EXPERIMENTAL PETROLOGY AND VOLCANOLOGY FOR DECIPHERING EXTRATERRESTRIAL VOLCANISM

ALESSANDRO PISELLO

Dipartimento di Fisica e Geologia, Università di Perugia, Via A. Pascoli snc, 06123 Perugia

INTRODUCTION

Since the beginning of human history, we have been able to get to know the world that surrounds us using five senses. Among them, sight is certainly one of the most powerful: not only colours, but even shapes and fabrics can be distinguished by using just our eyes.

We know that our eyes are nothing but spectroscopes detecting visible light, and our brain elaborates this information to distinguish shape, fabrics and material of an object. The brain can be therefore compared to an extremely sophisticated computer, which extensively uses machine-learning approaches.

Around the world and around the solar system, there are plenty of spectroscopes whose detection capability is certainly larger than our eye's one. In order to use spectroscopes as eyes and computers as brains to know the world which is surrounding us, it is however necessary to build up a memory made up of a database of spectroscopic data, which relates what something is to how does it reflect, emit, or absorb light.

By bringing these concepts to the study of planetary science, it is easy to understand how spectroscopic tools integrated within satellites can be extremely powerful to explore the earth and, possibly, the solar system, and the goal of this work is indeed to furnish a novel spectroscopic database of geologic materials.

Different launching projects involved spectroscopes-integrated satellites to explore the earth with the aim to observe our planet from a wider angle for a wide range of purposes. For what concerns planetary science, there is a great number of laboratories (Christensen *et al.*, 2000; Maturilli *et al.*, 2019; Veneranda *et al.*, 2020) whose aim is to build up large databases of rocks, to interpret data coming from other planets. Among the others, there have been projects whose aim was to furnish information on igneous rocks, in order to be able to reconstruct geological history of other planets (Feely & Christensen, 1999; Christensen *et al.*, 2000). The aim of this work is to add something that was only secondarily taken into account: the spectral response of silicate glasses. Glasses are amorphous materials, which exist within volcanic rocks and can constitute a large portion of them, like in obsidianaceous or hypohyaline rocks, or even in volcanic ashes and pyroclasts. Even if glasses lack long range order and their spectral response is often blurred and lacks of evident features, the aim of this work is to show how their spectra can still be interpreted to infer chemical features, that could tell something about the geological evolution of a planet.

Terrestrial planets in the Solar system (Mercury, Venus, and Mars) show relevant presence of volcanic structures and terrains, which in some case can cover large portions of the surface of such planets and of their natural satellite bodies (Wilson, 2009; Platz *et al.*, 2015; Byrne, 2019). In order to understand the history and the geology of these planets, it is therefore necessary to study how volcanism developed on them. For this purpose, within this study, we propose an innovative approach, which uses experimental petrology to produce glassy samples to be investigated, rather than individuating analogue sites on earth, in order to build up databases and possible interpretative models for igneous products on other planets.

This work presents original glasses produced for this study and their relative spectral response for a suite of different techniques: reflectance in the mid-infrared region, Raman spectroscopy, reflectance in the visible and near-infrared region, emissivity in the thermal-infrared region with varying temperature, and finally Brillouin spectroscopy.

This study wishes to enrich the experience of the scientific community, in order to feed the memory of computer-brains around the world and empower the capability of our spectroscopes-eyes to decipher the universe that is surrounding us.

SAMPLE PREPARATION METHODOLOGY

Glasses were synthesized starting from couples of natural rocks collected on different volcanic areas. For each of the collection site the two rocks that were collected represent respectively the mafic end-member (low-silica) and the felsic end- member (high-silica) for the geochemical characteristics of the area.

