# The mid-IR spectral effects of darkening agents and porosity on the silicate surface features of airless bodies

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**Abstract**

We systematically measured the mid-IR spectra of different mixtures of three silicates (antigorite, lizardite, and pure silica) with varying effective porosities and amounts of darkening agent (iron oxide and carbon). These spectra have broad implications for interpretation of current and future mission data for airless bodies, as well as for testing the capabilities of new instruments. Serpentines, such as antigorite and lizardite, are common to airless surfaces, and their mid-IR spectra in the presence of darkening agents and different surface porosities would be typical for those measured by spacecraft. Silica has only been measured in the plumes of Enceladus and presents exciting possibilities for other Saturn-system surfaces due to long range transport of E-ring material. Results show that the addition of the IR-transparent salt, KBr, to simulate surface porosity affected silicate spectra in ways that were not predictable from linear mixing models. The strengthening of silicate bands with increasing pore space, even when only trace amounts of KBr were added, indicates that spectral features of porous surfaces are more detectable in the mid-IR. Combining iron oxide with the pure silicates seemed to flatten most of the silicate features, but strengthened the reststrahlen band of the silica. Incorporating carbon with the silicates weakened all silicate features, but the silica bands were more resistant to being diminished, indicating silica may be more detectable in the mid-IR than the serpentines. We show how incorporating darkening agents and porosity provides a more complete explanation of the mid-IR spectral features previously reported on worlds such as Iapetus.

**Introduction:**

Studies of surface composition can help unravel the mysteries of Solar System origins and facilitate understanding in planetary processes through time. Airless bodies have the added benefit of having no atmospheric processes to obscure or destroy what has been preserved on the surface. Examples of such bodies include: Earth's Moon, Deimos, Phobos, Mercury, near-Earth and main belt asteroids, dwarf planet Ceres, and asteroids and moons of the outer solar system. The formation history of the giant planets in the outer solar system is critical to solar system formation models, and this history may also be preserved in the surfaces of the airless bodies orbiting those planets. Moreover, a number of the moons present in these planetary systems are suspected to harbor subsurface oceans, and compositional studies can aid in the search for the requirements of life on these moons. In addition to the ability to observe impact craters, collision debris, and current or past geologic activity manifested on the surface without complication of an atmosphere, there are surface processes that only occur on airless bodies.

Without an atmosphere, the space environment is allowed to interact directly with the surface, resulting in the degradation of the surface through space weathering processes. The term "space weathering" encompasses a number of ways that the space environment may change the surface, such as: micrometeorite bombardment, solar and cosmic energetic photons, and implantation of solar and cosmic ions (e.g., Bennett et al., 2013; Pieters and Noble, 2016). Space weathering can result in featureless, or otherwise puzzling spectra we might not expect in light of a given body's likely bulk composition, making it difficult to identify the constituents present. The end result of space weathering is to produce an amorphous surface layer on grain rims, often containing sub-micron nanophase opaques. In the case of lunar grains, the nanophase opaques are pure iron, but iron sulfide, iron oxide, and carbon absorbers are also expected on other bodies. All darken the surface, while the smallest particles (<40 nm) both darken and "redden" the spectra.

Enigmatic dark surfaces are present on many types of bodies both in the inner and outer Solar System and are particularly pronounced on Saturn's moons: Iapetus, Phoebe, Hyperion, Rhea, and Dione. Transport of dark material from an external source is currently the most accepted explanation, although endogenic processes or a combination of external and internal sources is a possibility (Smith et al., 1982). In the visible wavelengths, spectra from Iapetus have shown evidence for nanophase metallic and oxidized iron and carbon (Clark et al., 2012), all of which can be products of space weathering. The spatial distributions (Clark et al. 2008; Stephan et al. 2010) and similar compositions (Clark et al. 2008; Buratti et al. 2005; Palmer & Brown 2011; Tosi et al. 2010; Dalton et al. 2012) of the dark material on these moons have pointed to a common source external to the Saturn System. However, there is also a strong case that it was transported to Iapetus and Hyperion from the Phoebe ring (Tosi et al., 2010), which is made up of predominantly small particles produced by collisions of Saturn's irregular satellites (Tamayo et al., 2016). Studies of rayed craters on Dione report short retention rates of rays, indicating that bombardment by plasma and E ring particles, as well as deposition of dark particulates similar to those found on other Saturnian satellites, occurred relatively recently (Hirata and Miyamoto, 2016).

Both the rings of Saturn and Uranus are composed of a fraction of dark material. Saturn's rings are almost all water ice, with a minor dark component thought to be organics (Dale Ore et al., 2012; Le Gall et al., 2014) or due to micrometeorite bombardment of silicates (Zhang et al., 2017). By contrast, Uranus' rings have a much lower albedo than those of Saturn and therefore a higher concentration of dark material. The inner moons of Uranus also are dominated by a dark component and have low albedos. The composition of Uranus' rings and moons and their potential association with the processes responsible for the dark material in the Saturn System remains an open question. No spacecraft has been to Uranus since the Voyager 2 flyby (which did not carry a compositional spectrometer), but several mission concept studies have been performed to plan a return (e.g., Hubbard et al., 2010; Saikia et al., 2014; Arridge et al., 2014; Turrini et al., 2014; Bocanegra-Bahamón et al., 2015; Bramson et al., 2017).

There have been numerous shorter wavelength studies of the dark material in the Saturn System. However, at regions as cold as the outer solar system, the peak radiation is emitted at the longer wavelengths. The mid-infrared (mid-IR) is the region of the strongest fundamental vibrations of simple and complex molecules and is rich in emissivity features (e.g., Hand et al., 2009). Yet, few spacecraft studies have investigated outer airless bodies in the mid-IR to date, due to a lack of missions to the outer solar system and the challenges of low signal to noise ratios in the mid-IR for missions that have carried a spectrometer. Despite the paucity of missions to the outer solar system over the entire history of human space exploration, there are exciting upcoming missions, such as the Lucy Trojan asteroid mission and the James Webb Space Telescope (JWST), for which mid-IR laboratory analog spectra of typical airless body compositions will aid in the data analysis.

