Distinct Geologic Settings of Opal-A and More Crystalline Hydrated Silica on Mars

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Abstract Opaline silica deposits have been identified on Mars from orbital near-infrared (NIR) reflectance spectra, but opal types (e.g., −A/CT) have not yet been conclusively determined due to insufficient laboratory spectra acquired at Mars-relevant conditions. NIR reflectance spectra of siliceous deposits acquired by the Compact Reconnaissance Imaging Spectrometer for Mars instrument are compared with new laboratory spectra of terrestrial opal samples that we measured under Mars-relevant conditions. Opal-A occurrences on Mars are commonly associated with bedrock exposures, whereas more crystalline hydrated silica (opal-CT and quartz/chalcedony) is primarily observed in unconsolidated sediments. These differences suggest some opal-bearing deposits may have experienced prolonged interaction with near-surface water or diagenetic fluids, and bedrock exposures dominated by opal-A may have experienced less post-formation diagenesis compared with those containing more crystalline hydrated silica. NIR data thus provide a useful tool to assess opal maturity and relative potential for biosignature preservation for different siliceous deposits on Mars.

Plain Language Summary Opals form in habitable aqueous environments and are known to preserve biosignatures on Earth. Opal-bearing deposits have also been identified on Mars, but the distribution of opal type is not well-understood and carries astrobiological implications. Opal-CT and quartz phases are more crystalline than opal-A and often result from post-formation interaction of opal-A with water; such progressive diagenesis could lead to less pristine biosignature preservation. In this work, we distinguish between opal-A and more crystalline hydrated silica (opal-CT and quartz/chalcedony) on Mars for the first time using orbital data. These orbital observations are compared to analogous near-infrared reflectance spectra of terrestrial opal-A, opal-CT, quartz, and chalcedony. On Mars, we find that opal-A is associated with bedrock, whereas more crystalline hydrated silica is associated with aeolian sediment. Because opal-A transforms into more crystalline forms in the presence of water, these findings suggest that silica in aeolian deposits has experienced longer interaction with water than opal-bearing bedrock throughout Martian history. These methods can help better identify and prioritize hydrated silica outcrops on Mars according to their likely degree of post-formation interaction with water and thus their potential to preserve biosignatures. These results have applications towards future astrobiological investigations such as the National Aeronautics and Space Administration’s Mars 2020 rover.

1. Introduction

Hydrated or opaline silica (SiO2·nH2O; e.g., J. B. Jones & Segnit, 1971) has been identified across the Martian surface based on orbital near-infrared (NIR) reflectance spectra, thermal infrared emissivity spectra, and in situ rover observations at Gusev and Gale craters (Bandfield, 2008; Milliken et al., 2008; Morris et al., 2016; Ruff et al., 2011; Skok et al., 2010; Squyres et al., 2008). Along with numerous occurrences of hydrated clay and sulfate minerals, these observations add to the mounting evidence for widespread water-rock interaction in diverse geologic settings throughout Martian history. Hypothesized formation mechanisms for Martian opal deposits are wide ranging and include sedimentary deposition, direct precipitation, and acid leaching of basaltic protoliths (e.g., Bandfield et al., 2013; Bishop et al., 2008; Frydenvang et al., 2017; Milliken et al., 2008; Ruff et al., 2011; Skok et al., 2010). On Earth, opal can form abiotically under a wide range of temperature and pH conditions (Krauskopf, 1956) that include marine (e.g., Kastner et al., 1977), diagenetic, hot spring (e.g., B. Jones & Renaut, 2003), and volcanic fumarolic settings (e.g., Rodgers et al., 2002). While opal alone is a poor indicator of specific aqueous chemistry and geologic conditions, opaline silica has an advantageous characteristic in that its degree of
crystallinity can be correlated with the duration of interaction with water (e.g., Siever, 1962; Williams & Creer, 1985; Williams et al., 1985). Continued interaction of amorphous opal-A with water, as may occur during diagenesis, leads to increased crystallinity and transformation to hydrated silica phases such as opal-CT, opal-C, and microcrystalline quartz or chalcedony, hereafter collectively referred to as more crystalline hydrated silica. The apparent persistence of opal-A on Mars over long timescales has been taken to indicate limited water availability on that planet over the past several billion years (Tosca & Knoll, 2009), although more geochemically mature silica phases such as opal-CT and even tridymite have recently been identified at Gale crater (Morris et al., 2016). Distinguishing opal types is also important for understanding relative differences in biosignature preservation potential for various siliceous deposits on Mars (Cady & Farmer, 1996; Konhauser et al., 2003). Biosignatures in rocks containing more crystalline hydrated silica (e.g., opal-CT and quartz) may be more susceptible to textural overprinting or dissolution by fluids during opal maturation compared with less-altered deposits that retain opal-A (Campbell et al., 2015; B. Jones & Renaud, 2003). Discerning opal types on Mars via orbital NIR spectroscopy could thus help constrain the relative duration of water-rock interaction between different silica-bearing deposits as well as identify which siliceous outcrops may be ideal targets for future in situ exploration.

