



Research Paper

Microwave heating behavior and thermal homogeneity of kaolinite-, illite-, and smectite-rich clay systems with additives

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ARTICLE INFO

Keywords:

Microwave heating

Clay minerals

Dielectric properties

Dehydroxylation

Heating behavior

Temperature homogeneity

Brick clays

ABSTRACT

Microwave heating of clay-based materials has the potential to make ceramic processing more energy efficient. However, the influence of mineralogical composition and bulk density of the clays on heating behavior is not yet fully understood. This study investigates the microwave response at 2.45 GHz of kaolinite-, illite-, and smectite-rich clays with and without additives, as well as brick clays. The heating behavior was evaluated in relation to mineralogical, thermal (experimental), and dielectric (literature) analyses. Smectite-rich systems exhibited the strongest initial microwave coupling, followed by illite-rich clays, while kaolinite-rich systems showed comparatively weak absorption. During dehydroxylation, the net heating rate decreased in all systems due to the endothermic nature of the reaction, which was most pronounced in kaolinite. Kaolinite-rich clays exhibited significant dielectric changes during phase transitions, shifting from weakly to strongly absorbing regions. This promoted local overheating and temperature inhomogeneities. In contrast, illite- and smectite-rich clays maintained a more stable absorption over several reaction intervals, resulting in more uniform energy distribution and homogeneous temperature profiles. Additional phases altered the overall microwave interaction: iron oxides and carbonaceous components enhanced absorption, while most carbonates had only a minor influence. Increased bulk density, especially in quartz-containing systems, contributed to more uniform temperature fields. Overall, the microwave heating response of the investigated clay systems was governed by the interplay between mineralogical composition, additive content, and sample density. This provides a framework for improving the evaluation of thermal treatment strategies for ceramic raw materials using microwave heating.

1. Introduction and background

In the context of decarbonizing high-temperature ceramic processes, microwave-based methods are once again being discussed as they could potentially reduce energy consumption and process times through volumetric heating (Chandrasekaran et al., 2012; Oghbaei and Mirzaee, 2010). Corresponding approaches have been investigated since the 1980s, but their industrial implementation has been limited by economic factors, limited process control and, in some cases, the low microwave absorption of ceramic raw materials (National Research Council, 1994). However, with the emission targets of the European Climate Law, electrically powered sintering technologies are once again coming into focus (European Parliament, 2021).

Microwaves enable the direct volumetric coupling of electromagnetic energy into materials. Heating is based on the interaction of the electromagnetic field with dipoles and charge carriers. Part of the en-

ergy is stored (the real part of the permittivity, ϵ'), while another part is dissipated (the imaginary part of the permittivity, ϵ''). The loss tangent $\tan \delta$ (Eq. (1)) describes the ratio of these components and is a key parameter for evaluating microwave coupling (Solymar et al., 2018):

$$\tan \delta = \epsilon'' / \epsilon' \quad (1)$$

Materials with $\tan \delta > 0.01$ exhibit medium coupling to the microwave field, while values above 0.1 indicate excellent absorption (Vermeiren et al., 2024). Since raw clays, such as brick clays, consist mostly of multi-phase systems, predicting their microwave heating behavior is challenging. Brick clays are complex raw clay systems consisting of quartz-rich fractions, clay minerals such as illite, smectite, and kaolinite, and variable amounts of carbonates, iron-bearing minerals, and organic matter (Jasmund and Lagaly, 1993).

Fig. 1 summarizes literature data on the temperature-dependent loss behavior of selected materials, which can be seen in Table 1 and in more

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detail in the supplementary data (Table S1). After drying, microwave absorption initially decreases due to the loss of physically bound water (Vorhauer-Huguet et al., 2022). Kaolinite-rich clays behave as weak absorbers ($\tan \delta < 0.01$) within the subsequent temperature range, whereas illite- and smectite-rich clays continue to exhibit medium interaction with the electromagnetic field ($0.01 < \tan \delta < 0.1$). As the temperature increases, the loss tangent rises for all clays. This is related to processes such as dehydroxylation (DTI, 2017), defect formation and thermally activated charge carriers (Reinosa et al., 2019).

Other mineral phases exhibit different microwave couplings: tectosilicates (DTI, 2017) and many carbonates (DTI, 2017; Vermeiren et al., 2024) mainly have low $\tan \delta$ unless they are in hydrated form, in which case they are lossy until dehydration (Lovás et al., 2010). Iron-containing phases, however, can achieve significantly higher absorption during thermal transformations or as a result of these transformations (Lovás et al., 2010; Pickles et al., 2005). These differences demonstrate that mineralogical composition is crucial for heating behavior in a microwave field.

The heating behavior of ceramic test specimens in a microwave field is also affected by their low thermal conductivity. If more microwave energy is converted into heat locally than can be compensated for by heat conduction, temperature gradients and hot spots can occur (Wu, 2002). For dry kaolinitic clays, thermal conductivity increases with sample density, rising from approximately 0.3 W/(mK) (1450 kg/m³) to 0.55 W/(mK) (1900 kg/m³) (Gualtieri et al., 2010) and rising moderately with temperature (Michot et al., 2008).