To demonstrate how laboratory mid-IR spectra can be used to assist with interpretation of spacecraft data, we consider the first mid-IR emissivity feature detected on any icy moon, found on Iapetus' dark terrain. Young et al., 2015 attributed the feature (centered at ~11.7 μm) to silicates, particularly magnesium-rich phyllosilicate serpentines. Phyllosilicates are also found in meteorites (e.g., McAdam et al., 2015) and inferred on other airless extraterrestrial objects, such as Phobos and Deimos (Fraeman et al. 2014; Giuranna et al. 2011; Glotch et al., 2015). Additionally, nanophase silica, which could ultimately be sourced from Enceladus' plumes (Hsu et al., 2015), is another plausible silica-rich phase in the Saturn system. However, neither serpentine nor silica is dark enough to explain the low albedo of Iapetus' leading side. Previous studies conducted of Iapetus at visible wavelengths have provided evidence for nanophase iron and/or carbon darkening phases, along with water and carbon dioxide ice (e.g., Clark et al., 2012). Asteroids also display a similarly located feature in the mid-IR that may be attributed to silicates as well and/or high surface porosities (Vernazza et al., 2012). The low thermal inertias of many of the Saturn satellites can be attributed to fine particle size and/or high surface porosity (Howett et al., 2010).

We hypothesize that on many of these bodies, silicates exist in the presence of darkening agent(s) and that the grain size and surface porosity of these components will affect the feature expression. The goal of this study is two-fold: (1) to present general surface analog measurements to help facilitate interpretation of mid-IR spectra of airless bodies from present and future mission data; and (2) to show the utility of mid-IR laboratory analog studies of the impacts of darkening agents and surface porosity on silicate spectral features, using Iapetus as a case study. To this end, a systematic approach to investigate the effects of darkening agents and surface porosity on silicate surfaces was employed.

**Materials and methods:**

Nanophase silica (5 – 15 nm), purchased from Sigma-Aldrich, and two phyllosilicate serpentines (antigorite and lizardite), acquired through Excalibur Mineral Corp., were selected as the silicates used to represent extraterrestrial surfaces in this study. The antigorite was obtained from Cornwall, England, and the lizardite was found at Cedar Hill Quarry, Pennsylvania, USA.

An IR-transparent salt, potassium bromide (KBr) 99+% metal basis, was used to simulate porosity (Vernazza et al., 2012; King et al., 2011), and nanophase iron oxide (<50 nm) and aquarium activated carbon were chosen for darkening agents. Both KBr and nanophase iron oxide were procured through Sigma-Aldrich.

Mid-IR spectra tend to be very sensitive to grain size (e.g., Wray et al., 2014). Sub-micron grains are indicated by the low thermal inertias observed on Iapetus (Howett et al., 2010; Rivera-Valentin et al., 2011), and exhibit enhanced transparency features (~11–14 μm or 710–910 $cm^{-1}$) and diminished reststrahlen bands (~9–12 μm or 830–1100 $cm^{-1}$) (Logan et al., 1973) over coarser grains. To create appropriately sized grains, serpentines and activated carbon were ground with deionized water in a wet-micronizing mill for 20 minutes to produce very fine grains, on the order of a few microns (O'Connor and Chang, 1986). These grains were produced in a slurry. Water was evaporated off, but clumping of micron-sized grains into ~0.2 mm aggregates suggests that thin films of moisture remained. Efforts to further dry and break apart these clumps revealed no changes in spectra compared to the clumped micronized samples. There was no need to micronize the silica and iron oxide since these were purchased in the nanophase.

Spectral libraries are available for many materials at Earth ambient atmosphere conditions, but spectral features have the potential to shift in position and strength with changes in temperature (e.g., Dalton et al., 2005; Wray et al., 2014) and pressure (Logan et al., 1973; Wray et al., 2014). For each silicate mineral, spectra were taken in JPL's Icy Worlds Simulation facility within the Ocean Worlds Lab under ambient conditions in a $N_2$ purged glovebox and under ultra-high vacuum (typically less than $1x10^{-8}$ torr) and Iapetus-like temperature (125 K) conditions. Both setups used the same model MIDAC Fourier Transform IR spectrometers

(FTIR) in similar, bi-conical geometries, with 90$^{o}$ between the source and the detector. Emissivity was calculated as one minus the measured spectral reflectance.

We performed a systematic investigation to determine the effects of varying porosity and darkening agents on mineral spectra. Binary mixtures with high, mid, and low concentrations by mass of each component were measured. Care was taken consistently across samples to provide an optically thick layer of material in the sample cup and to not compact the grains, as different grain packing configurations may influence spectral features.

**Results and Discussion:**

Past laboratory analog studies of silicates present in extraterrestrial soils have found slight variations in the positions of the diagnostic silicate transparency, reststrahlen, and Christiansen (~7 - 9 μm or 1100 – 1400 cm$^{-1}$) spectral features in different thermal emission environments (Donaldson-Hanna et al., 2012a, 2012b, 2014). For each silicate used in this study, spectra were collected at cold vacuum conditions and compared to spectra collected for the same silicate at ambient atmosphere in the vacuum chamber and the $N_2$ purged glovebox. We observed no appreciable differences in band positions based on pressure and temperature for the serpentines, lizardite and antigorite, nor for nanophase silica (Fig. 1). Although slight variations were present for each silicate, all of these were within the noise level of the FTIR measurements. These initial results justify obtaining spectra of complex mixtures under ambient conditions alone, though future work with an emission spectrometer with the appropriate thermal environment would be necessary to fully confirm this result. The finding that measurements under Earth atmospheric temperatures and pressures are sufficient for the identification of the diagnostic features for these silicates is of importance to a wide range of laboratory analog investigations due to the ubiquitous nature of silicates on planetary surfaces.