Variations in NIR absorptions have been observed in laboratory reflectance spectra of terrestrial opals (commonly acquired under ambient conditions; e.g., Rice et al., 2013) and in spectra acquired by the Compact Reconnaissance Imaging Spectrometer for Mars (CRISM) instrument (Milliken et al., 2008; M. R. Smith et al., 2013), and these spectral variations are inferred to represent differences in opal crystallinity/type. Absorptions at 1.4, 1.9, and 2.2 μm reflect the bonding environments of SiOH and H2O, which have different H-bonding behaviors and therefore different spectral characteristics for various opal types (e.g., Boboñ et al., 2011; Brown et al., 2003; Burneau et al., 1990; Langer & Flörke, 1974; Sun, 2017). However, the shape and strength of these features is also influenced by the amount of H2O in the opal, which can vary with relative humidity (RH), temperature (T), and pressure (P; e.g., Milliken et al., 2008). Water content may thus differ between opal types when measured under Earth versus Mars-like atmospheric conditions, an effect suggested by earlier measurements by Milliken et al. (2008) and Rice et al. (2013) and confirmed in a more detailed suite of spectral measurements by Sun (2017). Previous orbital studies of Martian opals relied primarily on comparison with laboratory spectra acquired under conditions not directly relevant to Mars; thus, it reasons that specific opal types on Mars have yet to be conclusively identified from NIR reflectance spectra.

We address this issue by comparing CRISM spectra of opal-bearing deposits on Mars with new laboratory data of natural opals measured under Mars-relevant conditions (Sun, 2017). We demonstrate that different opal types are associated with different geologic settings and discuss these findings in terms of implications for water-rock interaction on Mars through time.

2. Methods

NIR reflectance spectra of opaline silica are characterized by vibrational absorptions at 1.41–1.46, 1.91–1.96, and 2.21–2.26 μm. Absorptions with reflectance minima near 1.41, 1.91, and 2.21 μm (Type A) are respectively due to OH stretch overtones (from SiOH and H2O), a combination bend and stretch mode of H2O, and SiOH vibrations. Partially overlapping absorptions with minima near 1.46, 1.96, and 2.26 μm (Type B) are due to analogous H2O and SiOH groups that are H bonded to additional H2O groups (Langer & Flörke, 1974; Figure S1). Different opal types show differences in these absorptions due to variations in their distribution and abundance of H2O and SiOH. Amorphous opal-A contains more H2O than the more crystalline hydrated silica phases (e.g., Campbell et al., 2001; Herdianita et al., 2000) and adsorbs H2O as a single layer bonded to surface SiOH groups, resulting in deeper 1.41-, 1.91-, and 2.26-μm absorptions corresponding to Type A H2O bonded to Type B SiOH. Though hydrated glasses are also amorphous, silicate glasses of compositions expected on a predominantly basaltic planet such as Mars (e.g., Stolper, 1982; Tornabene et al., 2013) can be spectrally distinguished from opal-A (Milliken et al., 2008) and are not considered here (Text S1). By contrast, more crystalline phases like opal-CT/C, microcrystalline quartz, and chalcedony contain less total H2O than opal-A (e.g., Campbell et al., 2001; Yamagishi et al., 1997) and adsorb H2O in multiple layers, resulting in comparatively deeper 1.46-, 1.96-, and 2.21-μm absorptions corresponding to Type B H2O and Type A SiOH (Text S1).