Consequently, the question arises how strongly mineralogical differences influence the actual heating behavior of clay-based systems in the microwave field. While numerous studies have investigated the dielectric properties of individual mineral phases, comparative studies under actual heating conditions are lacking. This study systematically addressed this gap by investigating the influence of clay mineral composition—from individual minerals to complex clay systems—through heating samples of different compositions under identical microwave conditions up to a core temperature of 1050 °C.

Table 1

Summary of Material information: corresponding to Fig. 1, including frequency conditions and literature references. Abbreviation: n.r. = not reported in patent-microwave regime, typically around 2.45 GHz. More information is provided in supplementary data (Table S1).

Material group	Material no.	Frequency (GHz)	Reference
Clays			
kaolinitic clays	#1 #2	2.45	(Vermeiren et al., 2024)
	#3	2.47	(DTI, 2017)
	#4	n.r.	(López Buendía et al., 2020)
	#5	2.45	(Microwave Properties North, 2026)
illitic-kaolinitic clay	#6	n.r.	(López Buendía et al., 2020)
illitic clay	#7	2.47	(DTI, 2017)
smectitic clays	#8	2.47	(DTI, 2017)
	#9	n.r.	(López Buendía et al., 2020)
Other minerals			
albite, orthoclase, quartz, calcite		2.47	(DTI, 2017)
siderite, magnesite		2.216	(Lovás et al., 2010)
limestone		2.45	(Vermeiren et al., 2024)
goethite	#1 / #2	0.912 / 2.46	(Pickles et al., 2005)
hematite		0.912	(Pickles et al., 2005)

2. Materials and methods

2.1. Literature data collection

Literature data were digitized from published dielectric diagrams using WebPlotDigitizer (online). Extracted ϵ' and ϵ'' values were subsequently converted to $\tan \delta$ using Eq. (1).

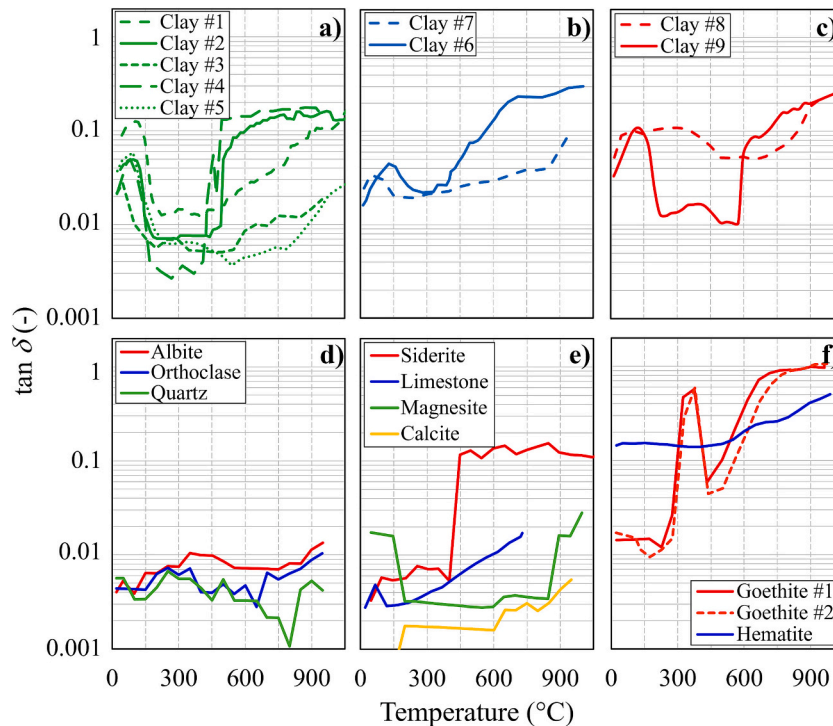


Fig. 1. Temperature-dependent $\tan \delta$ of various mineral groups: (a–c) clay minerals, (d) tectosilicates, (e) carbonates and (f) iron oxides; data were derived from literature sources; corresponding compositions, measurement methods and references are summarized in Table 1.

2.2. Selection of materials

This work used eight different clays and eight different additive phases.

Five clays can predominantly be attributed to a single clay mineral, while three represent natural mixed clays: KC (kaolinite-rich), IC A and IC B (illite-rich), SC A and SC B (smectite-rich) and BC A to BC C (brick clays). The brick clays are predominantly kaolinitic (BC A, BC C) or illitic (BC B). BC A has an increased quartz content and thus a lower clay mineral content than BC C. The clays were provided by Schlagmann Poroton GmbH & Co. KG. They originate from different production lines or suppliers, which are not specified here.

The additional phases include iron oxides and (hydr)oxides (hematite and goethite from LANXESS Deutschland GmbH), carbonates (calcite and hydromagnesite from Diacleanshop and siderite from TROPAG, Oscar H. Ritter Nachf. GmbH), a tectosilicate (quartz from Gebrüder Dorfner GmbH & Co. KG) and carbon-based materials (charcoal from a local supplier and hard coal from Kremer Pigmente GmbH & Co. KG). These carbon-based materials represent organic matter and charred residues present in clay-based raw materials.