On the Vulcano Island (Aeolian Islands, Italy) a shoshonite (mafic) and a rhyolite (felsic) were collected. Vulcano is emplaced on an active subduction, and their products have a particularly alkaline character (De Astis *et al.*, 1997). Two other rocks were collected in the Snake River Plain area (USA, hereon SRP): a basalt (mafic) and again a rhyolite. This area is emplaced in an intraplate tectonic setting, and its products have a sub-alkaline character (Morgavi *et al.*, 2013). Two other rocks were collected in the area of Mount Etna (Sicily): a Hawaiiaite (mafic) and a Benmoreite (felsic). Products from this area show sub/alkaline to moderate alkaline character (Corsaro & Métrich, 2016). Finally, the two rocks that were collected on the Pantelleria island are a basalt and a pantellerite (peralkaline rhyolite). This latter island is built up on the Strait of Sicily Rift Zone, presenting a suite of peralkaline products (White *et al.*, 2009).

In this way, eight rocks were collected from areas having different chemical character and showing large chemical variability themselves. Such rocks were brought to the PVRG laboratory at the University of Perugia and pulverized down to grain size lower than 200 µm. Subsequently the powders were separately molten within a Nabertherm HT 04/17 furnace in air at supra-liquidus temperature (1400-1500 °C) for 4 hours and then quenched to a glass. Each glass was subsequently re-crushed and pulverized and the melting-quenching procedure was again repeated identically in order to obtain chemically homogeneous glasses (see Vetere *et al.*, 2015 for further details on the procedure). These glasses were again crushed to powders with size ranging from 500 to 200 µm to be mixed together in order to obtain a series of products with intermediate compositions. For the Vulcano series, powders were mixed to obtain five different mixtures having, in weight proportion, shoshonite:rhyolite ratios of 100:0, 70:30, 50:50, 30:70, and 0:100 (here named as S, S7, S5, S3, and RS, respectively). Similarly, for SRP, six mixtures were obtained by mixing end-members to obtain basalt:rhyolite ratios of 100:0, 80:20, 60:40, 40:60, 20:80 and 0:100 (here named B, B8, B6, B4, B2, and RB). For Etna products, three mixtures were obtained by mixing the hawaiite:benmoreite in the ratios 100:0, 50:50, and 0:100 (here named as M, M5, and V). Basalt and pantellerite from Pantelleria were mixed in the following ratios: 100:0, 75:25, 50:50, 25:75, and 0:100 (here named as BP, P7, P5, P3, and PP). Each mixture was again molten and quenched twice with the above-mentioned procedure. Finally, from each glass two specimens were prepared: a fine powder for spectral analysis, and bulk samples for EMPA and Raman characterization. Chemical characterization of each sample was indeed performed through Microprobe analyses, whose results are reported in Table 1.

MIR REFLECTANCE ANALYSES METHODS AND RESULTS

Samples spectroscopy was investigated at the Italian Institute for Astrophysics and Planetology (IAPS-INAF Rome) through FTIR analyses on powder, using a Fourier Transform spectrometer (Bruker Vertex 80) operating in the range of 2-14 µm with a spectral resolution of 2 cm^{-1} for SRP and Vulcano series, and 1 cm^{-1} for Etna and Pantelleria series. Samples consisted in < 38 µm powders.

In Fig. 1, all the acquired, smoothed and normalized spectra are reported, with spectra vertically stacked for the sake of visualization.

As expected, spectra lack of sharp features, and the shape of the spectra is rather a composition of sinuous bands, mainly defined by Reststrahlen effect, which in this case determines a large bulge for mafic compositions which evolves into two local maxima when increasing silica content. Such peaks are roughly centred at *ca.* 8.5 µm, 9.5 µm, and 12 µm, but their position seems to shift with evolving chemical composition. Qualitatively it is interesting how by looking at intermediate compositions we actually see how these peaks slowly arise when going from mafic towards felsic compositions. The observed local maximum at *ca.* 9.5-10 µm is usually recognised as the centre of characteristic Reststrahlen Bands (hereon RBpeak). The peak at *ca.* 12 µm generally increases with

increasing silica. However, this peak can be recognised as the Transparency Feature (hereon TF) and it is linked to grain size of the sample (Mustard & Hays, 1997).