Recent measurements of opals acquired under ambient (22 °C, 1 atm) and Mars-relevant pressure (0.016 mbar P. H. Smith et al., 2009) presented in Sun (2017) showed that opal-A can be distinguished from more crystalline
hydrated silica using the wavelength position of the ~1.4-μm absorption feature as well as the ratio of the 2.26- to 2.21-μm absorption strength. The ~1.4-μm band position is particularly well-suited for distinguishing between these hydrated silica groups under a range of T, P, and RH conditions (Figure 1). Details of the samples and experimental procedures are provided in Text S2. These results are consistent with those of Rice et al. (2013), which included a wide range of silica-rich materials at ambient conditions and one sample measured at Martian atmospheric pressure.

The position of the ~1.4-μm feature occurs at progressively longer wavelengths with increasing opal crystallinity, and this trend is retained for spectral measurements of opals at Mars-relevant conditions (Figure 1). The ~1.4-μm feature shifts to shorter wavelengths for all opal samples measured under low P and RH conditions due to H₂O loss (Figure S2). This results in an overlapping wavelength range (1.405–1.413 μm) in Figure 1 that does not allow unique discrimination of opal-A from more crystalline hydrated silica and corresponds to cases where opal-A is in a high hydration state and crystalline silica is in a low hydration state with respect to molecular H₂O. However, no opal-A samples plot at wavelengths longer than 1.413 μm, and no opal-CT, quartz, or chalcedony samples plot at wavelengths shorter than 1.405 μm (Figure 1b). This trend holds for opal-A even when it is exposed to 100% RH for several days at room temperature. Similarly, dehydration of more crystalline silica samples via prolonged exposure to low (<1%) RH indicates that its ~1.4-μm feature is unlikely to shift to wavelengths shorter than 1.41 μm unless thermally dehydrated (Sun, 2017). These properties can be used to identify zones within the spectral parameter space of Figure 1 that distinguish opal-A from more crystalline hydrated silica such as opal-CT, quartz, and chalcedony. For example, a spectrum for a terrestrial or Martian opal with a band position of 1.43 μm is highly unlikely to indicate opal-A, and a spectrum with a position of 1.39 μm is unlikely to indicate a crystalline hydrated silica phase.

CRISM spectra of Martian opal occurrences were analyzed using the same methods that were applied to the laboratory data (Text S2). CRISM data cubes covering the 1.0–2.6-μm wavelength region were processed in the Environment for Visualizing Images software package using the CRISM Analysis Toolkit v6.6. Standard photometric, atmospheric, and data spike corrections were applied (McGuire et al., 2009; Parente, 2008). Parameter maps (Pelkey et al., 2007) and manual inspection were used to identify opaline silica in CRISM spectra from previously reported silica-bearing locations on Mars (Figure 2a). The ~1.4-μm position and 2.26/2.21-μm ratio parameters described above were calculated for these CRISM detections to determine which opal types are likely present at the Martian surface. Values for the ~1.4-μm position were determined by finding the minimum reflectance point in continuum-corrected spectra (Clark & Roush, 1984). The ~2.2-μm feature was modeled with band depths and with two Gaussians centered at 2.21 and 2.26 μm. The ratio of the resulting amplitudes is presented as the 2.26/2.21-μm ratio parameter in Figures 1 and 2.

Opal-bearing materials in the CRISM data were further studied in High Resolution Imaging Science Experiment (McEwen et al., 2007) and Context Camera (Malin et al., 2007) images to determine their geologic setting. Opal detections were categorized as bedrock, aeolian or unconsolidated materials, or periglacial materials on the basis of their tone (relative albedo) and texture. Examples of these geologic settings are shown in Figure S3.

3. Results: Spectral Variations Associated With Opal on Mars

CRISM spectra for various opal-bearing deposits (Figure 3) plot within the fields for both opal-A and more crystalline hydrated silica (Figure 2b), suggesting both classes of hydrated silica are present at the optical surface of Mars. These spectrally distinct materials can sometimes occur in close spatial proximity. The most
1.4-μm position. The binned appearance along the x axis is due to the Compact Reconnaissance Imaging Spectrometer for Mars’s 6.55-nm spectral sampling (Murchie et al., 2007).