Various mixtures of KC, IC A and SC A (Table 2) were produced to investigate the influence of a targeted shift in the clay mineral content. In addition, combinations with quartz and defined impurities resulting from the additional phases were considered.

2.3. Mineralogical and chemical characterization

The quantitative mineral composition (Table 3 upper part) was determined using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) analyses. For XRD analysis, powder preparations and glycerol-saturated powder preparations were used to improve the identification of expandable clay mineral phases.

XRD patterns were recorded using a Bruker D2 Phaser X-ray diffractometer equipped with a Cu tube ($\lambda = 1.54184 \text{ \AA}$) operated at 30 kV and 10 mA. Measurements were carried out in the range of $3\text{--}63^\circ 2\theta$ using a coupled Two Theta/Theta scan geometry in continuous scan mode. The divergence slit and receiving slit were both set to 0.6 mm. Data acquisition was performed with a step time of 1 s over 4001 steps at a goniometer speed of $1^\circ/\text{min}$ using a scintillation detector. FTIR measurements were performed on a Thermo Nicolet NEXUS 470 in the range of $4000\text{--}400 \text{ 1/cm}$ using potassium bromide (KBr) pellets prepared according to the KBr pressing method (2 mg sample mixed with 198 mg KBr).

Combined XRD and FTIR evaluation of the total sample allows quantitative estimation of the mineral phases with an error of $\leq 10 \text{ wt\%}$, assuming a variation range of approximately 1 wt% for content $\leq 10 \text{ wt\%}$. The calculated mineral composition of the mixtures in Table 2 can be found in the supplementary data (Table S2).

The chemical composition (Table 3 lower part) was determined using quantitative X-ray fluorescence analysis (WDS-XRF) in accordance with DIN EN ISO 12677. Fused tablets for the analysis of 30 oxides were produced from finely ground sample material ($< 63 \mu\text{m}$). Measurements were performed on a PANalytical AXIOS PW 2400 (2.4 kW, Rh anode). Loss on ignition (LOI) was determined at 1050°C according to DIN 51081. Minor deviations from 100 wt% result from analytical uncertainty and rounding.

Table 2
Material composition (wt%) of the mixed clay samples.

Component	K ₆₆ I ₃₃	K ₃₃ I ₆₆	K ₅₇ I ₂₈ S ₁₅	K ₂₈ I ₅₇ S ₁₅	(K ₂₈ I ₅₇ S ₁₅) ₈₀ -Q ₂₀	(K ₂₈ I ₅₇ S ₁₅) ₈₀ -Q ₁₆ -R ₄
KC	67	33	57	28	22	22
IC A	33	67	28	57	46	46
SC A	–	–	15	15	12	12
quartz	–	–	–	–	20	16
siderite, hematite goethite, hard coal	–	–	–	–	–	each 1

2.4. Sample preparation

The clays and clay mixtures were conditioned with water in a intensive mixer (EIRICH RV02E) at 1000 rpm for ten minutes. The water content was adjusted to between 13 wt% and 20 wt%, depending on the material, to ensure sufficient plasticity for shaping. The mixtures were then uniaxially compacted at a pressure of 10 N/mm^2 using a Form+Test DPM ALPHA 3–3000 S and dried at 105°C until mass consistency was achieved.

Test specimens were cut into 400 g cubes with a central 18 mm diameter drill hole extending to the center of the specimen to enable temperature measurement in the specimen core. The density of the dried specimens was determined from their mass and dimensions.

2.5. Characterization of thermal behavior

Thermal analyses were performed using simultaneous thermal analysis (STA) on a Netzsch STA 449 F5, combining thermogravimetry (TG) and differential scanning calorimetry (DSC). The material to be examined was ground to an analysis fineness in a Retsch PM 200 planetary mill and placed in alumina crucibles. Measurements were taken at a heating rate of $10^\circ\text{C}/\text{min}$ up to 1100°C in synthetic air (20 vol% O₂, 80 vol% N₂) using an empty alumina crucible as a reference. Sample masses of $50.0 \pm 0.5 \text{ mg}$ were generally used; however, for materials with a low powder density (e.g. hydromagnesite), the crucible was filled completely.

Dilatometry (DIL) was performed using a Netzsch DIL 402 PC at a heating rate of $5^\circ\text{C}/\text{min}$. The cylindrical samples had a length of approximately 25 mm. The dilatometer had a measuring range of $\pm 2.5 \text{ mm}$. The measurements were also performed in synthetic air.

2.6. Experimental setup for microwave heating

2.6.1. Temperature measurement

Determining the core temperature under microwave irradiation at high temperatures is challenging from an experimental perspective. Metal-based sensors can significantly affect temperature measurements because they interact with the microwave field (Pert et al., 2001). Commercially available fiber optic measurement systems are of limited use in the high-temperature microwave conditions present here.