4b).

For all of the silicate minerals measured, the addition of increasing levels of KBr salt (Fig. 2) to simulate surface porosity tended to make emissivity features more pronounced. This is a surprising effect that is not readily obvious from individual pure spectra. The KBr also served to make transparency features of the serpentines around 900 $cm^{-1}$ much wider (Fig. 2a, b) and shifted the band centers toward lower wavenumbers. The emissivity features of the silica were made deeper by the addition of salt, but retained their original width at the salt concentrations shown in Fig. 2c. Even wider, deeper emissivity features were observed at very high concentrations (~98%) of KBr mixed with serpentines, and transparency band centers decreased in energy by ~100 $cm^{-1}$. Deeper features for silica were also present, although the bands did not change appreciably in width or shift in position (Fig. 3).  Silicates with very high porosity have stronger emissivity features than those with fewer pore spaces, and may be more realistic for some optically bright icy planetary surfaces. Mid-IR thermal emission features are challenging to obtain over bright terrains due to the lower temperatures, and resulting signal to noise ratio, of these surfaces. However, these results imply that the silicate features of extremely porous surfaces tend to be much more pronounced, and thus should be easier to detect above the noise.

As an example of how surface porosity variation may be used as a tool to help interpret spacecraft data, we consider the silicate mid-IR surface feature identified by Young et al. 2015 on Iapetus' dark terrain (Fig. 4a). Previously, the main feature centered at ~855 $cm^{-1}$ had been compared to that of Mars silicates (Christensen et al., 2004) and was most consistent with pure lizardite and antigorite (Bishop et al., 2008), pointing to a meteoritic origin. However, the width and position of the primary feature closely matches that of the main feature in our nanophase silica sample, indicating that silica could be a viable option, in addition to the serpentines (Fig. 4b).

While the band depths do not correspond precisely between the two plots, we contend that the widths and centers of the features are the most informative aspects to compare. Silica alone is not the only surface constituent, and adding more components to the mixture can change the relative band depths. Furthermore, as Fig. 2c shows, silica tends to retain the width of its mid-IR spectral features with increasing porosity, while the band depths were highly dependent on surface porosity. A nanophase silica identification of Iapetus' mid-IR surface feature could point to a source from the Enceledus plumes, and although this is a large distance, plume particles have been observed traveling even farther, leaving the Saturn system entirely (Kempf et al., 2005). A similar mid-IR feature may be expected to exist along the Tiger Stripe regions of Enceladus if silica particle aggregation is occurring inside the plume, leading to surface deposits. Silica under vacuum conditions also exhibits a shorter wavelength reflectance minimum at ~3 µm (Fig. 5) that would be expected to be observed in conjunction with mid-IR features. A feature at ~3 µm has been observed on Iapetus by Clark et al. (2012), although any water-bearing phase (including ice) could also produce this band.

We acknowledge that not all of the features seen in the observational data are present in the measured silica spectra. There is a large trough in the silica spectra at lower wavenumbers that is not seen in the observation data, which have a doublet in this region. Young et al. (2015) attributed the doublet in the observed data to serpentine or $CO_2$ ice. This trough may be influenced by the addition of ices and/or darkening agent(s), which has not yet been examined.

Darkening agents are responsible for making these surfaces warmer, helping to increase the signal to noise ratio of individual spectra, but they can also make the composition more obscure by reducing spectral contrast. The emissivity of pure nanophase iron oxide increases with decreasing wavenumber and is spectrally featureless. The effect of adding nanophase iron oxide to serpentines, such as lizardite and antigorite, is to dampen all silicate features and steepen the slopes of serpentine spectra (Fig. 6a and 6b). This is also generally the observed impact of mixing nanophase iron oxide with nanophase silica, except that the iron oxide serves to intensify the reststrahlen bands of the silica (Fig. 6c). The reststrahlen bands are often the most diagnostic feature of silicates, and iron oxide present with silica would cause them to deepen. This would increase detectability at the larger wavenumbers, which is the noisiest part of mid-IR spectra at icy moon surface temperatures. Unfortunately, features at the less noisy smaller wavenumbers are simultaneously weakened by addition of nanophase iron oxide. Pure micronized carbon has a flatter slope than nanophase iron oxide, which results in weaker spectral features at all wavenumbers when added to both serpentines (Fig. 7a and 7b) and silica (Fig. 7c). However, adding equal amounts of carbon to serpentines and silica seems to reduce spectral contrast most in the serpentines, indicating that silica features are more resistant to weakening due to carbon mixing.

The presence of nanophase darkening agents, often produced by space weathering, may cause featureless mid-IR spectra for various planetary surfaces. Of the few studies that have been conducted of icy moons in the mid-IR, the vast majority report no spectral features (e.g., Carvano et al., 2007; Howett et al., 2016). However, for the icy moons, darkening agents can help the surface to have an increased signal over optically bright surfaces. When present with darkening agents, silica surfaces should be easier to detect than serpentine, and features would be deepened by high surface porosities. All of these conditions are expected to be present on the dark side of Iapetus and may provide a more complete explanation for the detectability of the spectral feature reported by Young et al. (2015).

Due to the challenges of retrieving mid-IR spectral information from icy moon surfaces, the datasets may require more finessing for scientific interpretation, including developing automated programs that use statistical methods to detect features. These software tools would be particularly important for instruments facing an increase in noise over signal, which can happen for various reasons during a mission, especially near the end of mission life. We are continuing to develop such methodologies in order to assess the value of mid-IR hyperspectral instruments to the study of icy moon surface compositions on future space missions. Other bodies that have similar surface conditions may also present challenges. For example, across vast dust-covered regions of Mars, the only feature is at ~840 $cm^{-1}$ and is attributed to fine-grained silicates (Christensen et al., 2004). In contrast to Mars, VIS/NIR and mid-IR measurements of spectral features on Phobos and Diemos point to phyllosilicates and/or nanophase iron from space weathering (Fraeman et al., 2014; Giuranna et al., 2011; Glotch et al., 2015), and these definitively known features may be used to test automated detection algorithms. Shocked silicates/impact glasses also produce similar features (Morlok et al., 2016; Lederer et al., 2016), may occur in concert with space weathering processes, and should also be investigated as other potential physical mechanisms through which these features can form.

