

Real-time measurement of dielectric properties of lunar regolith simulants

C. O'Connell

c.o'connell@surrey.ac.uk

University of Surrey

B. Garcia-Bañós

Universitat Politècnica de València

J. M. Catalá-Civera

Universitat Politècnica de València

J. D. Gutiérrez-Cano

Universitat Politècnica de València

A. Lucca Fabris

University of Surrey

S. Lim

University of Surrey

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Abstract

Establishing a permanent settlement on the Moon will require infrastructure capable of protecting astronauts and equipment from the abrasive effects of lunar dust, radiation, micrometeorite impacts, and extreme thermal fluctuations. Lunar regolith is a dielectric material that can be efficiently heated to its melting temperature using microwave energy, providing an abundant, and cost-effective feedstock for energy-efficient construction. In this study, we measured the complex permittivity of two lunar regolith simulants, the mare simulant JSC-1A and the highland simulant NU-LHT-4M, from room temperature to melting. A dual-mode permittivity measurement technique enabled simultaneous measurement of complex permittivity while the simulant was heated using a single-mode microwave system operating at approximately 2.45 GHz. For both simulants, the real and imaginary parts of the permittivity increases significantly with temperature. The resulting temperature-dependent data will enable more accurate numerical modelling of microwave heating of lunar regolith, supporting the optimisation of microwave heating devices and improved energy efficiency in lunar ISRU processing.

1. Introduction

Space agencies are increasingly focused on developing surface infrastructure on the Moon to enable repeated landings and sustained operations in the same region. This will require constructing affordable infrastructure, such as landing pads and roadways, to mitigate rocket-exhaust-induced regolith erosion, limit dust dispersion, and protect surface assets, including solar arrays and lunar rovers, from contamination and mechanical wear. Regolith-based infrastructure may also play a critical role in safeguarding astronauts from dust exposure, micrometeorite impacts and ionising radiation.

The dielectric properties of lunar regolith make it a promising material for thermal processing using microwave energy¹. Whilst conventional furnace heating methods are limited by the regolith's very poor thermal conductivity, microwave processing offers volumetric heating, enabling energy to be deposited directly into the bulk of the material, reducing the need to heat the surrounding furnace structure and potentially improving energy efficiency. This method is particularly advantageous for In Situ Resource Utilisation (ISRU) applications due to its rapid heating, reduced processing time, minimal mechanical complexity, and the absence of consumable binders or additives required from Earth. The resulting sintered or molten regolith offers a readily available, sustainable and cost-effective feedstock for in situ construction on the lunar surface^{1,2}.

Numerical modelling of microwave heating in lunar regolith enables detailed analysis and optimisation of the process while providing an efficient means of approximating lunar operating conditions, including vacuum and the high-temperatures required for melting, which are technically challenging and costly to reproduce experimentally. Accurate simulation of microwave heating up to melting temperatures requires reliable temperature-dependent thermophysical and dielectric properties. Among these parameters, complex permittivity is particularly important because it governs the interaction between the material and the electromagnetic field and therefore strongly influences microwave energy absorption and heating efficiency.

The real part of complex permittivity, ϵ' (also known as the dielectric constant), describes how much electrical energy from an external electromagnetic field is stored in the material. The imaginary part of permittivity, ϵ'' (also known as the loss factor or dielectric loss), is a measure of how much energy is dissipated as heat in the material³. In microwave heating, this 'loss' is desirable because it heats the material.

The complex permittivity of lunar regolith simulants varies significantly with temperature. Temperature-resolved dielectric measurements are therefore essential for improving the predictive accuracy of numerical microwave-heating models, as previous studies have assumed constant permittivity in the absence of temperature-dependent data⁴.

The Cavity Perturbation Method (CPM) has previously been applied to characterise lunar regolith simulants at ambient temperature, as well as more recently to investigate their temperature-dependent dielectric properties up to melting conditions⁵⁻⁷. In these studies, samples were heated to a prescribed temperature in a conventional furnace, transferred to a microwave cavity for complex-permittivity measurement, and then rapidly returned to the furnace for heating to the next target temperature. As some cooling inevitably occurred during sample transfer and measurement, the actual measurement temperature was estimated from previously determined cooling rates.

In this study, we report for the first time measurements of the real and imaginary components of the complex permittivity of the lunar mare regolith simulant JSC-1A and the highland regolith simulant NU-LHT-4M during direct microwave heating from room temperature to the melting conditions.

Measuring dielectric properties under direct microwave heating provides a more representative description of the interaction between microwave fields and lunar regolith simulants than measurements conducted under conventional furnace heating. This approach eliminates uncertainties associated with sample transfer and cooling and allows continuous monitoring of changes in complex permittivity as thermal and phase transformations occur. Moreover, direct microwave heating may reveal dielectric behaviour that is not fully captured under conventional heating because of differences in heating rate, temperature distribution, and electromagnetic field–material interactions.

2. Method

2.1 Simulants

Silicate minerals make up over 90% of the lunar surface, with the dominant mineral phases being plagioclase feldspar (Ca, Na)(Al, Si)₄O₈, pyroxene (Ca, Fe, Mg)₂Si₂O₆ and olivine (Mg, Fe)₂SiO₄. Lunar regolith also contains substantial amounts of glass derived from volcanic activity and impact melts. In addition to these glasses, agglutinates, which are the major source of glass in lunar regolith⁸, consist of aggregates of soil, rock and mineral fragments welded together by glass formed during micrometeoroid impacts⁹.

In this study, two high-fidelity lunar simulants were used: the low-Ti mare simulant JSC-1A and the highland simulant NU-LHT-4M. Their compositions are given in Tables 1 and 2, together with those of returned Apollo lunar regolith samples.