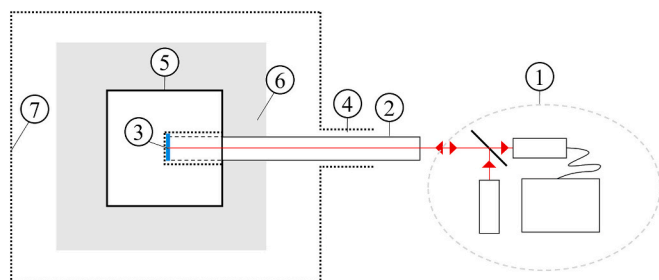
Against this background, an established high-temperature Fabry–Perot temperature measurement system (Pechstedt and Sposito, 2018; Zhu et al., 2004) was adapted at Technology Campus Teisnach (TCTS) for microwave applications (Fig. 2, components 1–3). Temperature measurement is based on an uncoated sapphire etalon positioned at the tip of an alumina tube with a maximum diameter of 15 mm. The optical path difference (OPD), or in other words the change in thickness of the sapphire etalon, is measured and the corresponding temperature is calculated (Pechstedt and Sposito, 2018; Zhu et al., 2004) using the temperature-dependent material properties of sapphire (Thomas et al., 1998; White and Mingos, 1997).

With a $\tan \delta$ of approximately 10^{-7} , sapphire is highly transparent, resulting in minimal interaction with electromagnetic radiation (Kusunoki et al., 2002). Further details on the measurement setup can be found in supplementary data (Chapter S1).

Table 3

Mineralogical and chemical composition (wt%) of clays.

component	KC	SC A	SC B	IC A	IC B	BC A	BC B	BC C
kaolinite ¹	82	5	-	12	13	34	24	53
illite ²	10	3	3	67	67	13	26	23
illite/smectite	-	-	-	-	-	5	10	2
smectite	-	79	78	-	-	5	-	-
chlorite	-	2	2	4	5	4	12	-
quartz	5	1	1	2	5	32	14	15
crystalobalite	-	2	13	-	-	-	-	-
feldspar ³	3	3	2	2	2	3	3	2
calcite	-	3	< 1	12	7	1	5	1
Fe-bearing phases ⁴	-	-	-	-	-	2	2	3
accessory phases ⁵	-	1	-	1	< 1	1	2	1
organic matter	-	< 1	< 1	< 1	< 1	1	1	1
SiO ₂	50.4	54.2	65.9	44.4	47.2	64.1	49.2	52.9
Al ₂ O ₃	33.8	21.2	16.1	19.3	20.8	20.5	20.6	24.92
Fe ₂ O ₃	0.5	3.0	1.2	6.5	7.0	3.5	7.4	7.1
TiO ₂	0.4	0.3	0.2	0.7	0.8	1.0	1.0	1.2
P ₂ O ₅	0.5	< 0.1	< 0.1	0.3	0.3	0.1	0.3	0.1
CaO	0.1	2.9	2.5	7.3	4.1	0.5	3.9	0.3
MgO	0.2	3.0	3.6	3.3	3.4	0.6	1.9	0.7
MnO	< 0.1	0.2	< 0.1	< 0.1	< 0.1	< 0.1	0.2	< 0.1
K ₂ O	2.1	0.7	1.8	6.1	6.2	1.9	3.8	2.0
Na ₂ O	< 0.1	5.0	0.9	0.1	0.1	0.2	0.1	0.1
SO ₃	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.7	< 0.1
LOI	11.4	9.4	7.8	11.8	9.9	7.4	11.3	10.8

¹ ordered/disordered kaolinite.² illite, mica and sericite.³ albite and K-feldspar.⁴ hematite, goethite and siderite.⁵ anatase, dolomite and gypsum.**Fig. 2.** Schematic illustration of the microwave heating setup: (1) sensor setup, (2) alumina tube, (3) sapphire etalon, (4) feed-through, (5) specimen, (6) alumina insulation, (7) domestic microwave oven.

2.6.2. Microwave setup

Heating tests were performed using a Panasonic NN-CS89LB domestic microwave oven. Its usable space measures 410 mm in length, 320 mm in depth and 240 mm in height. The device uses inverter technology to control power continuously. Fig. 2 (components 4–7) illustrates the joint test setup.

To allow the rigid sensor to be inserted into the specimen inside the household microwave oven, the cavity was modified slightly. A feed-through was integrated into one side wall of the cavity. To maintain microwave shielding, a metallic waveguide operating below its cutoff frequency (with a length of 50 mm and an inner diameter of 20 mm) was connected to the cavity wall. No measurable microwave leakage radiation was detected during operation.

The sample was positioned centrally within the usable space. An 18 mm tile drill was used to bore a cylindrical hole to the center of one side, ensuring that the sensor tip was in the center of the sample. To protect the household microwave oven from heat, the sample was surrounded by aluminosilicate fiber insulation (Kerform KVS 121 from Rath).

The clay samples were irradiated at a microwave power of 300 W until a core temperature of 1050 °C was reached. To evaluate thermal inhomogeneities during microwave heating, surface luminosity was

qualitatively assessed from photographs taken immediately after heating under identical conditions. The samples were classified according to glow intensity (non-luminous, orange luminous, or yellow/white luminous) and the spatial distribution of the luminous regions (partial or complete surface luminosity).