**Conclusions:**

The surfaces of airless bodies present opportunities to investigate the physical processes impinging on planetary systems over time, without the need to account for surface-atmosphere interactions. Silicate surfaces mixed with fine-grained optically dark material with varying degrees of porosity are ubiquitous on many airless surfaces. Although the mid-IR is rich in emissivity features of important molecular classes, including organics, the mid-IR is less studied for many airless surfaces and presents challenges in signal-to-noise ratio, especially for the colder outer solar system bodies with fine-grained surfaces. We systematically measured the mid-IR spectra of different binary mixtures of three silicates (antigorite, lizardite, and pure silica) with varying effective porosities and amounts of darkening agent (iron oxide and carbon), and these spectra have broad implications for interpretation of current (e.g., Cassini, MGS) and future (e.g., Lucy, JWST, OSIRIS-Rex, Europa Clipper) mission data for airless bodies of both the inner and outer solar system. The serpentines, represented here by antigorite and lizardite, are common to airless surfaces, and their mid-IR spectra in the presence of darkening agents and different surface porosities would be typical for those measured by spacecraft. Although pure silica has only been measured in the plumes of Enceladus, it presents exciting possibilities for other surfaces due to long range transport, and it is important to investigate how its spectral signature would be manifested in the mid-IR for detection purposes.

Measurements of silicates and darkening agents performed at vacuum and cold temperature showed no differences when compared to spectra taken at Earth ambient atmospheric conditions. These findings illustrate that there may be no need for time-intensive cold vacuum measurements of these materials, which streamlines the process. The addition of the IR-transparent salt, KBr, to simulate surface porosity affected silicate spectra in surprising ways that were not predictable from the individual spectra (or linear mixing models). The addition of KBr to the serpentines tended to deepen and widen the transparency feature, while shifting its center to lower wavenumbers (longer wavelengths). The KBr had little influence on the width or position of bands in the silica spectra, but did substantially deepen them. The strengthening of silicate bands with increasing pore space, even when only trace amounts of KBr were added, indicates that spectral features of porous surfaces are more detectable in the mid-IR. A case study for the 11.7 μm feature previously detected at Iapetus showed the feature more closely matches those silica mixtures exhibiting some degree of porosity, further demonstrating the utility in making these laboratory measurements. Combining iron oxide with the pure silicates seemed to flatten most of the silicate features, but strengthened the reststrahlen band of the silica, which is typically a highly diagnostic feature. Incorporating carbon with the pure silicates weakened all silicate features, but the silica bands were more resistant to being diminished. Fine-grained darkening agents can help increase the mid-IR signal from cold surfaces by raising the temperature, but they can also dampen spectral features. The special behavior observed for silica, combined with increasing levels of darkening agents, would allow better preservation of spectral features over the serpentines.

Overall, this work provides a library of mineral binary mixtures to facilitate investigating current and future mid-IR datasets of airless bodies, and higher order mixtures are forthcoming in a companion paper. These results are also applicable to the development of future missions to airless bodies, and our continuing efforts will help determine if mid-IR spectroscopy is worthwhile for surface compositional studies of some of these bodies due to the limited spectral features observed. Mixtures such as those presented here would be quite valuable to measure for testing/calibration of mid-IR spectrometer instrument concepts, by confirming detectability of spectral features on the surfaces of interest.

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## Figures

**Figure 1**. Spectra collected at cold (125 K) vacuum conditions compared to spectra collected at ambient atmosphere in the vacuum chamber and the $N_2$ purged glovebox for (a) lizardite, (b) antigorite, and (c) silica.