Table 1 - Chemical composition of produced glasses. For each oxide, relative abundance in wt.% is reported.

|	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
B	48.32	1.82	16.23	11.7	0.18	7.62	10.81	2.57	0.36	0.34
B8	53.06	1.6	15.52	10.34	0.15	6.14	8.88	2.69	1.22	0.29
B6	57.75	1.35	14.94	8.8	0.13	4.68	7.08	2.83	2.08	0.24
B4	62.68	1.11	14.26	7.23	0.1	3.19	5.26	2.91	2.97	0.22
B2	67.49	0.88	13.71	5.6	0.08	1.7	3.42	3.05	3.84	0.17
RB	72.13	0.65	13.2	4.02	0.04	0.25	1.62	3.17	4.75	0.14
S	54.23	0.69	16.02	7.94	0.17	4.13	7.68	5.34	3.11	0.49
S7	60.38	0.51	15.37	6.12	0.14	2.94	5.71	4.84	3.63	0.29
S5	63.83	0.39	14.78	5.06	0.12	2.17	4.44	4.68	4.08	0.27
S3	67.57	0.29	14.28	3.89	0.1	1.4	3.09	4.49	4.51	0.17
RS	73.25	0.12	13.53	2.11	0.07	0.25	1.12	4.17	5.13	0.05
M	48.5	1.53	14.53	10.9	0.18	7.82	12.13	2.61	1.23	0.45
M5	55.36	1.36	15.64	8.23	0.17	4.58	7.62	4.34	2.2	0.37
V	63.25	1.2	17.11	5.05	0.17	1.45	3.18	4.92	3.22	0.35
BP	47.74	3.55	14.32	13.44	0.2	5.6	10.26	3.29	0.96	0.65
P2	52.28	2.73	13.2	11.94	0.17	4.16	7.67	4.17	1.88	0.58
P5	57.25	2.1	12.6	11.24	0.26	2.98	5.76	4.81	2.56	0.38
P7	63.51	1.24	11.4	9.76	0.27	1.52	3.03	5.69	3.53	0.2
PP	69.2	0.49	10.58	7.95	0.3	0.13	0.62	6.25	4.44	0.04
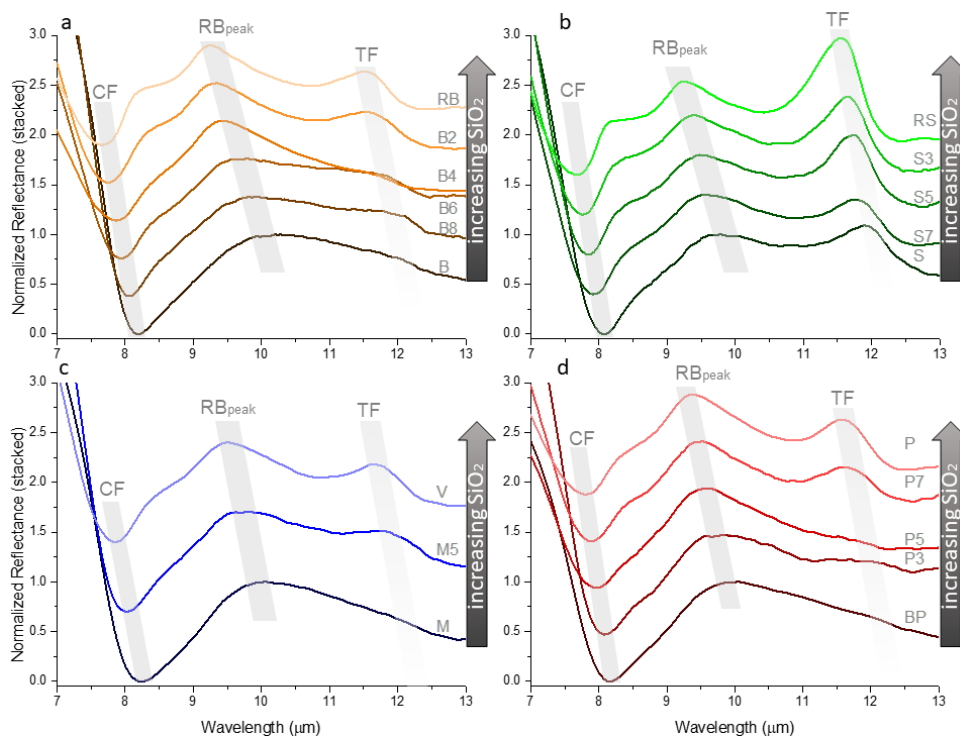
Fig. 1 - Sets of smoothed and normalized spectra divided for each of the sampling areas: a) SRP, b) Vulcano, c) Pantelleria and d) Etna. Spectra are vertically stacked to better visualize the evolving shape upwards, with the composition getting step by step enriched in silica until the felsic end-member at the top is reached. Rough positions of CF, RBpeak and TF are also enlightened.