Figure 2. (a) Opal detections in this work; circles indicate detections within impact craters (Sun & Milliken, 2015), and triangles indicate intercrater detections (Carter et al., 2013; Ehlmann & Edwards, 2014; Fraeman et al., 2016; Milliken et al., 2008; Pan & Ehlmann, 2014; Roach et al., 2010; Seelos et al., 2014; Skok et al., 2010; Weitz et al., 2013; Wray et al., 2011). Detections are colored according to their categorization by geologic material. (b) Spectral parameters for Martian opal deposits based on Compact Reconnaissance Imaging Spectrometer for Mars data. Silica in bedrock appears spectrally distinct from silica found in aeolian or periglacial materials. In general, silica in aeolian/periglacial settings is most similar to crystalline hydrated silica, whereas silica in bedrock are more consistent with opal-A. A hashed zone corresponds to the 1.405–1.413-μm wavelength range where opal-A and more crystalline hydrated silica may overlap in the 1.4-μm position. The spectrum from a nearby bedrock exposure exhibits a ~1.4-μm feature occurring at shorter wavelengths, a characteristic that is most consistent with opal-A (Figure 4b). The apparent lack of a 1.9-μm feature in this spectrum is unusual (compare with Figure 3) and suggests very low or no H₂O content; this has not been observed in terrestrial opal samples at ambient or Mars-relevant conditions and warrants future study. A similar contrast in opal type and geologic setting is found at Nili Patera (Figures 4e–4h), where opal-A is again associated with bedrock (Skok et al., 2010) and more crystalline hydrated silica is present along the lee sides of dunes 20 km south.

Variations observed in laboratory spectra of opal-A and more crystalline hydrated silica are strongly related to differences in H₂O distribution, abundance, and bonding environment within opal, as described previously, but the natural world is inherently more complex. To verify that the spectral differences observed in the CRISM data are most consistent with differences in opal type, we first consider several other factors that may influence spectra of opal-bearing deposits on Mars.

Seasonal differences in atmospheric conditions such as RH may influence the hydration state of Martian opals that could in turn influence CRISM spectra of opal deposits acquired at different times of year. However, the two detections at Elorza crater occur in close spatial proximity within the same CRISM image (Figures 4a–4d). This implies that these opal occurrences are subject to similar atmospheric and RH conditions, yet the deposits still exhibit distinctly different SiOH and H₂O absorptions. These observations indicate that different atmospheric conditions cannot account for all of the spectral variation observed in the CRISM data.

Scattering effects due to differences in particle size are known to affect absorption band depths (e.g., Milliken & Mustard, 2007) and may cause bedrock and particulate materials to have different spectral properties. However, particle size effects cannot cause wavelength shifts in absorption features, which is the spectral characteristic used here to distinguish opal-A from more crystalline hydrated silica. Particle size may also influence H₂O adsorption, with particulates adsorbing water more readily than bedrock due to increased surface area. However, this is not supported by comparisons of laboratory spectra for powder and rock chips derived from the same opal sample. Those data demonstrate that the ~1.4- and ~2.2-μm spectral parameters yield the same distinctions between opal-A and more crystalline hydrated silica regardless of whether the opal is in powder or chip form (Sun, 2017).

Finally, it is possible that some occurrences of Martian opal are mixed with other H₂O-bearing phases that may shift the position of the ~1.4-μm feature. Hydrated minerals including clays, sulfates, and zeolites have been identified on Mars (e.g., Carter et al., 2013; Ehlmann & Edwards, 2014) but exhibit diagnostic absorptions, separate from opal’s features, which are not observed in our CRISM spectra. Al clays are also characterized by absorptions at 1.4, 1.9, and 2.2 μm but can be distinguished from opal by their narrower spectral features. These differences are captured in the 2.26/2.21-μm ratio value, which is effectively zero when there is more than 5–10 wt% Al clay in mixture with opal (Sun, 2017). The majority of the CRISM spectra examined here have 2.26/2.21-μm ratios well above zero (Figure 2b), indicating that non-opaline hydrated minerals are absent or spectrally weak in these deposits. A possible caveat is that some zeolites only exhibit H₂O absorptions and no other diagnostic NIR features; thus, their mixture with opal could result in a spectrum that plots within the more crystalline hydrated silica field of Figure 2b. Though such a scenario cannot be ruled out.