2.6.3. Data evaluation

For each composition, at least three experiments were conducted. However, error bars are only displayed in representative figures to maintain readability. The experiment with the median total duration was selected as representative heating curve. Standard deviations were calculated across all experiments at identical temperature levels (temperature-based evaluation) and evaluated in 50 °C increments for clarity. The heating rate was calculated as the temperature difference over a time interval of one minute ($\Delta T/\Delta t$) and assigned to the corresponding temperature. An example is provided in the supplementary data (Fig. S1).

3. Results

3.1. DSC, TG and DIL

To determine the relevant reaction periods for subsequent experiments, DSC, TG and DIL were conducted on clays KC, IC A, IC B, SC A, and SC B (Fig. 3), as well as on selected clay mixtures and brick clays (Fig. S2). Additionally, the additives were investigated using TG and DSC (Table 4). The assignment of thermal events of clays follows established reference ranges reported in literature (Jasmund and Lagaly, 1993; Salmang et al., 2022).

KC (Fig. 3 left) lost ~0.5 wt% of adsorbed water at temperatures up to ~150 °C. Between 450 °C and 700 °C, the kaolinite underwent dehydroxylation to form metakaolinite, resulting in a mass loss of ~11 wt% and shrinkage—overlapping with the dehydroxylation of illite. Between 980 °C and 1000 °C, an exothermic effect occurred, which is attributed to the formation of Al–Si spinel, accompanied by further compaction.

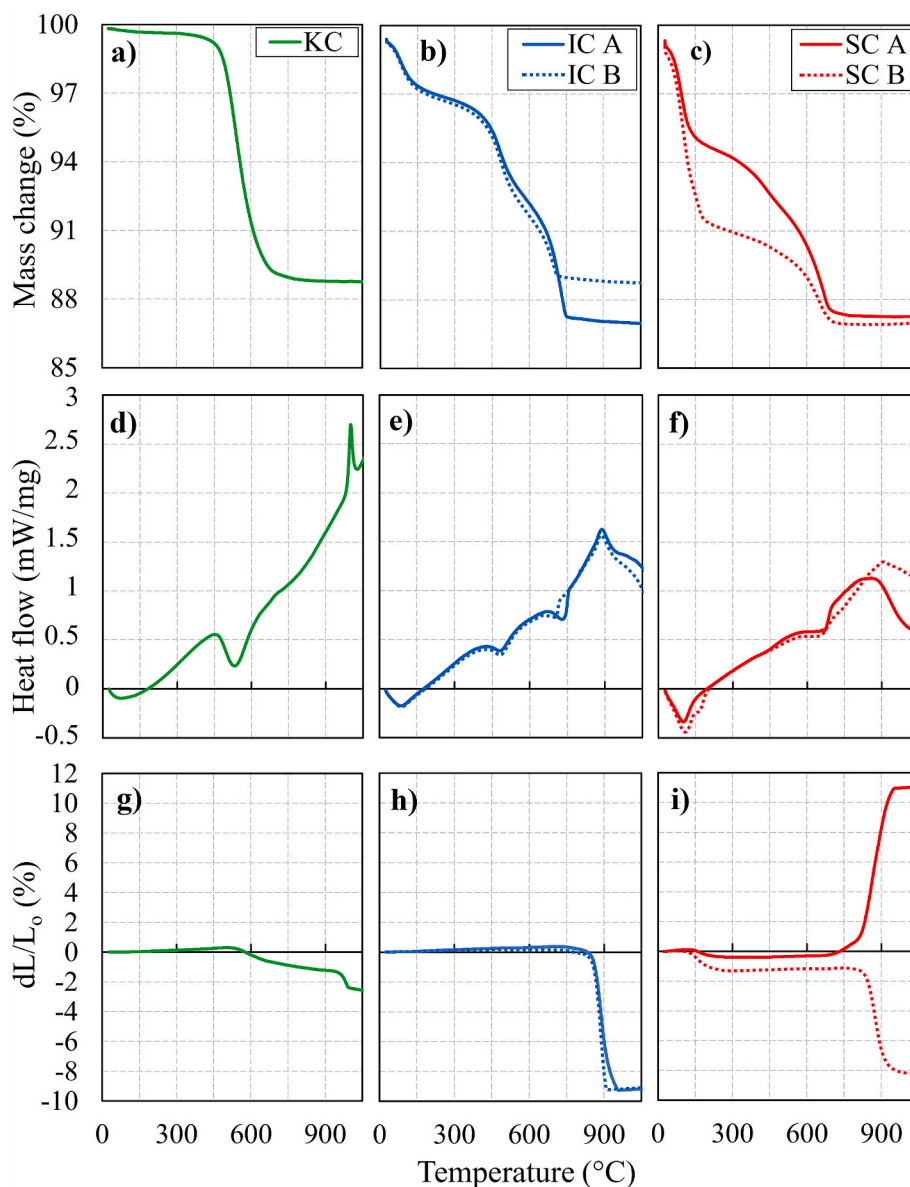


Fig. 3. Thermal analyses: KC (a,d,g), IC A&B (b,e,h) and SC A&B (c,f,i); rows correspond to TG (a–c), DSC (d–f) and DIL (g–i).

Table 4

Summary of thermal transformations derived from TG/DSC measurements of the additives.