Generally, the shape of the infrared spectrum in the 7-13 μm interval depends on two factors: Qn speciation (King *et al.*, 2004) of tetrahedral units, which is determined by the chemical composition of glasses, and the occurrence of TF, which is determined by the presence of fine material, but is considerably weaker for mafic glasses than for felsic glasses. All spectra present a local negative peak at *ca*. 8 μm, that can be recognised as the Christiansen Feature (hereon, CF), that is often used as a diagnostic feature (Cooper *et al.*, 2002).

CF, RBpeak and TF seem to gradually shift towards lower wavelengths with increasing silica content. Spectral shift of spectra was parameterized by means of the shift of CF and RBpeak, finding a good correlation with content of silica and main network formers. CF shift was observed to be particularly linear, and therefore we have proposed a model to be used to determine chemical composition of glassy occurrences, for which it was observed a promising correlation coefficient of 0.985 for SiO_2 mol.% and of 0.989 for $SiO_2+Al_2O_3+TiO_2$ mol.%. However, such model was also applied to natural rocks, showing good results for constrains on intermediate and felsic compositions.

VNIR REFLECTANCE ANALYSES METHODS AND RESULTS

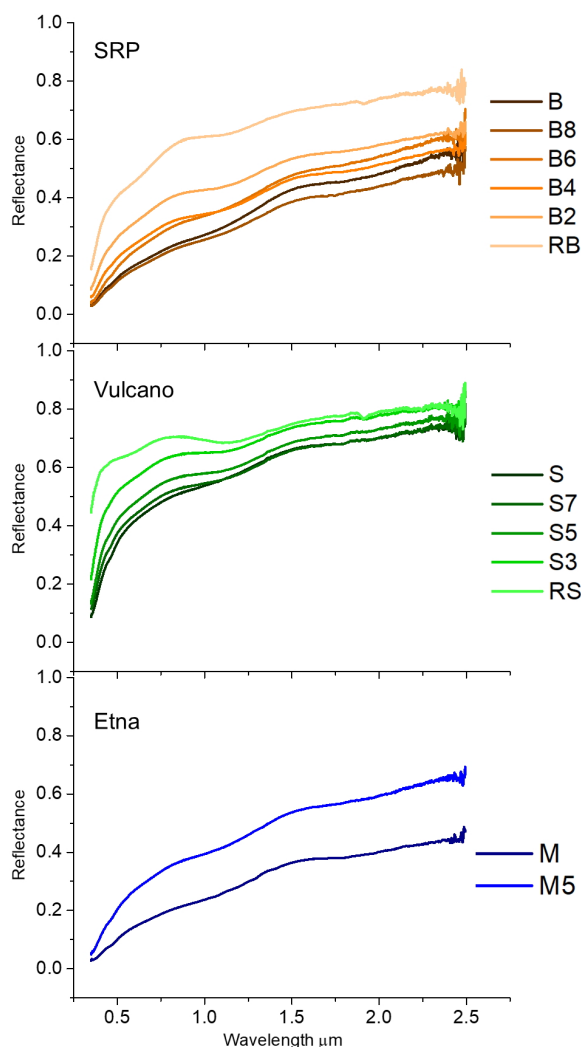Three series of samples have been characterized at the C-Lab of the Institute for Space Astrophysics and Planetology (IAPS-INAF) in Rome, Italy. The instrument we used is described in detail in De Angelis *et al.* (2014) and consists of a Field Spec-4 spectrometer, having a detection spectral range of 0.35-2.5 μm. Samples consisted in < 38 μm powders.

All spectra acquired with FieldSpec 4 are reported in Fig. 2. Spectra belonging to mafic products have lower albedo and steeper slope, and the albedo is getting higher and the slope getting flattened with increasing silica content and decreasing iron content.