Table 1
Lunar regolith simulant major mineralogy (wt%)^{6,9}

Simulant	Region	Plagioclase Feldspar	Pyroxene	Anorthosite	Norite	Olivine	Ilmenite	Glass	Agglutinate
JSC-1A ⁹	Low-Ti Mare	37.83	19.43			12.44	0.11	26.67	
Apollo, averaged ⁹	Low-Ti Mare	28.46	24.67			6.89	3.86	32.98	
NU-LHT-4M ^{6,9}	Highlands			42.49 ⁹	19.86 ⁹	5.32 ⁹	0.6 ⁹	4.53 ⁹ 0 ⁶	27 ⁹ 35 ⁶
Apollo, averaged ⁹	Highlands	59.05	7.92			5.11	0.15	27.77	

Table 2
Lunar regolith simulant bulk chemistry (wt%)^{6,9}

Simulant	Region	SiO ₂	TiO ₂	Al ₂ O ₃	FeO	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	Cr ₂ O ₃	MnO	P ₂ O ₅
JSC-1A ⁹	Low-Ti Mare	46.8	1.8	16.4	11.4		8.7	10.0	3.2	0.8			
JSC-1A ⁶	Low-Ti Mare	46.80	1.84	16.40		12.70	8.72	10.00	3.19	0.83	0.01	0.19	0.70
Apollo, averaged ⁹	Low-Ti Mare	45.62	2.09	13.2	16.45		10.19	10.65	0.42	0.19			
NU-LHT-4M ⁹	Highlands	47.2	0.4	23.5	4.3		8.7	12.8	1.5	0.2			
NU-LHT-4M ⁶	Highlands	47.47	0.41	23.59		4.78	8.74	12.96	1.50	0.16	0.11	0.07	0.10
Apollo, averaged ⁹	Highlands	45.62	0.84	24.47	6.6		6.98	14.65	0.48	0.2			

2.2 Method of measurement

The Cavity Perturbation Method (CPM) was implemented in a dual-mode microwave cavity to enable the simultaneous microwave heating of lunar regolith simulants and the in-situ measurement of their complex permittivity^{10,11}. This combined approach was achieved using two independent microwave sources, as illustrated in Fig. 1(a). The samples were heated in a single mode microwave with power supplied at 2.45 GHz and up to 150 W through a horizontal probe inserted into the cavity sidewall, while dielectric characterisation was performed at approximately 2.3 GHz using a vertical probe coupled through the cavity base. A cross-coupling filter provided high isolation between the two microwave sources, thereby minimising interference between the heating and measurement systems.

The complex permittivity was derived using an enhanced CPM from the relative shifts in the resonant frequency and quality factor between the unloaded and sample-loaded cavity¹⁰.

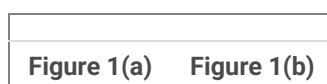


Figure 1. Dual mode cylindrical cavity for simultaneous microwave heating and measurement of complex permittivity¹⁰. (a) Schematic view. Labelled parts are – 1: microwave heating port, 2: microwave measurement port, 3: quartz tube, 4: regolith simulant, 5: infrared thermometer. (b) Experimental system located at the ITACA Institute, Universitat Politècnica de València. Labelled parts are – 1: microwave heating port, 2: optical camera, 3: infrared thermometer, 4: N₂ gas inlet to quartz tube.

Before testing, the powdered samples were heated in a vacuum furnace to remove surface-bound moisture. Approximately 2.2 g of each sample was loaded into a high-purity quartz tube with an internal diameter of 10 mm. The powders were lightly compacted by manual tapping without external pressure, resulting in bulk densities of approximately 1.86 g cm⁻³ for JSC-1A and 1.74 g cm⁻³ for NU-LHT-4M. The samples were subsequently heated under a nitrogen atmosphere to maintain inert processing conditions.

The sample temperature was monitored externally using a pyrometer with a stated accuracy of 0.1°C. The instrument had a maximum calibration temperature of 1215°C, whereas the quartz tubes had a short-term operating temperature limit of 1400°C.

The bulk sample temperature was determined using a calibration procedure that integrated data from four independent techniques to establish a correlation between the measured surface temperature and the actual bulk temperature during microwave heating. Differential thermal analysis (DTA) and Raman spectroscopy measurements of calibration materials were compared with the corresponding dielectric-permittivity data. The agreement between the detected changes and phase transitions occurring at known temperatures was used to validate the temperature correlation^{11,12}.

To ensure efficient power transfer throughout sample processing, an automatic control system dynamically adjusted the operating frequency and input power to maintain a heating rate of approximately $0.25^{\circ}\text{C s}^{-1}$. The position of the coupling antenna could also be adjusted slightly to preserve efficient coupling and a stable heating rate as the dielectric properties of the sample evolved with temperature. The estimated measurement uncertainties were 3% for the real component of permittivity, ϵ' , and 10% for its imaginary component, ϵ'' ¹⁰.

The real and imaginary permittivity of the lunar mare simulant JSC-1A and the lunar highland simulant NU-LHT-4M were measured from room temperature to their respective melting temperatures, and subsequently during cooling back to ambient.

3. Results

Figure 2(a) and 3(a) show the real permittivity, ϵ' , and imaginary permittivity, ϵ'' , of three JSC-1A lunar mare samples and three NU-LHT-4M lunar highland samples. The glass transition temperature (T_g), and onset of melting temperature (T_m) are from recent Differential Scanning Calorimetry (DSC) studies¹³.

Room temperature dielectric measurements obtained in this study are consistent with previously published data for Apollo mare and highland samples, as well as for mare and highland regolith simulants measured at comparable frequency and density^{5,6,14–17}.