Material	Transformation	Temperature (°C)	TG (%)
goethite	dehydroxylation (Pickles et al., 2005)	240–340	–11
hematite	no significant reaction	–	–
calcite	decomposition (Karunadasa et al., 2019)	640–850	–43
hydromagnesite	dehydration, (Hollingbery and Hull, 2010)	180–340 340–500	–15 –37
siderite	dehydroxylation/decomposition (Kądziołka-Gaweł et al., 2023)	420–730	–31
quartz	no significant reaction	–	–
charcoal	oxidation	350–550	–90
hard coal	oxidation	430–700	–80

IC A and IC B (Fig. 3 middle) lost ~2 wt% of adsorbed water up to ~150 °C. A further mass loss of ~5 wt% began between 350 °C and

400 °C in connection with the dehydroxylation of illite, which additionally overlapped with the dehydroxylation of disordered kaolinite. At ~640 °C, the calcite decomposed (Karunadasa et al., 2019) with a further mass loss of ~5 wt% (IC A) and ~3 wt% (IC B). Above ~850 °C, DSC showed an endothermic shift without significant mass change, accompanied by strong shrinkage up to the measuring limit of the device—sintering begins.

SC A and SC B (Fig. 3 right) exhibited significantly increased mass loss up to ~200 °C due to adsorbed water and interlayer water: SC A ~4 wt%, SC B ~8 wt%. This is accompanied by slight shrinkage. The mass loss continued up to ~700 °C. The process began with the dehydroxylation of a small amount of disordered kaolinite and/or illite from ~400 °C. This was followed for both by the dehydroxylation of the main mineral smectite, whose reaction ranges were between 550 °C and 700 °C. After SC clays were dehydrated the total mass loss was ~5 wt% (SC B) and ~8.5 wt% (SC A). At ~800 °C, both clays exhibited an endothermic shift in the DSC signal, indicating the onset of sintering. These shifts led either to an increase in volume up to the measurement limit (SC A) or shrinkage of up to ~8 wt% (SC B).

All additional mineral and carbon-containing phases were analyzed

and summarized in Table 4. BC B and BC C underwent organic burnout at temperatures between 200 °C and 400 °C, resulting in an exothermic effect.

3.2. General microwave heating behavior

To compare the heating behavior and heating rates of the samples in the following chapters, the observed heating inhomogeneity is first examined. Fig. 4 shows representative final images of selected samples, where surface luminosity serves as an indicator of temperature distribution in relation to kaolinite-to-illite ratios and density. The shape of the marking indicates smectite/chlorite content.

A tendency towards increased surface luminosity was observed with increasing illite content and sample density. SC A and SC B also exhibited distinctly luminous surfaces. This trend was particularly evident in the mixed series with an illite ratio of $I/(K + I) \approx 60$ and $I/(K + I) \approx 10$. Within the $I/(K + I) \approx 60$ series, luminosity increased gradually from the $K_{33}I_{66}$ and $K_{28}I_{57}S_{15}$ mixture with a density of $\sim 1.6 \text{ g/cm}^3$, via the quartz-modified variants $(K_{28}I_{57}S_{15})_{80}\text{-}Q_{20}$ and $(K_{28}I_{57}S_{15})_{80}\text{-}Q_{16}\text{-}R_4$ with a density of $\sim 1.85 \text{ g/cm}^3$, reaching a maximum in the corresponding brick clay BC B with a density of $\sim 2 \text{ g/cm}^3$. In the case of $I/(K + I) \approx 10$, sample density increased from $\sim 1.5 \text{ g/cm}^3$ to $\sim 1.7 \text{ g/cm}^3$ by adding 20 wt% quartz. Even when highly absorbent phases were added, such as goethite or carbonaceous materials, kaolinite-rich samples in the low-density range did not show any significant increase in surface luminosity. Additionally, illitic clays with increased alkali and iron mineral content exhibited significant expansion during heating (Fig. S3). This strong bloating behavior caused cracks. The same applied to SC A; SC B had already cracked due to shrinkage after drying.

3.3. Heating behavior of kaolinite-, illite- and smectite-rich clays

KC (Fig. 5a) exhibited an increased heating rate at the start of the test, up to $\sim 150^\circ\text{C}$. The heating rate then remained largely constant at around 5°C/min up to $\sim 400^\circ\text{C}$. Between $\sim 450^\circ\text{C}$ and 600°C , the heating rate dropped significantly, reaching a minimum of $\sim 1^\circ\text{C/min}$. After this minimum, the heating rate rose slightly up to $\sim 750^\circ\text{C}$ – 800°C , then increased sharply above this range, reaching values of up to 15°C/min at 1050°C .