Alkaline mafic products show generally larger albedo and flatter slopes, even for more mafic products, if compared to the other mafic samples. All spectra show three weak absorption bands, two of them relative to Fe^{2+} absorption located at *ca*. 1 and *ca*. 2 μm (Carli *et al.*, 2016) and another small absorption shoulder at *ca*. 0.45 μm, particularly evident only for the Vulcano series. In general observation can be summarized as follows:

i) Homogeneously fine powders of silicate glasses always present positive slopes. For sub-alkaline series, glasses presenting mafic composition have a steeper slope if compared to felsic composition, differently from alkaline series, which have generally a flatter aspect.

ii) Variation of albedo is similar for all series, with mafic glasses being always darker than felsic glasses.

iii) Silicate glasses show two weak absorptions, at *ca*. 1.15 μm and 1.93 μm. A small shoulder at *ca*. 0.45 μm is observable for felsic compositions, in particular for alkaline series.

Fig. 2 - Spectral dataset of reflectance within VNIR range for three series.

RAMAN SPECTRAL ANALYSES METHODS AND RESULTS

Raman spectra were acquired at the Mineralogical State Collection of Munich using a XploRa-Raman-System, calibrated on a silicon standard equipped with a 532 nm laser and a microscope to explore the area to be

analysed. The laser beam was projected on the surface through a 100× lens on a *ca*. 2 μm spot of polished sample, with a power of 7.15 mW and an exposure time of 60 seconds (3 times). Laser beam was focused 6 microns below the surface of the glass, for maximising Raman signal.

Raman spectra were smoothed and refined as reported in Fig. 3, which shows how the spectral appearance is determined by the different spectral shape within three different areas: one between *ca*. 200 cm^{-1} and 650 cm cm^{-1}, an intermediate one between *ca*. 650 cm^{-1} and 850 cm^{1}, and a third one between *ca*. 850 cm^{-1} and 1300 cm^{1}.

We have revised the two models for the chemical characterization of silicate glasses using Raman spectroscopy proposed by Di Genova *et al.* (2015) and González-García *et al.* (2020). Both methodologies work well with the determination of chemical parameters more or less directly connected to the cations involved in the building of tetrahedral network (mainly Si, Al and Ti), and the parameters expressed as SM and SCFM. In this work we have offered parameters based on our database to apply such models to retrieve chemical information of glasses.
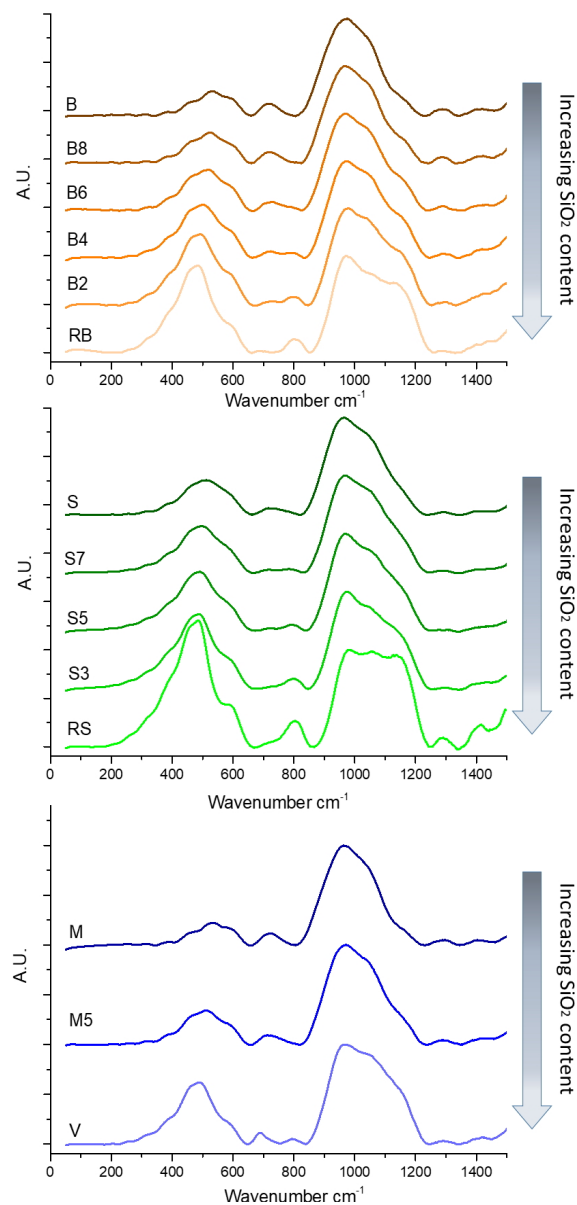
Fig. 3 - Corrected, smoothed and baseline-subtracted Raman spectra for three series: SRP at the top, Vulcano in the middle and Etna at the bottom. Spectra are vertically stacked for the sake of visualization.