The results demonstrate that under microwave heating, ϵ' and ϵ'' of both lunar simulants are strongly temperature dependent, resulting in significantly enhanced microwave absorption at higher temperatures. Previous furnace heating studies have also reported increasing ϵ' and ϵ'' with temperature^{5–7}, although the temperature-dependent dielectric response differs markedly from that observed during microwave heating.

3.1 Differences between JSC-1A and NU-LHT-4M

Figure 2(a) and 3(a) show that both ϵ' and ϵ'' of JSC-1A are higher than those of NU-LHT-4M across the measured temperature range, particularly ϵ'' . These differences are likely to reflect variations in bulk density and mineral composition between the simulants. Tables 1 and 2 show that JSC-1A contains a greater abundance of Fe and Ti-bearing phases. The higher Fe and Ti content contributes to the greater bulk density of JSC-1A relative to NU-LHT-4M, and previous studies have shown that at ambient temperature, ϵ' is a function of bulk density^{14–16,18}. These studies have also shown that ϵ'' increases with Fe and Ti content^{14–16,18}, reflecting enhanced dielectric loss mechanisms associated with Fe and Ti phases such as ilmenite, resulting in greater dissipation of microwave energy as heat within the material⁷.

Figure 2(a)	Figure 3(a)
Figure 2(b)	Figure 3(b)
Figure 2(c)	Figure 3(c)
Figure 2(d)	Figure 3(d)

Figure. 2. Temperature-dependent properties of JSC-1A during heating from room temperature to melting temperature. Results are shown for three independent samples (Tests 1–3). T_m and T_g are from¹³. (a) ϵ' (solid line) and ϵ'' (dashed line) on heating; (b) Arrhenius plots of $\ln(\epsilon'')$ as a function of $(1000/\text{Temperature})$ for three samples, with a linear fit to the selected region (pink dashed line); (c) temperature increase with time; (d) microwave power absorbed by the samples on heating.

Figure. 3. Temperature-dependent properties of NU-LHT-4M during heating from room temperature to melting temperature. Results are shown for three independent samples (Tests 1–3). T_m and T_g are from¹³. (a) ϵ' (solid line) and ϵ'' (dashed line) on heating; (b) Arrhenius plots of $\ln(\epsilon'')$ as a function of $(1000/\text{Temperature})$ for three samples, with a linear fit to the selected region (pink dashed line); (c) temperature increase with time; (d) microwave power absorbed by the samples on heating.

3.2 JSC-1A on heating

JSC-1A exhibits a gradual, steady increase in both ϵ' and ϵ'' from ambient temperature to approximately 470°C, after which the rate of increase in permittivity accelerates, as shown in Fig. 2(a). An Arrhenius plot of ϵ'' as a function of temperature in Fig. 2(b) indicates that below 470°C, ϵ'' is weakly temperature dependent. However, in the region between approximately 470°C and T_g at 650°C, a linear fit suggests a single dominant thermally activated loss mechanism. Close to the melting temperature, a rapid increase is observed in both ϵ' and ϵ'' , with the sharp increase in ϵ'' indicating the onset of melting^{6,11}, which is also supported by the DSC data¹³. During the microwave heating process, ϵ' more than triples, while ϵ'' increases by more than 100-fold. The heating rate was maintained at approximately 0.25°C s⁻¹ throughout the heating process, as shown in Fig. 2(c).

The nonlinear increase in the power absorbed by the sample with temperature, shown in Fig. 2(d), is consistent with enhanced microwave absorption as the simulant temperature rises.

3.3 NU-LHT-4M on heating

The permittivity response of the NU-LHT-4M samples differs markedly from that of JSC-1A. From ambient temperature to a critical temperature, which varies slightly between 750°C and 800°C across samples, both ϵ' and ϵ'' increase gradually and steadily. An Arrhenius plot, Fig. 3(b), indicates that below approximately 520°C, ϵ'' is weakly temperature dependent. However, in the region between approximately 520°C and T_g at 780°C, a linear fit suggests a single dominant thermally activated loss mechanism.

At the critical temperature, a sudden and rapid increase in both ϵ' and ϵ'' is observed. Notably, ϵ'' increases by approximately an order of magnitude, accompanied by a temperature rise of more than 100°C, as shown in Fig. 3(a) and Fig. 3(c). The onset of this rapid increase in permittivity is preceded by a temperature decrease of approximately 10°C and a sharp increase in microwave power absorption by the material, as shown in Fig. 3(d). This behaviour is consistent with a change in dielectric loss mechanism, resulting in a substantial increase in volumetric microwave heating.

Above this critical temperature, although the material temperature continues to increase rapidly (Fig. 3(c)), ϵ' and ϵ'' remain relatively constant, as shown in Fig. 3(a). However, at approximately 950°C, where DSC data indicates the glass phase is mostly or entirely crystalline¹³, both ϵ' and ϵ'' increase again, with a further rapid increase in ϵ'' indicating the onset of melting. Overall, ϵ' increases by a factor of 3, while ϵ'' increases by a factor of 300.

Figure 3(d) shows that above the critical temperature at which the rapid increase in absorbed power occurs (750–800°C), microwave power absorption by the simulant subsequently decreases before reaching a plateau, while the sample temperature continues to increase very rapidly. This is followed by a steady increase in absorbed power, accompanied by a relatively steady rise in temperature, before a second rapid increase in absorbed power approaching T_m . As with JSC-1A, stable microwave heating becomes increasingly difficult to maintain due to rapidly changing cavity resonance and coupling conditions associated with changing dielectric properties.

At the melting temperature, permittivity peaked, then decreased slightly, and then peaked again as the simulant temperature increased beyond 1215°C. This may indicate the sequential melting of different mineral phases within the simulant.