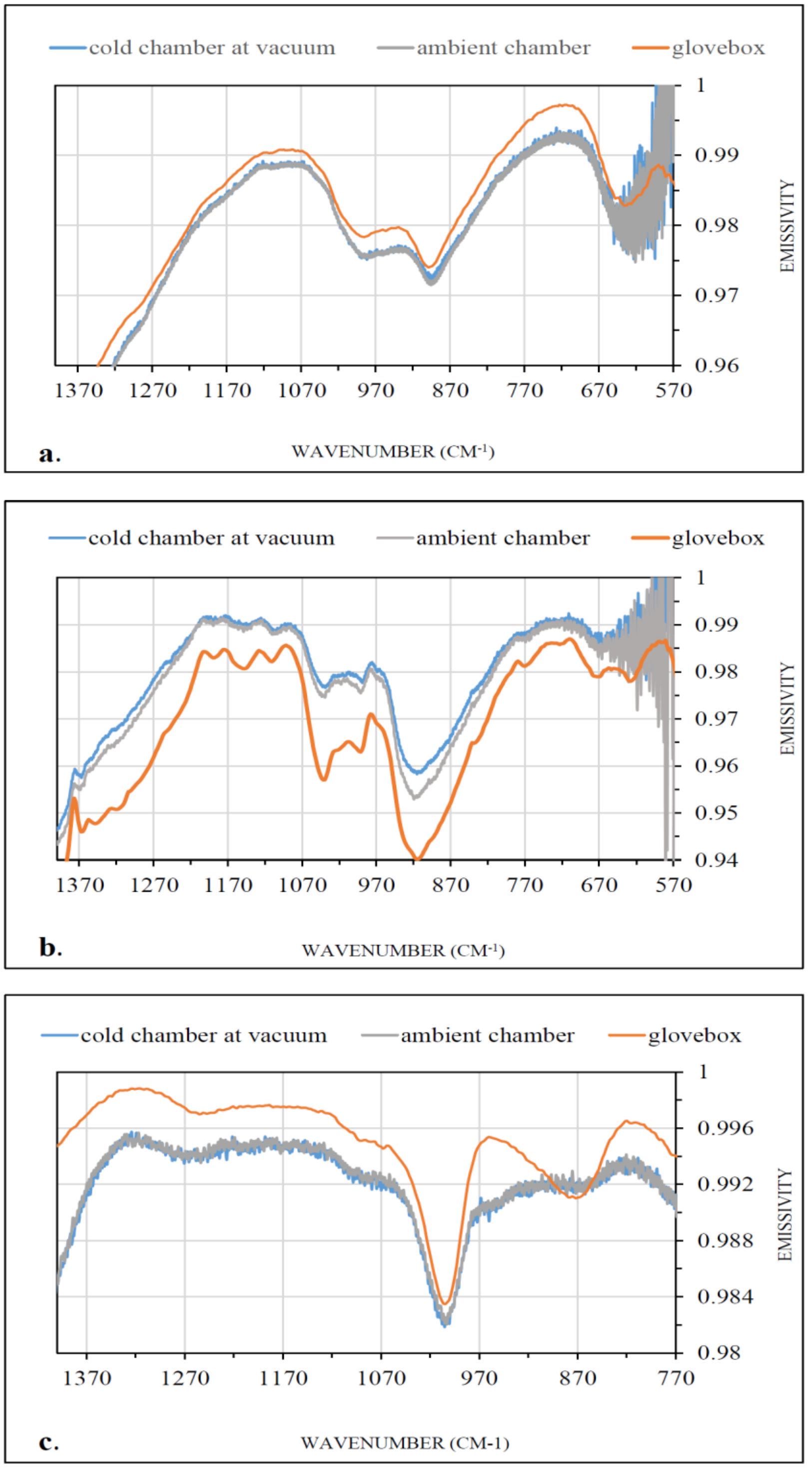


**Figure 2**. The spectral effects of adding pure KBr to (a) lizardite, (b) antigorite, and (c) silica.

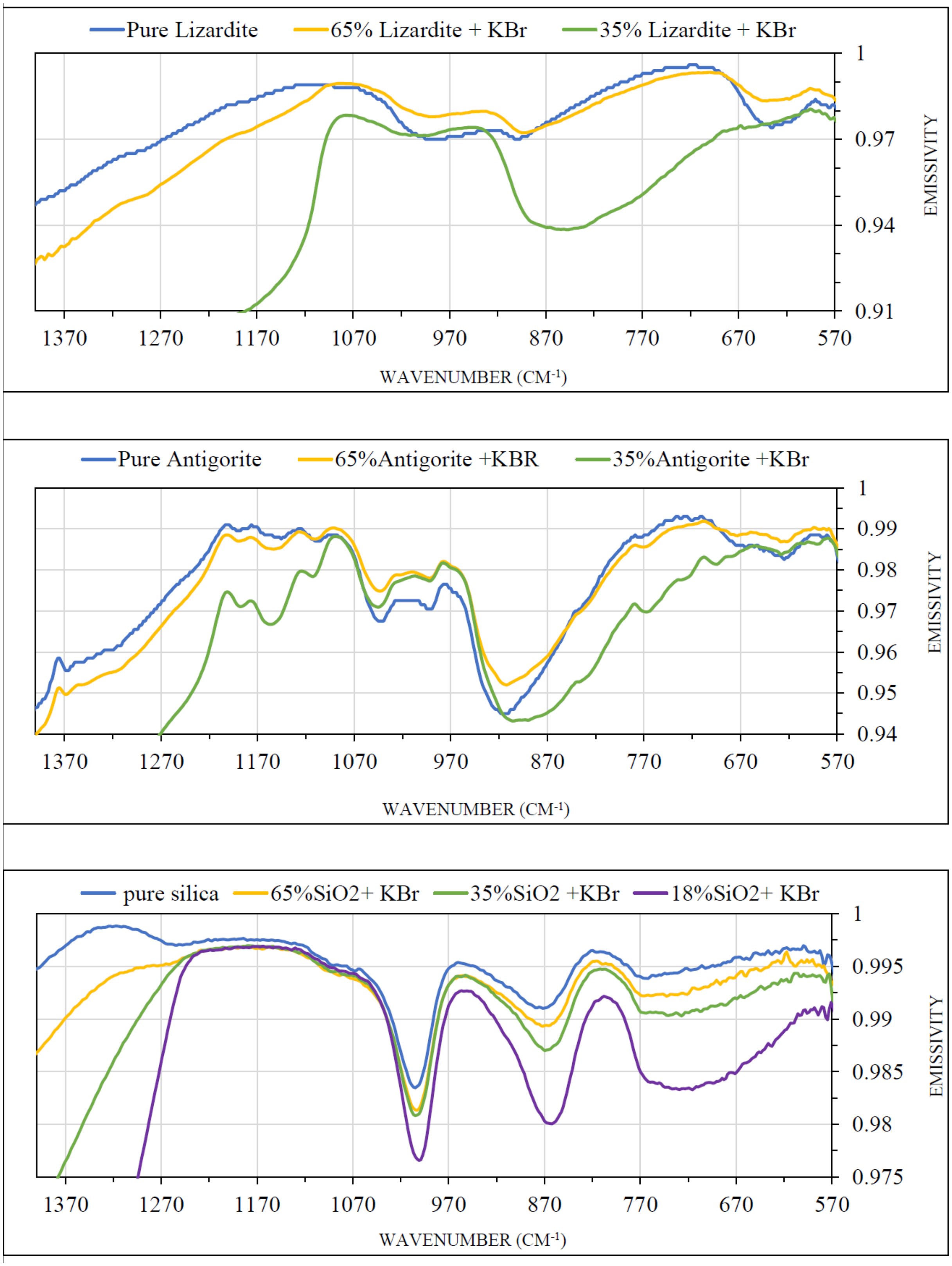


**Figure 3**. Mixtures of 98% KBr with 2% silicate show deepening of bands for silica and deepening and widening of bands for serpentines (lizardite and antigorite).