FTIR EMISSIVITY ANALYSES METHODS AND RESULTS

Spectroscopic characterization was performed at PSL (Planetary Spectroscopy Laboratory) at the DLR, Berlin, Germany (Maturilli *et al*., 2019). At PSL, two identical Bruker VERTEX 80 V Fourier transform infrared (FTIR) spectrometers are operating and equipped for spectral measurements. Bidirectional reflectance and transmittance measurements can be obtained in the extended wavelength ranging from 0.4 to 16 μm. Emissivity can be measured in the 1.4-16 μm spectral range. The high-temperature chamber of the Vertex 80 V allows heating (*via* an induction coil system) the samples to temperatures up to *ca*. 700 °C under vacuum conditions. Reflectance measurements were performed on our experimental samples with an evacuated spectrometer on samples at room temperature of 20 °C and the same samples after heating (above 500 °C). Emissivity measurements were performed in vacuum on heated powders with experimental temperatures of 150, 300, 450 and 600 °C. Samples consisted in < 25 μm powders.

In Fig. 4, as an example, all Vulcano emissivity spectra are reported. In general, all spectra present a peak of maximum emissivity (minimum reflectance) at *ca*. 8 μm, where emissivity is close to the unity and reflectance is close to zero. We are able to refer at this emissivity maximum/reflectance minimum as the Christiansen Feature (hereon CF). CF shifts to lower wavelengths with increasing silica content. Into a lesser extent, also temperature of acquisition of the spectra influences the shift of the whole spectra, included CF.
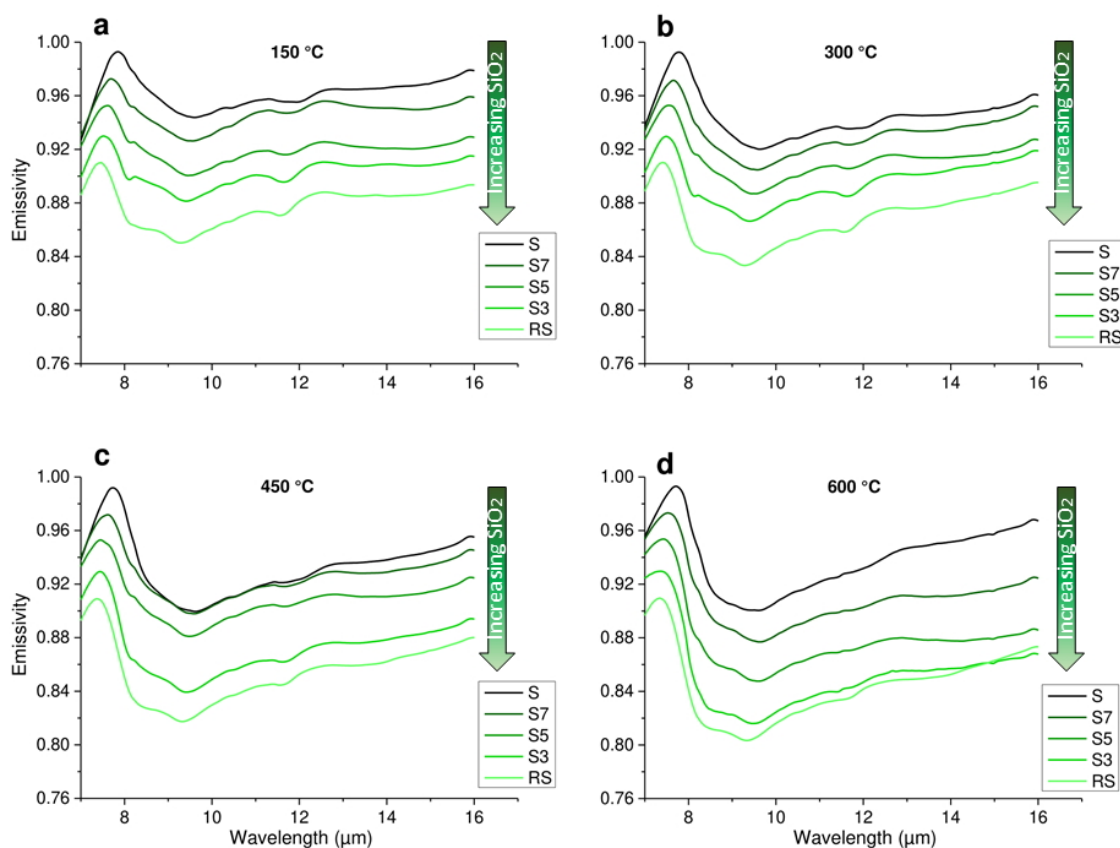