3.4 Permittivity on cooling

On cooling from melting temperature to ambient temperature, the samples were solid with a sintered outer shell and some loose, unsintered powder remaining on the surface. A central void was observed, with smaller vesicles distributed throughout the surrounding solidified material. Glassy areas were also visible at the edge of the void. This is consistent with previous microwave heating studies¹. JSC-1A samples were dark grey, whereas NU-LHT-4M samples were lighter grey. No visual evidence of red colouration associated with oxidation to Fe₂O₃ was observed in either sample type.

Figure 4(a)	Figure 5(a)
Figure 4(b)	Figure 5(b)

Figure. 4. Temperature-dependent properties of JSC-1A during cooling from melting temperature to room temperature. Results are shown for three independent samples (Tests 1–3). T_m and T_g are from¹³. (a) permittivity on heating (lighter line) and cooling (darker line), with datasets vertically offset for clarity; (b) ϵ' (solid line) and ϵ'' (dashed line) on cooling. Tests 1 and 3 are cooled under microwave power. Test 2 shows unpowered cooling.

Figure. 5. Temperature-dependent properties of NU-LHT-4M during cooling from melting temperature to room temperature. Results are shown for three independent samples (Tests 1–3). T_m and T_g are from¹³. (a) permittivity on heating (lighter line) and cooling (darker line), with datasets vertically offset for clarity; (b) ϵ' (solid line) and ϵ'' (dashed line) on cooling. Tests 1 and 3 are cooled under microwave power. Test 2 shows unpowered cooling;

At ambient temperature, the permittivity of the cooled solid samples was generally higher than that of the powdered samples prior to heating (Fig. 4(a), Fig. 5(a)). The increase in permittivity of the NU-LHT-4M samples was greater than that observed for JSC-1A.

Additionally, samples in which permittivity reached higher values during heating generally exhibited correspondingly higher values on cooling, as shown in Fig. 4(b) and Fig. 5(b).

In most tests, the samples were cooled under microwave power at a controlled rate of $-0.25^\circ\text{C s}^{-1}$. Figure 4(b) and Fig. 5(b) also present data for a single sample of each simulant that was cooled without microwave power, yielding a significantly faster cooling rate. For both JSC-1A and NU-LHT-4M, between the melting temperature and approximately 900°C , both ϵ' and ϵ'' are slightly lower during unpowered cooling than during powered cooling, although this observation is based on a single experiment for each material.

3.5 Consistency

For both simulants, permittivity measurements are highly consistent across successive experimental repetitions at lower temperatures. However, above T_g , increased variability is observed. This scatter is attributed to microvariations in the local composition of individual powder specimens, such as grain composition and morphology, despite thorough premixing. At lower temperatures, where dielectric properties increased gradually with temperature, these local differences have little effect on bulk permittivity. However, above T_g , the larger differences in dielectric properties between constituent phases are likely to promote local variations in electric-field concentration during microwave heating, thereby contributing to differences in the high-temperature dielectric response between experimental runs.

4. Discussion

4.1 Temperature-dependent permittivity via microwave heating

In low-absorption materials, dielectric loss at low temperatures arises predominantly from bound-charge displacement associated with dipolar and atomic polarisation¹⁹. At lower temperatures, as shown in Fig. 2(a) and Fig. 3(a), for both simulants, ϵ' and ϵ'' are weakly temperature-dependent, increasing gradually with temperature due to enhanced dipole mobility and increased lattice polarisability. In ilmenite, Fe-O and Ti-O bond distances increase linearly with temperature²⁰, weakening restoring forces and promoting greater ionic displacement under the applied field. In addition, in heterogeneous materials, temperature-dependent contrasts in permittivity and conductivity between phases increase interfacial (Maxwell-Wagner) polarisation^{21,22}.

Figure 2(a) and Fig. 3(a) show that as the temperature approaches T_g , both ϵ' and ϵ'' increase more noticeably. This behaviour is consistent with enhanced microwave absorption above approximately $0.4\text{--}0.5 T_m$, associated with increased charge-carrier mobility at elevated temperatures¹⁹. Thermally activated lattice defects lead to the formation of vacancies, which enable ionic conduction under an applied microwave field²³. In mixed ionic-covalent minerals such as regolith simulants (Tables 1 and 2), charge transport is expected to be dominated by oxygen-vacancy migration, with contributions from cation vacancies. In Fe-bearing phases such as ilmenite, electronic conduction via small-polaron hopping between Fe^{2+} and Fe^{3+} increases with temperature, thereby contributing to the observed increase in conductivity and dielectric loss²⁴.

This transition to conduction-dominated dielectric behaviour is supported by the Arrhenius-type plots of ϵ'' , shown in Fig. 2(b) and Fig. 3(b), which display linear regions from approximately $0.55 T_m$ up to T_g for both simulants. This suggests that dielectric loss

follows an Arrhenius-type temperature dependence over this range. This behaviour is consistent with thermally activated charge-transport mechanisms governed by defect formation and vacancy-mediated ionic motion²³.

4.2 Specific characteristics of microwave heating

Because microwave power absorption is governed by ϵ'' , phases with higher dielectric loss preferentially absorb microwave energy, leading to localised heating. In regolith simulants, the strong temperature dependence of ϵ'' at elevated temperatures (Fig. 2(a) and Fig. 3(a)) amplifies this effect, so small increases in temperature may result in large increases in microwave absorption¹⁹. This positive feedback promotes non-uniform heating and hotspot formation, with hotspots previously observed to move within the material and split during microwave heating²⁵.

In powdered materials, electric field intensification at particle contact points may further enhance localised microwave absorption²². Combined with the low thermal conductivity of regolith simulant powder, this also promotes the development of local temperature gradients.