striking example is at Elorza crater, where two different hydrated silica spectra occur 4 km apart in the same CRISM scene (Figures 4a–4d). One spectrum in an aeolian deposit is consistent with more crystalline hydrated silica, indicated by the longer wavelength position of its ~1.4-μm feature. The spectrum from a nearby bedrock exposure exhibits a ~1.4-μm feature occurring at shorter wavelengths, a characteristic that is most consistent with opal-A (Figure 4b). The apparent lack of a 1.9-μm feature in this spectrum is unusual (compare with Figure 3) and suggests very low or no H₂O content; this has not been observed in terrestrial opal samples at ambient or Mars-relevant conditions and warrants future study. A similar contrast in opal type and geologic setting is found at Nili Patera (Figures 4e–4h), where opal-A is again associated with bedrock (Skok et al., 2010) and more crystalline hydrated silica is present along the lee sides of dunes 20 km south.
The preferential occurrence of more crystalline hydrated silica in aeolian materials and opal-A in bedrock may also account for chemical changes observed for carbonates in glacial sediments on Earth (Fairchild et al., 1993). The observation of more crystalline hydrated silica in aeolian and periglacial deposits suggests these siliceous materials are more geochemically mature if the sediment interacted with thin films of water may enable diagenetic maturation of opal-A to opal-CT and quartz, perhaps similar to chemical changes observed for carbonates in glacial sediments on Earth (Fairchild et al., 1993).

The observation of more crystalline hydrated silica in aeolian and periglacial deposits suggests these siliceous sediments are more geochemically mature and likely experienced longer integrated water-rock interaction compared with silica in bedrock. This difference may be attributed to several geologic processes. Within periglacial environments, silica may be more geochemically mature if the sediment interacted with thin films of water, possibly due to seasonal melting of ice. Integrated over geologic time, even short-lived interaction with thin films of water may enable diagenetic maturation of opal-A to opal-CT and quartz, perhaps similar to chemical changes observed for carbonates in glacial sediments on Earth (Fairchild et al., 1993).

The preferential occurrence of more crystalline hydrated silica in aeolian materials and opal-A in bedrock may also indicate differences in the surface exposure ages of opal in these two settings. In one possible scenario, opal-A-bearing rock exposed at the surface could be aqueously altered to more crystalline silica; subsequent physical weathering could erode more crystalline hydrated silica from the outcrop to produce siliceous sediment and leave behind a fresh bedrock surface containing opal-A. Aeolian silica deposits in Nili Fossae were interpreted as lag deposits formed in a similar manner (Ehlmann et al., 2009). Bedrock silica may therefore represent relatively young surface exposures that have not yet undergone opal maturation and thus retain an opal-A composition. This would imply that silica-bearing bedrock units previously matured faster than the erosion rate and were a source of more crystalline silica, but that the opposite is now true and conversion to more crystalline hydrated silica is outpaced by erosion rates in the modern Martian environment. Such an interpretation also carries implications about how the availability of near-surface liquid water may have evolved through time in these locations.

An alternate explanation for the distinct correlation of opal type with geologic material is that unconsolidated sediment could experience enhanced rates of chemical weathering due to increased surface area compared with intact outcrop. In this scenario, both bedrock and aeolian silica may begin as opal-A, the latter perhaps based solely on the spectral data, the inferred geologic setting of opals with wavelength positions >1.41 μm suggests this is an unlikely explanation (section 4).

In summary, atmospheric conditions, particle size effects, and mixing with other hydrated phases is unlikely to explain all of the observed spectral variation in the ~1.4-μm feature for Martian opal deposits. When comparing with results from laboratory spectral measurements acquired under ambient terrestrial and Mars-relevant T/P/RH conditions, the spectral variation in Figure 2 is best interpreted as reflecting differences between opal-A and more crystalline hydrated silica.