Fig. 4 - Emissivity spectra collected for the Vulcano (S-RS) series. Each subfigure represents the emissivity of the entire series of glasses, collected at (a) 150 °C, (b) 300 °C, (c) 450 °C, and (d) 600 °C. Spectra are stacked in order to better highlight differences.

In general, it was observed how for silicate glasses emissivity and reflectance features correlate to each other, and the joint observation of shift and shape of such spectra can give us information on the degree of evolution of a magma (expressed as silica content), and on the alkaline character of the composition. Such information, all together, will contribute to enhance our capability to observe, interpret and lucubrate on the type of volcanism occurring on other planetary bodies in the Solar System.

BRILLOUIN SPECTROSCOPY ANALYSES, METHODS AND RESULTS

For Brillouin characterization, glass fragments of *ca*. 5 mm diameter were used. Brillouin light scattering (BLS) spectroscopy detects light that is inelastically scattered by acoustic waves. The frequency shift between the incident and the scattered light was analysed by a high resolution and high contrast Sandercock-type tandem 3+3 pass Fabry-Pérot interferometer, at the Department of Physics and Geology of the University of Perugia (Scarponi *et al.*, 2017). The polarization of incident and scattered light was selected perpendicular to the scattering plane (VV scattering geometry), so that the experiment only revealed light that interacted with longitudinal acoustic

waves. Measurements were performed at ambient temperature using a 532 nm green laser in backscattering geometry.

Spectra are reported in Fig. 5, and the results indicate that the frequency position of the Brillouin peak depends on a complex interplay between network-former (mainly Si, Al, and Ti) and network-modifier cations. SCFM parameter (Walter & Salisbury, 1989) describes a masterplot with shifting Brillouin peak for all the investigated glasses.
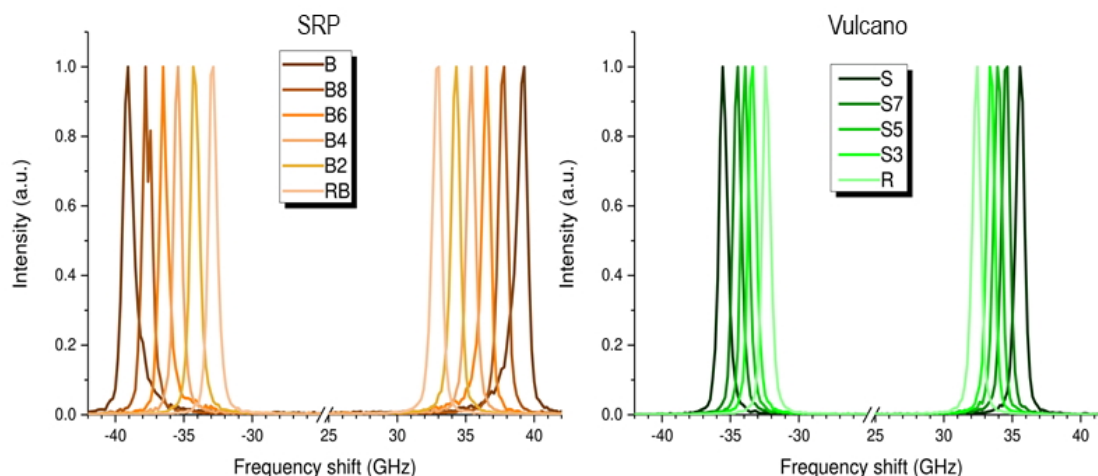