IC A and IC B (Fig. 5b) also exhibited high heating rates up to $\sim 150^\circ\text{C}$. After this, the heating rates rose continuously from $\sim 8^\circ\text{C/min}$ to $\sim 10^\circ\text{C/min}$ up to $\sim 550^\circ\text{C}$. Between 550°C and 650°C , the heating

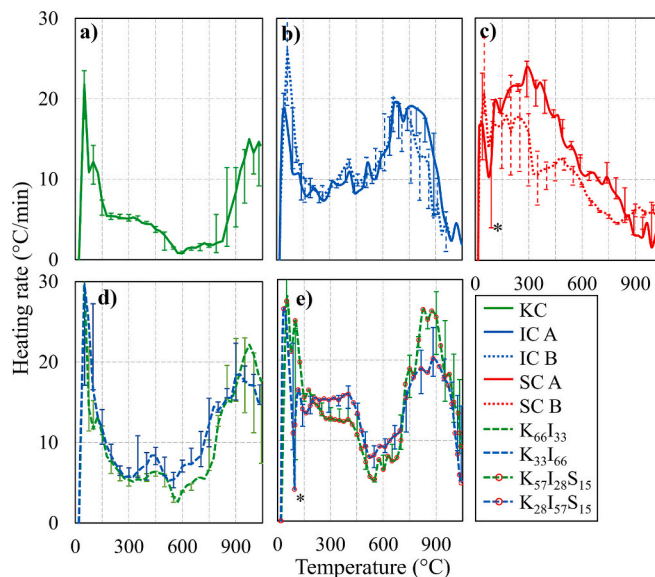


Fig. 5. Heating rate of the core temperature during microwave heating: a) KC, b) IC A&B, c) SC A&B, d) KC-IC mixtures and e) KC-IC-SC mixtures; sample mass: $\sim 400 \text{ g}$; power: 300 W at 2.45 GHz ; *evaporation plateau due to water phase change.

rate increased to 20°C/min and remained there before dropping significantly to 3°C/min – 5°C/min above 750°C .

SC A and SC B (Fig. 5c), exhibited very high initial heating rates of $\sim 15^\circ\text{C/min}$ – 25°C/min . From $\sim 300^\circ\text{C}$, the heating rate decreased continuously, reaching a constant level of $\sim 3^\circ\text{C/min}$ – 5°C/min above 800°C . Both samples exhibited a similar overall trend, with the heating rate of SC B remaining below that of SC A up to $\sim 800^\circ\text{C}$.

The kaolinitic-illitic clay mixtures (Fig. 5d) exhibited a combined heating rate curve derived from the systems shown in (Fig. 5a) and (Fig. 5b). Following an initial increase in the heating rate up to $\sim 150^\circ\text{C}$, there was an initial drop, followed by a gradual increase continuing up to $\sim 450^\circ\text{C}$. This was followed by a further drop in the heating rate. From $\sim 550^\circ\text{C}$, the heating rate rose slightly again, increasing significantly above 700°C until reaching a maximum of $\sim 20^\circ\text{C/min}$ at $\sim 900^\circ\text{C}$. The heating rate then dropped slightly to $\sim 16^\circ\text{C/min}$. The kaolinite-rich mixture showed a more pronounced decrease in heating rate at $\sim 600^\circ\text{C}$ than the illite-rich sample.

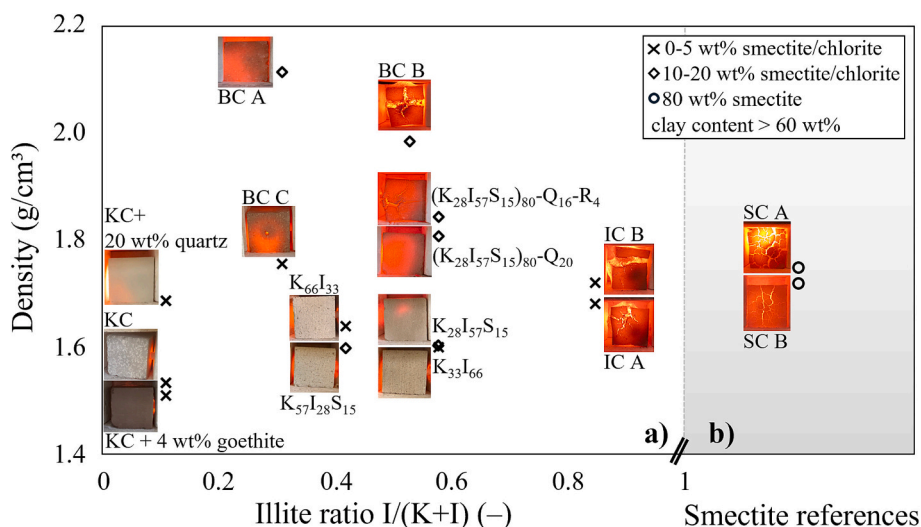


Fig. 4. Surface luminosity after microwave heating to a core temperature of 1050°C : a) mixed clay systems plotted against the illite ratio ($I/(K + I)$) and bulk density, b) additional smectite-rich reference clays plotted against bulk density; sample mass: $\sim 400 \text{ g}$; power: 300 W at 2.45 GHz .

Kaolinitic-illitic-smectitic clays (Fig. 5e) exhibited an initial heating rate of ~ 15 °C/min up to ~ 400 °C, which was notably higher than that of the variant without smectite content. However, in the high-temperature range, the heating rate dropped more sharply, reaching values of ~ 5 °C/min. Overall, the trend was similar to that of the variants without smectite (Fig. 5d).