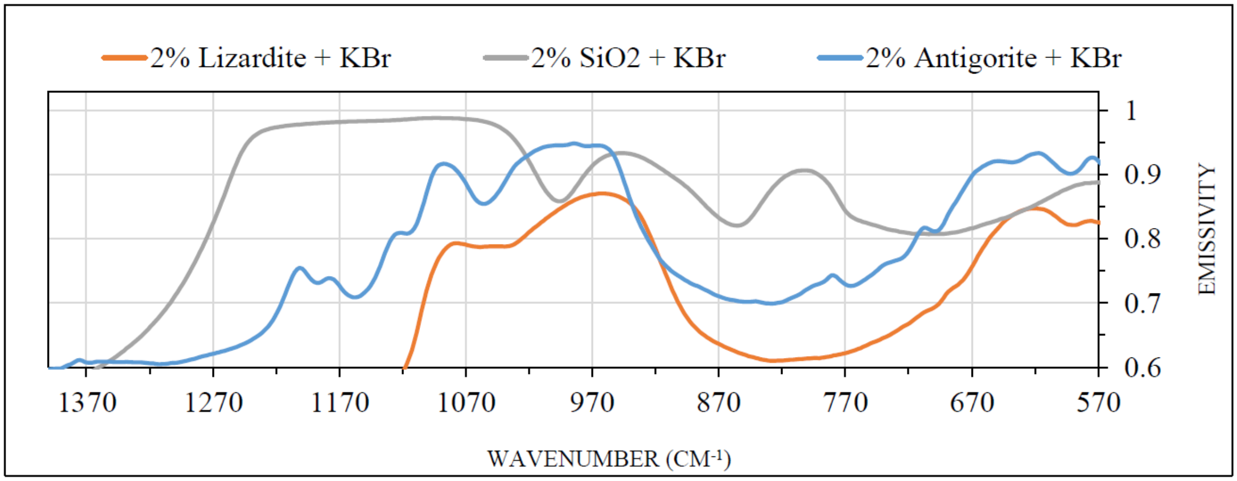


**Figure 4**. (a) Spectral changes for pure silica with increasing concentrations of KBr in the mixture to simulate porosity. (b) The feature centered at ~855 $cm^{-1}$ reported by Young et al (2015) had been compared to that of Mars silicates (Christensen et al., 2004) and was most consistent with pure lizardite and antigorite (Bishop et al., 2008), pointing to a meteoritic origin. However, the width and position of the primary feature closely matches that of the main feature in the silica, indicating that silica could be a viable composition in addition to the serpentines. Note that the region to the left in (b) is greyed out because CIRS data is too noisy to be reliable there.