Fig. 5 - Brillouin spectra measured for two series of glasses.

These findings can have important implications for the use of Brillouin spectroscopy as a new tool for the study of Earth and planetary materials. In particular, it would be important to increase the dataset of Brillouin spectra for silicate melts erupted on the Earth in different geologic times and areas. This would provide a solid basis of comparison for possible data retrieved from planetary surfaces during possible future space missions equipped with Brillouin systems. The possibility of retrieving structural information, physical characterization and/or the composition of natural glasses from Brillouin measurements represents, indeed, a very promising, still rapid method, that might fuel our understanding of Earth and planetary evolution.

CONCLUSIONS, OUTLOOKS AND PERSPECTIVES

The different studies which were performed have shown how for silicate glasses, whose spectral response is often blurred if compared to crystalline phases, it is possible to perform parameterizations allowing to systematically retrieve information about chemical features. In particular, it was observed how the different applied techniques give information that can be grouped in three major groups:

i) techniques and spectral ranges which characterize a rock by taking into account features linked to T-O bonds, where T mainly represents Si, but also Al and Ti. These are all the techniques which take into account mid-infrared (or thermal infrared) or Raman shift;

ii) techniques which give information concerning iron oxides arrangement through reflectance in the VNIR range;

iii) techniques concerning physical properties as a proxy for chemical composition by means of SCFM, such as Brillouin spectroscopy.

For what concerns the first group, it was observed how there was a good correlation between spectral features of silicate glasses and chemical composition, especially when accounting network former cations, such as Si, Al, and Ti. The abundance of such elements in magmatic products is generated by the degree of differentiation that a magma has been gone through during its geological history, and therefore it indicates the degree of evolution of a volcanic system. The majority of planetary products are considered to have mafic or ultramafic composition,

and they are therefore considered to be primitive rather than evolved. Nevertheless, we have observed how the occurrence, within localized areas, of possible episodes of advanced differentiation of magmatic products represents a constant exception which is of extreme interest to constrain geological history of extraterrestrial volcanism.

Spectral information acquired within this study from Raman and mid-infrared spectroscopy, will certainly be useful for the interpretation of data that will be gathered during future missions, in particular ESA's BepiColombo (Rothery *et al.*, 2020) and ESA's ExoMars (Vago *et al.*, 2013).

Concerning VNIR, almost featureless spectra are indeed observed on planetary bodies (Cannon *et al.*, 2017) and the information gathered here will be surely useful for the Ma_miss apparatus on the Rosalind Franklin rover of the ExoMars mission, especially because this mission will be able to integrate VNIR results with Raman measurements.

For what concerns the last proposed Brillouin study it has already been said how, currently, no mission carrying such technology is planned, but the fact that this one is the only spectral technique which leads to a very sharp spectral response of silicate glasses might eventually result in a future tool for the identification of volcanic amorphous materials, on Earth and other planets.

The long-term outlook of this thesis is to promote a methodology of interpretation of spatial data which would not be based on the bare comparison of spectra taken from other planets with spectra of rocks which are common on the Earth, but rather with lab-made rocks which can be produced by varying systematically the diagnostic characteristics. In this way, exhaustive databases will be built where the features are interpreted from both a physical and chemical point of view. The idea of this project is that in a near future, with the creation of such databases for planetary systems, interpretation and decoding of spectroscopic data will be possible to directly link them to specific chemical compositions and textural characteristics.

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