4. Discussion: Correlation of Opal Type With Geologic Setting

When orbital detections of Martian opal deposits are classified by the morphological attributes of their host deposits, an association between opal maturity and geologic setting is observable on a global scale (Figures 2 and S3). Silica occurrences associated with bedrock tend to have ~1.4-μm positions short of 1.41–1.42 μm and are most consistent with opal-A. This is consistent with the identification of opal-A in bedrock at Gusev crater (Ruff et al., 2011; Squyres et al., 2008) and Gale crater (Morris et al., 2016). From an orbital perspective, bedrock occurrences of more crystalline hydrated silica are present but relatively uncommon (Figure 2). In contrast, spectra of silica in unconsolidated deposits (hereafter aeolian silica) and potential periglacial deposits tend to have band positions longer than 1.41–1.42 μm and are more consistent with relatively crystalline hydrated silica. Given the strong global correlation between aeolian materials and spectra with longer ~1.4-μm positions, it is unlikely that this spectral characteristic is due to opal-A being mixed with a nonopaline H₂O-bearing phase (section 3). This would require that nearly all aeolian deposits with detectable opal also contain such a similar additional phase, whereas bedrock occurrences do not, despite most hydrated mineral detections on Mars being associated with bedrock (Carter et al., 2013; Ehlmann et al., 2011).

The observation of more crystalline hydrated silica in aeolian and periglacial deposits suggests these siliceous deposits and potential periglacial deposits tend to have band positions longer than 1.41–1.42 μm and are more consistent with relatively crystalline hydrated silica. Given the strong global correlation between aeolian materials and spectra with longer ~1.4-μm positions, it is unlikely that this spectral characteristic is due to opal-A being mixed with a nonopaline H₂O-bearing phase (section 3). This would require that nearly all aeolian deposits with detectable opal also contain such a similar additional phase, whereas bedrock occurrences do not, despite most hydrated mineral detections on Mars being associated with bedrock (Carter et al., 2013; Ehlmann et al., 2011).

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being sourced from the former, and both may experience identical interaction with water through time. However, the aeolian silica could convert to more crystalline silica at a faster rate due to its increased surface area. Studies of terrestrial aeolian materials suggest that they are better able to retain fluids after wetting compared to bedrock, which could facilitate dissolution and reprecipitation of silica (Folk, 1978) and result in eventual opal maturation via Ostwald ripening. Periods of precipitation on Mars were likely limited or lacking over the past several billion years (Jakosky & Phillips, 2001), so this slow opal maturation process could be facilitated by freeze-thaw cycles that are currently restricted to higher latitudes, similar to the mechanism proposed for opal maturation in Martian periglacial deposits. We note that sediment in the interpreted aeolian deposits is or was previously mobile, and more crystalline silica produced at higher latitudes or locations with enhanced water-rock interaction could be redistributed over time to lower latitudes or drier environments by aeolian transport. In this context, more crystalline hydrated silica observed in aeolian settings is not necessarily indicative of aqueous conditions in those locations either in the recent or ancient past.

5. Conclusions and Implications
Our results demonstrate that the relative frequency of opal-A and more crystalline hydrated silica on Mars is more comparable than previously thought (Tosca & Knoll, 2009), and the two silica groups tend to occur in different geologic settings. The distinct association of opal-A with bedrock and more crystalline silica with aeolian and other unconsolidated deposits suggests that interpretations of opal type from orbital data must be assessed with full geologic context to make meaningful inferences about Martian paleoenvironments and...
local aqueous conditions. For example, the presence of more crystalline hydrated silica may not indicate pro-
longed aqueous activity in that location if the silica is hosted in mobile sediment. Similarly, a detection of
opal-A in bedrock may not indicate a water-limited environment if the opal-Ä represents a younger outcrop
exposure that was previously overlain by more crystalline hydrated silica.

A broad implication of this work relates to orbital NIR reflectance spectra, which provide a means to identify and
prioritize siliceous outcrops by their potential for biosignature preservation. Siliceous materials like opal are
among the best geologic mediums for biomarker preservation in the terrestrial record (Cady & Farmer, 1999;
Frydenvang et al., 2017; Konhauser et al., 2003; Summons et al., 2011), and the persistence of opal-Ä in certain
ancient strata on Mars indicates limited water-rock interaction in these locations. In contrast, bedrock and aeol-
ian materials that contain more crystalline hydrated silica may indicate enhanced interaction with diagenic
fluids or higher temperature processes that may diminish the preservation of biosignatures (Campbell et al.,
2015; B. Jones & Renaut, 2003). In addition to opal-Ä outcrops previously observed in situ at Gusev crater
(Ruff et al., 2011; Squires et al., 2008), a candidate landing site for NASA’s Mars 2020 rover, there exist a variety
of silica-rich outcrops across Mars that are compelling targets for future in situ astrobiological investigations.

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