Figure 3(c) shows that for NU-LHT-4M, below a critical temperature near T_g , the heating rate remains stable and readily controllable. However, once this threshold is exceeded, the heating rate increases rapidly from approximately $0.25^\circ\text{C s}^{-1}$ to 5°C s^{-1} , indicating the onset of material reactions that increase the material's capacity to absorb microwave energy. Under these conditions, microwave heat generation exceeds the rate of heat dissipation through the material, resulting in a self-accelerating temperature rise. This was not observed in JSC-1A, Fig. 2(c).

The measured temperatures and permittivities represent bulk, volume-averaged properties of the sample. However, during microwave heating, localised hotspots with substantially higher permittivity develop within surrounding regions of lower permittivity, whereas in furnace heating the material remains closer to thermal equilibrium. Spatially non-uniform energy dissipation and local temperature gradients²⁶ together with the application of microwave fields that induce additional microstructural motions in the material²⁷ have been shown to produce microstructural states that differ from those of furnace-heated materials at the same bulk temperature.

4.3 The effect of glass phases on permittivity

The softening of glass phases above T_g enables viscous flow and promotes liquid-phase sintering. Wetting of adjacent mineral grains by the softened glass forms liquid bridges at particle contacts, reducing porosity and increasing densification. The presence of a liquid phase enhances mass transport and densification, enabling sintering at lower temperatures than conventional solid-state sintering²⁸. During microwave heating, localised hotspot formation may promote glass softening and liquid-phase sintering at lower apparent bulk temperatures, thereby reducing porosity and increasing density. Higher densities of lunar regolith simulants have been shown to result in increased ϵ' and ϵ'' at room temperature^{15,16}.

Below T_g the glass network remains relatively rigid, limiting ionic mobility, but above T_g , ionic mobility increases²⁹. A previous study of regolith simulants suggested that the transition to a viscous liquid phase facilitates polarisation processes⁶, thereby contributing to the observed increases in ϵ' and ϵ'' .

JSC-1A is derived from basaltic volcanic tuff and contains volcanic glass rather than agglutinitic glass²⁵, whereas the glass fraction in NU-LHT-4M is entirely agglutinitic. Table 1 shows that JSC-1A contains 26.7% glass, whereas NU-LHT-4M contains 35% agglutinitic glass. The highland simulant NU-LHT-2M was developed from the same feedstocks and processed in the same way as NU-LHT-4M but contains 30 wt% agglutinate glass and 5 wt% 'good' (high quality, visibly uniform) glass. Previous furnace heating measurements showed that the ϵ' and ϵ'' of NU-LHT-2M were higher than those of NU-LHT-4M, particularly above T_g ⁶. This suggests that differences between otherwise similar simulants indicate that glass composition and structure influence the temperature-dependent permittivity. Glass composition and structure are also known to influence the melt temperatures of simulants¹³.

As the temperature approaches T_g , the two simulants JSC-1A and NU-LHT-4M exhibit markedly different dielectric behaviour, as shown in Fig. 2(a) and Fig. 3(a). JSC-1A shows a relatively steady increase in ϵ' and ϵ'' up to the onset of melting, whereas NU-LHT-4M shows a very rapid increase in ϵ' and ϵ'' close to T_g , followed by a plateau before increasing again as T_m is approached. It is

unclear whether, and to what extent, differences in glass content and structure influence the permittivity response near T_g , or whether these effects are more pronounced under microwave heating. This will be investigated further.

4.4 Volatile release

Volatile gases released during the heating of Apollo soils from both mare and highland regions originate from several sources, including chemical reaction products and gases trapped within vesicles and inclusions in the regolith³⁰. In regolith simulants, where non-lunar mineral phases may constitute up to approximately 10–20% of geological feedstocks used for production, additional volatile release arises from the decomposition of these non-lunar phases³¹. Minor and trace minerals may contribute disproportionately to gas evolution³¹. Thermal processing of JSC-1A and NU-LHT-4M under vacuum demonstrated significant decomposition of non-lunar phases, particularly in NU-LHT-4M³¹. H_2O and hydroxyl-derived species were the dominant volatile products, originating primarily from the dehydration of minerals and clays, with smaller contributions from physisorbed surface water at lower temperatures. CO_2/CO was also released mainly from carbonate decomposition, and SO_2/SO_3 from the breakdown of sulphates and sulphides.

Carbonate phases present in the simulants include calcite ($CaCO_3$), with dolomite ($CaMg(CO_3)_2$) and siderite ($FeCO_3$) also reported in NU-LHT-4M³¹. Thermal decomposition of these carbonates between approximately 550–850°C produces the corresponding metal oxides. In oxide materials, chemical reactions can modify the concentration of lattice vacancies, thereby influencing ionic conductivity³³. The resulting increase in conduction losses contributes to a higher ϵ'' , and greater dissipation of microwave energy as heat.

The total mass loss measured in the present study (approximately 0.3 wt% for JSC-1A and 0.9 wt% for NU-LHT-4M) is comparable to previous vacuum-furnace measurements³¹. However, the higher heating rates and stronger thermal gradients associated with microwave heating may alter the kinetics of volatile evolution, potentially leading to more rapid volatile release. Previous studies have shown that increasing microwave input power produced higher temperatures and more abundant, sudden gas release within regolith simulants compared with samples heated at lower powers¹.

Further work is planned to measure volatile release by mass spectrometry, in parallel with permittivity measurements during microwave heating, to investigate any correlation between volatile evolution and changes in dielectric properties. A previous furnace heating study of JSC-1A suggested that the NASA bakeout protocol used to remove non-lunar phases may reduce both ϵ' and ϵ'' , depending on bakeout temperature and processing atmosphere, although ϵ'' was similar above approximately 800°C⁶.