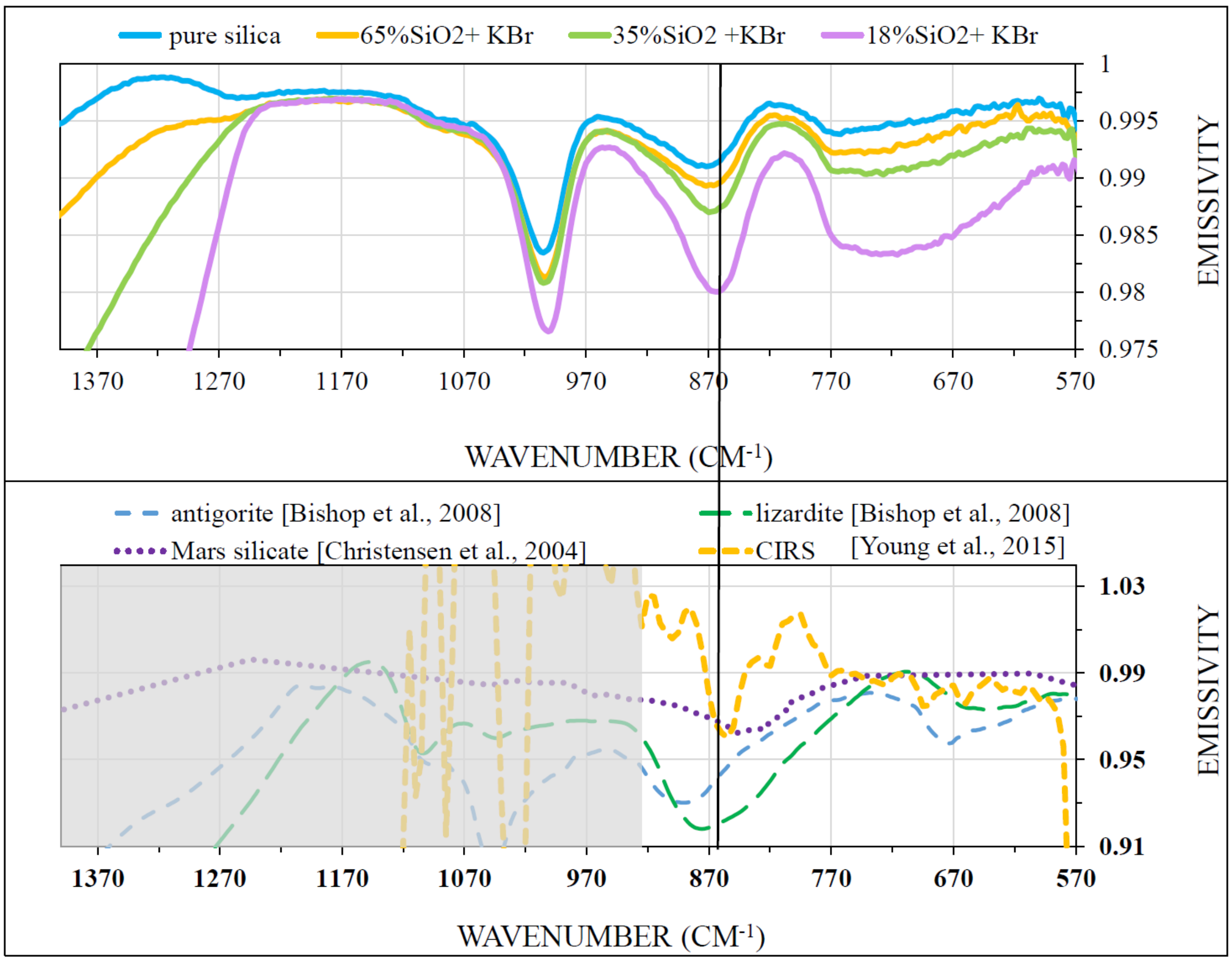


**Figure 5.** Reflectance minimum seen in silica under vacuum conditions at ~3 µm. Clark et al. (2012) observed a similarly located feature on Iapetus, although any water-bearing phase could also produce this band.

