goal of this work is to provide a low-energy method for producing MgO and H₂ for use in combination with silica as a binder system, it must be recognized that considerable amounts of energy (approximately 2.9×10^7 MJ or 7,970 MWh) would be required just to heat the materials (olivine and water—starting from ice) from an average Martian surface temperature of -63 °C to the reaction temperature of 20 °C. A small modular reactor (2 MW) for instance could provide the required initial energy in approximately 3 months. With further investigation it is expected that the efficiency of the process can be improved and the overall energy requirements reduced.

Numerous issues must be resolved before olivine can be used to produce a practical magnesium binder system for application on Mars. While virtually all of the required materials for producing concrete on Mars are available, such as MgO, amorphous silica, aggregate (regolith), and water (Dundas et al., 2018), one of the main complications for producing a workable high-strength MgO concrete is the need for superplasticizers. The development of deflocculants, or alternative superplasticizers, from in situ resources, therefore, will be required for the successful utilization of any concrete on Mars. Additional challenges include improving the recovery efficiency of MgO from the olivine. Using the assumptions outlined for H₂ production, 48,000 t of olivine would yield approximately 576 t of MgO, which if combined with amorphous silica would produce 1,400 t of binder over a period of 18 months for construction. The total energy cost of processing the raw materials and recovering both the minerals and H₂ gas would be well in excess of the 7,970 MWh required simply to heat the material to 20 °C.

These experiments demonstrate the concept that serpentinization and its secondary products, such as magnetite, has the potential to provide not only a timely source of fuel to be used on Mars but also a viable in situ product for future construction. Significant amounts of further investigation will be required before a practical method of mineral and energy recovery from olivine is available.

4. Summary

By utilizing the products of serpentinization, both construction materials and energy can be created on Mars. The distribution and availability of olivine, water, and volcanic glass to create these products is potentially challenging, though the Gusev crater area appears to contain all the necessary materials, with the possible exception being water. With continued characterization and higher resolution of the Martian surface and its minerals, additional suitable sites will become more apparent. With the objective to develop concrete and energy for Mars, this same strategy to create concrete may be feasible (potentially on an industrial scale) on Earth. Creating and utilizing MgO from Mg(OH)₂ required no release of CO₂ and minimal energy (i.e., low temperatures <400 °C), especially compared to current cement production which require temperatures well in excess of 1,000 °C. This Mg-binder system has the potential to significantly decrease global CO₂ emissions related to cement production. As for serpentinization and H₂ production as a source of energy on Earth, it would be too impractical compared to the other readily available energy resources already present. However, H₂ as a by-product of serpentinization-related cement production could be collected with minimal effort and be used as a supplemental energy resource.

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