4.5 Differences in permittivity between heating and cooling

The differences observed between the initial powdered samples and the cooled solid material, Fig. 4(a) and Fig. 5(a), suggest the dielectric response is influenced not only by temperature, but also by irreversible changes in material state during heating. At high temperatures, changes in the material's structure and composition, together with densification through sintering and/or partial melting, result in a permanent modification of the dielectric response on cooling.

The observation that samples which reached higher values of permittivity during heating generally exhibited correspondingly higher values on cooling, Fig. 4(a) and Fig. 5(a), is consistent with findings from another study⁶ and is thought to result from the incomplete melting of samples with lower permittivity than those with higher permittivity, which were more fully melted. The phase changes that occur during melting appear to contribute to the higher permittivity observed in the samples upon cooling.

The limited results show slightly lower ϵ' and ϵ'' during unpowered cooling than during powered cooling. If the difference exists, it may be attributed either to variations in material structure resulting from faster cooling, or to the presence of the microwave field during powered cooling. Previous studies by the research team demonstrated that the application of microwave energy could sustain charge-carrier mobility or polarisation processes that are absent under unpowered conditions²⁷. Further experimental work is required to confirm this behaviour and to distinguish field effects from differences in the material microstructure arising from cooling rate.

5. Conclusions and implications for microwave-based ISRU process modelling

This study reports measurements of the temperature-dependent permittivity of lunar regolith simulants that differ from previous studies in two key respects: abrupt increases in permittivity were measured in real time, and the measurements were performed during direct microwave heating, rather than under resistance-furnace heating⁶.

The results suggest that the ϵ' and ϵ'' of regolith simulants depend not only on bulk temperature, but also on the heating process and the resulting thermal state of the material. Although similar thermally activated mechanisms may increase permittivity during both microwave and furnace heating, the resulting thermal and microstructural evolution may differ. During furnace heating, temperature is externally imposed, resulting in a comparatively uniform thermal distribution. In contrast, because microwave power absorption is proportional to ϵ'' , local increases in dielectric loss directly increase localised heating and temperature. Differences in permittivity between simulant phases may further promote different thermal distributions during microwave heating.

In the present study, permittivity measurements during microwave heating were similar to those obtained via furnace heating⁶ up to approximately $0.5 T_m$ and again approaching T_m . However, the higher permittivity observed during microwave heating at intermediate temperatures, particularly for NU-LHT-4M, may reflect an increasing contribution from conduction-related losses. In particular, as the temperature increases, microwave heating may induce molecular- and microstructural-level transformations that differ from those produced by conventional heating. Consequently, dielectric properties measured under intense microwave fields may deviate from those obtained using other heating methods. Localised microstructural and chemical changes associated with volatile release and defect generation may further increase ϵ'' . In addition, microwave-induced localised heating may promote liquid-phase sintering and densification at lower bulk temperatures than during furnace heating, thereby contributing to the observed increase in permittivity.

Such behaviour may not be unexpected given that differences between microwave and furnace processing have previously been observed. For example, microwave sintering of low-loss ceramics has been reported to occur at lower apparent temperatures than during furnace heating³². Similarly, rapid microwave heating has been shown to produce gravel-shaped, solidified lunar regolith at temperatures approximately 300°C lower than those required during furnace heating³³.

The results of this study show differences in dielectric properties between cooling and heating. During heating, the powdered simulant densifies, sinters, and partially melts its crystalline phases; upon cooling, the material solidifies. These physical changes, together with probable chemical changes, result in a corresponding shift in permittivity⁷.

The increasing microwave response of regolith with temperature has important implications for thermal management and high-temperature processing. The increase in ϵ'' observed at high temperatures indicates regolith simulants become more effective microwave absorbers as they approach the temperatures required for sintering and melting. Once high-temperature processing conditions are established, increased microwave energy absorption may assist in maintaining elevated temperatures, supporting continuous processes such as densification, melting and regolith-based 3D printing. For layer-by-layer construction processes, maintaining controlled thermal conditions during deposition is an important consideration.

Understanding the temperature dependence of ϵ' and ϵ'' is important for designing efficient microwave heating processes for ISRU. Microwave heating models require dielectric properties as fundamental input parameters; however, the pronounced temperature-dependent changes observed in this study demonstrate the electromagnetic response of regolith simulants changes significantly during heating. Models based on room-temperature properties alone may not accurately predict energy deposition, temperature evolution, the formation and movement of localised hotspots, and the onset of thermal runaway across the temperature range required for lunar processing. Quantifying differences in permittivity between mare and highland simulants, which reflect their distinct chemical compositions, is also important for process optimisation and the selection of appropriate processing conditions for different lunar feedstocks.

The measurements in this study provide essential input data to improve the accuracy of coupled numerical models of microwave heating behaviour in lunar regolith⁴. Incorporating temperature-dependent dielectric properties will enable simulations to capture the dynamic feedback between temperature, permittivity and microwave energy absorption. Maintaining energy-efficient microwave processing may require strategies such as dynamic frequency tuning, impedance matching or power modulation to account for changes in microwave coupling and energy absorption as the material temperature increases. This is particularly important as the material approaches sintering and melting temperatures, where rapid changes in dielectric behaviour may strongly influence heating

stability. Improved accuracy in numerical models will support optimisation of microwave cavity geometry, power delivery strategies and process control as microwave heating is scaled from laboratory experiments towards larger ISRU systems.

Although the evolution of mechanisms underlying the observed increase in permittivity with temperature is not yet fully understood, the measurements presented here provide an important foundation for microwave process modelling and future ISRU technology development.

Declarations

Author contributions

S.L., C.O'C. and A.L.F. conceptualised the study. J.M.C.-C. and B.G.-B. designed the experiments. C.O'C. conducted the experiments, analysed the results and wrote the first draft of the manuscript. J.D.G.-C. analysed measurements. All authors contributed to interpreting the data and reviewing the manuscript.