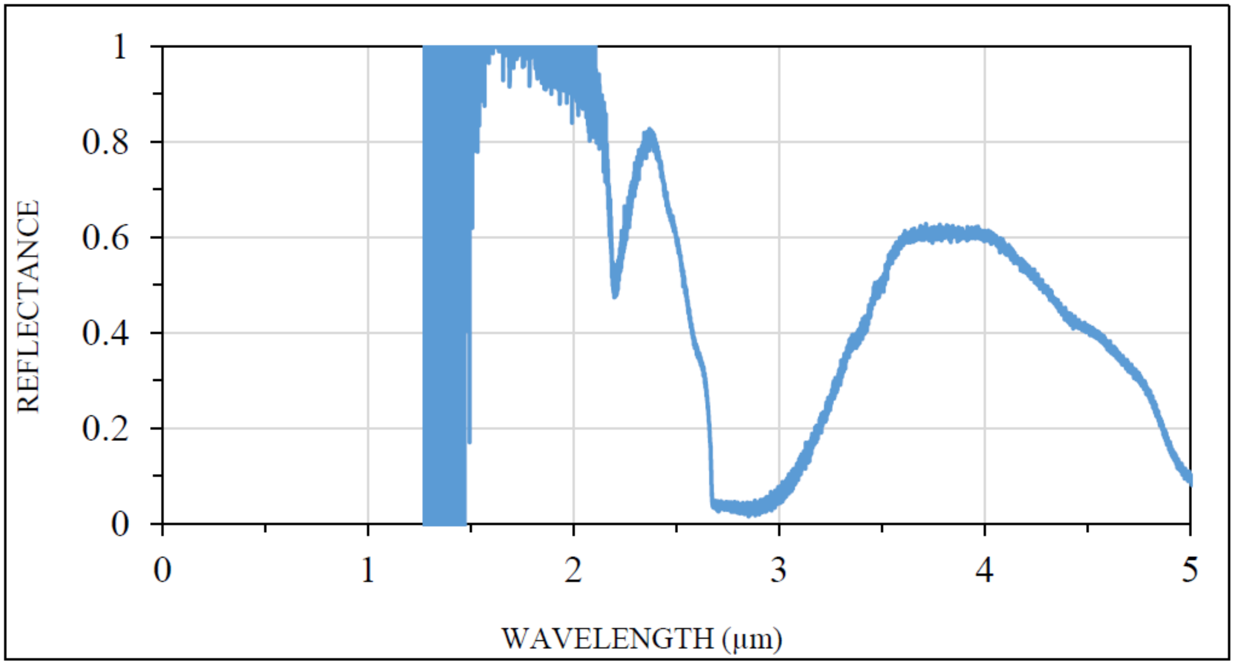


**Figure 6**. Spectral impact of adding nanophase $Fe_2O_3$ to (a) lizardite, (b) antigorite, and (c) silica.

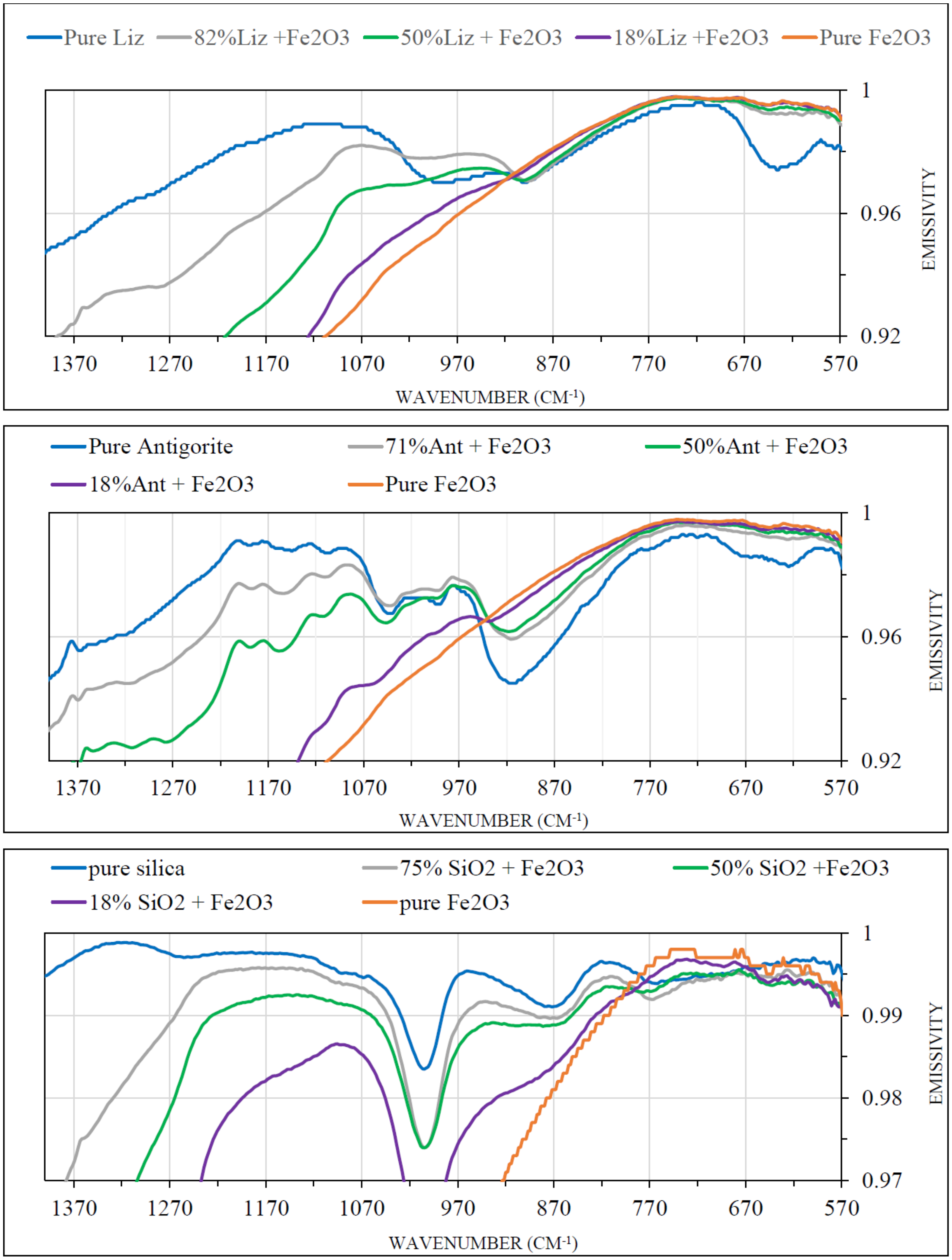
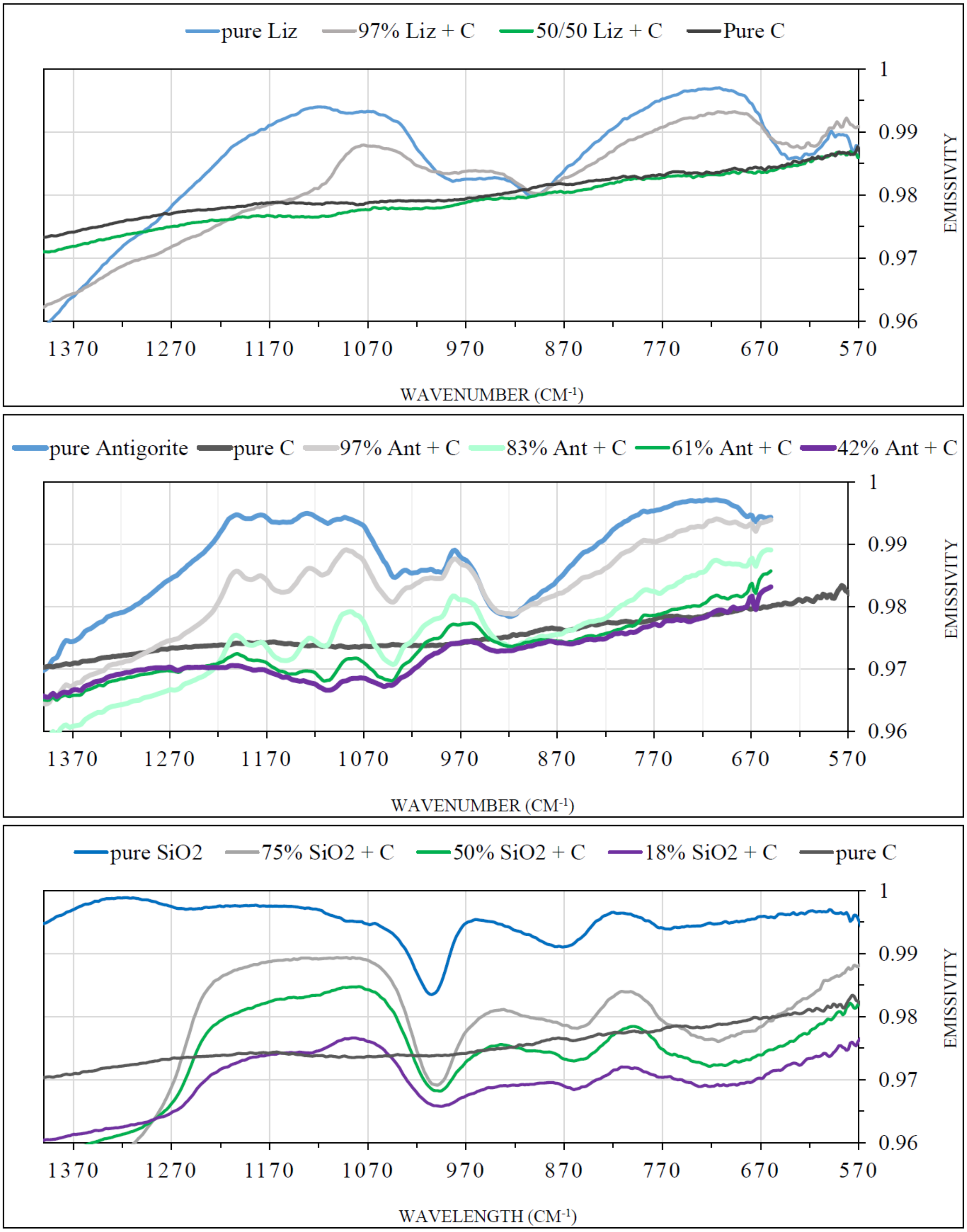


**Figure 7.** Spectral impact of nanophase C to (a) lizardite, (b) antigorite, and (c) silica.



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