Data availability statement

The datasets generated during the current study are available from the corresponding author on reasonable request.

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Competing interests

The authors declare no competing interests.

Additional information

Correspondence should be addressed to C.O'C.

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Figures

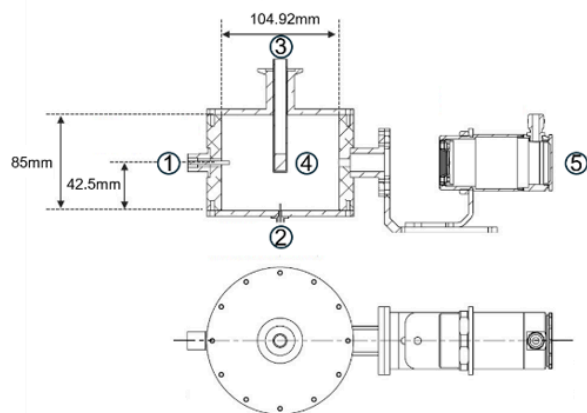


Fig. 1(a)

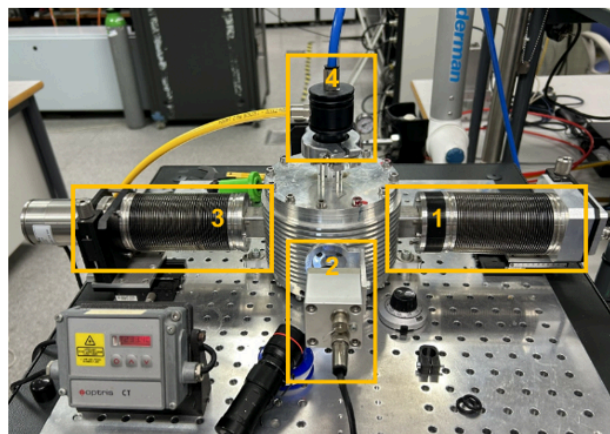


Fig. 1(b)

Figure 1

Dual mode cylindrical cavity for simultaneous microwave heating and measurement of complex permittivity¹⁰. (a) Schematic view. Labelled parts are – 1: microwave heating port, 2: microwave measurement port, 3: quartz tube, 4: regolith simulant, 5: infrared thermometer. (b) Experimental system located at the ITACA Institute, Universitat Politècnica de València. Labelled parts are – 1: microwave heating port, 2: optical camera, 3: infrared thermometer, 4: N₂ gas inlet to quartz tube.

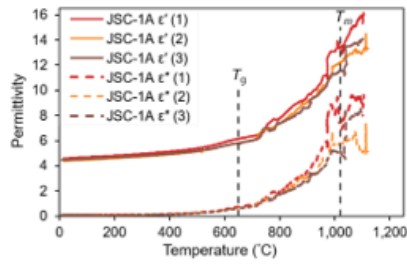


Fig. 2(a)

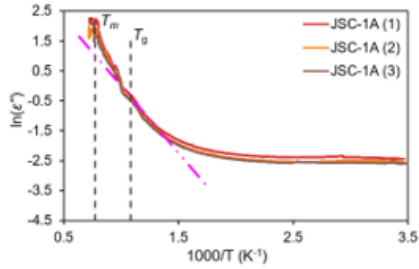


Fig. 2(b)

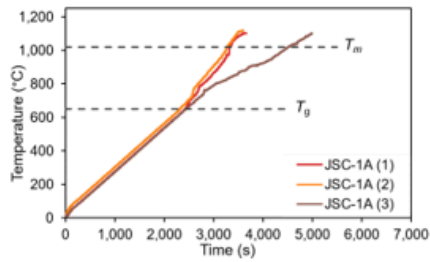


Fig. 2(c)

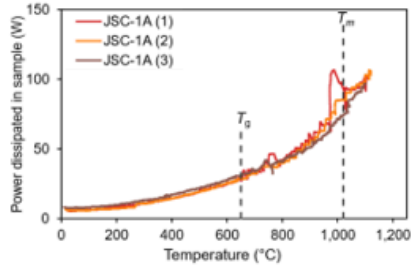


Fig. 2(d)

Figure 2

Temperature-dependent properties of JSC-1A during heating from room temperature to melting temperature. Results are shown for three independent samples (Tests 1-3). T_m and T_g are from¹³. (a) ϵ' (solid line) and ϵ'' (dashed line) on heating; (b) Arrhenius plots of $\ln(\epsilon'')$ as a function of ($1000/\text{Temperature}$) for three samples, with a linear fit to the selected region (pink dashed line); (c) temperature increase with time; (d) microwave power absorbed by the samples on heating.

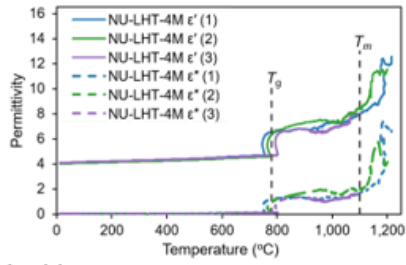


Fig. 3(a)

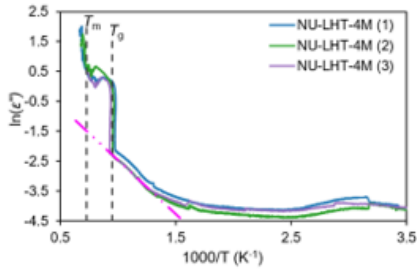


Fig. 3(b)

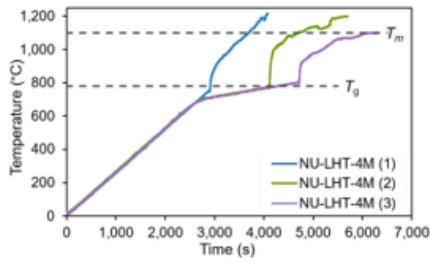


Fig. 3(c)

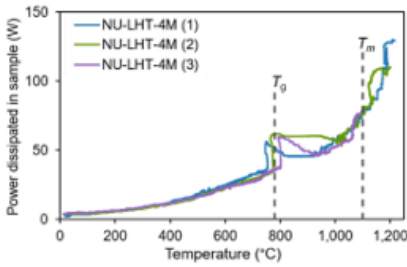


Fig. 3(d)

Figure 3

Temperature-dependent properties of NU-LHT-4M during heating from room temperature to melting temperature. Results are shown for three independent samples (Tests 1-3). T_m and T_g are from¹³. (a) ϵ' (solid line) and ϵ'' (dashed line) on heating; (b) Arrhenius plots of $\ln(\epsilon'')$ as a function of $(1000/\text{Temperature})$ for three samples, with a linear fit to the selected region (pink dashed line); (c) temperature increase with time; (d) microwave power absorbed by the samples on heating.

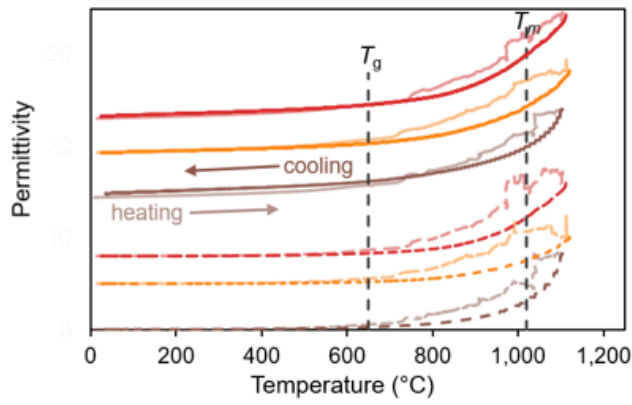


Fig.4(a)

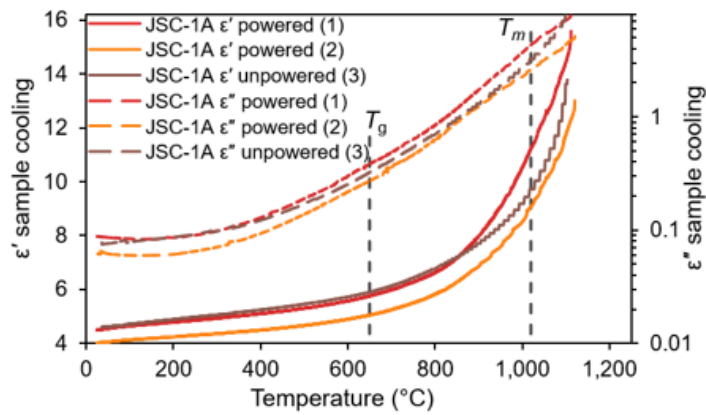


Fig. 4(b)

Figure 4

Temperature-dependent properties of JSC-1A during cooling from melting temperature to room temperature. Results are shown for three independent samples (Tests 1-3). T_m and T_g are from¹³. (a) permittivity on heating (lighter line) and cooling (darker line), with datasets vertically offset for clarity; (b) ϵ' (solid line) and ϵ'' (dashed line) on cooling. Tests 1 and 3 are cooled under microwave power. Test 2 shows unpowered cooling.

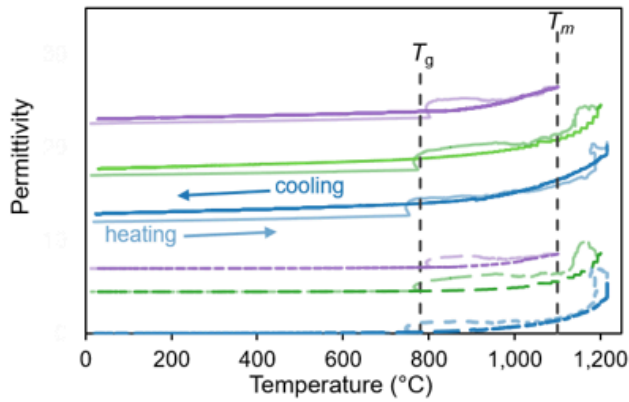


Fig. 5(a)

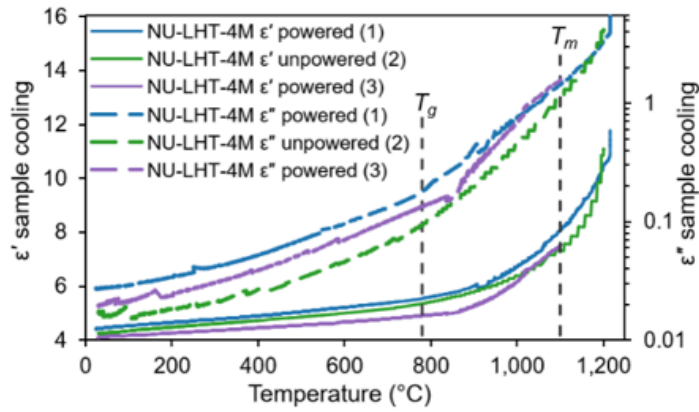


Fig. 5(b)

Figure 5

Temperature-dependent properties of NU-LHT-4M during cooling from melting temperature to room temperature. Results are shown for three independent samples (Tests 1-3). T_m and T_g are from¹³. (a) permittivity on heating (lighter line) and cooling (darker line), with datasets vertically offset for clarity; (b) ϵ' (solid line) and ϵ'' (dashed line) on cooling. Tests 1 and 3 are cooled under microwave power. Test 2 shows unpowered cooling;