3.4. Kaolinite-rich clay with additives

Goethite (Fig. 6a) exhibited a substantial rise in heating rate from ~ 300 °C to ~ 16 °C/min, whereas the heating rate of hematite increased more slowly. From ~ 450 °C, the heating rates of both systems dropped significantly, reaching a minimum of ~ 5 °C/min at just ~ 600 °C. The heating rate of goethite then increased again, stabilizing at ~ 12 °C/min above 700 °C. Hematite exhibited a similar trend, albeit with a lower overall heating rate.

The heating rate of charcoal (Fig. 6b) started at ~ 30 °C/min, fell to ~ 20 °C/min at ~ 450 °C and then dropped to ~ 5 °C/min above 450 °C. It then rose again, reaching between 10 °C/min and 20 °C/min at ~ 700 °C. The hard coal variant exhibited a similar, albeit weaker, effect.

Adding calcium carbonate (Fig. 6c) did not significantly alter the heating rate curve compared to the reference sample. Hydromagnesite (Fig. 6c: h-magnesite) yielded an increased heating rate up to ~ 300 °C. The mixture with siderite exhibited similar behavior to the calcium carbonate sample up to ~ 300 °C. After that, it showed an increase in heating rate, reaching a maximum at ~ 400 °C. After this point, the heating rate decreased again, reaching a minimum at ~ 550 °C. The further temperature curve was like that of goethite (Fig. 6a).

Adding 20 wt% of quartz (Fig. 6d) had only a minor effect on the heating rate compared to the reference sample up to ~ 900 °C. Above ~ 900 °C, the heating rate decreased to ~ 7 °C/min.

3.5. Brick clays and clay mixtures

Adding 20 wt% of quartz (Fig. 7a) resulted in a slightly lower heating rate than the reference sample $K_{28}I_{57}S_{15}$. After reaching a minimum of ~ 6 °C/min at ~ 550 °C, the heating rate of the quartz-containing sample increased only slightly, reaching a maximum of ~ 10 °C/min at ~ 800 °C. Reducing the 20 wt% quartz to 16 wt% and replacing the remaining 4 wt % with iron-containing mineral phases (3 wt%) and hard coal (1 wt%) increased the heating rate compared to the reference sample from ~ 150 °C to ~ 700 °C. Above ~ 800 °C, however, the heating rate dropped sharply.

BC A (Fig. 7b), which has the lowest clay mineral content of the brick clays, exhibited the lowest heating rate over the entire temperature range. The heating rate of this clay decreased up to ~ 300 °C, remaining

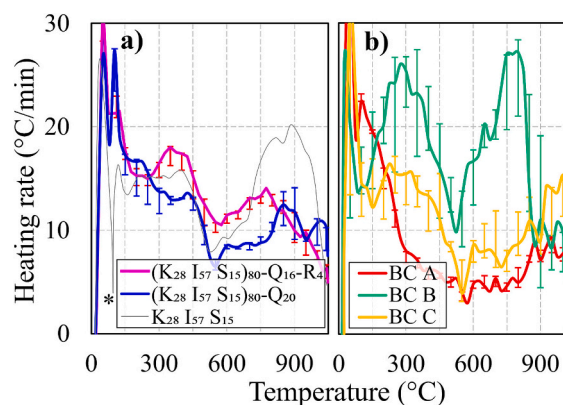


Fig. 7. Heating rate of the core temperature during microwave heating: KC-IC-SC mixtures containing only quartz, as well as those containing quartz, Fe-bearing phases and hard coal (a) and brick clays (b); sample mass: ~ 400 g; power: 300 W at 2.45 GHz; for clarity, the error bars for the reference curve $K_{28}I_{57}S_{15}$ have been omitted; *evaporation plateau due to water phase change.

relatively constant at ~ 5 °C/min up to ~ 800 °C, with a brief reduction to ~ 3 °C/min at ~ 550 °C. At the end of the test, the heating rate increased again to ~ 8 °C/min. BC C showed a curve comparable to BC A, but with heating rates that were ~ 3 °C/min higher across the entire temperature range. The illitic brick clay BC B had the highest heating rate. In the low temperature range, a maximum of ~ 25 °C/min occurred at ~ 300 °C. The heating rate then dropped to ~ 10 °C/min at ~ 550 °C, after which it rose again to ~ 25 °C/min at ~ 750 °C. Above 800 °C, the heating rate dropped sharply, with values reaching ~ 2 °C/min at the end of the test.

4. Discussion

4.1. Clay mineralogy

The heating rate of KC (Fig. 8g) was highest up to ~ 150 °C, which corresponds to literature values indicating a moderate loss tangent during the removal of adsorbed water (Fig. 8a). Initially, the dry KC remained in a weakly microwave-absorbing state, which kept the heating rate low. With the onset of dehydroxylation at ~ 450 °C (Fig. 8d), the heating rate reached its minimum near the peak of the endothermic reaction at ~ 580 °C—even though the $\tan \delta$ increases from the point of dehydroxylation. At ~ 700 °C—after dehydroxylation—the heating rate increased slightly, with a sharp rise from ~ 850 °C. It reached a maximum between 950 °C and 1000 °C, which coincided with

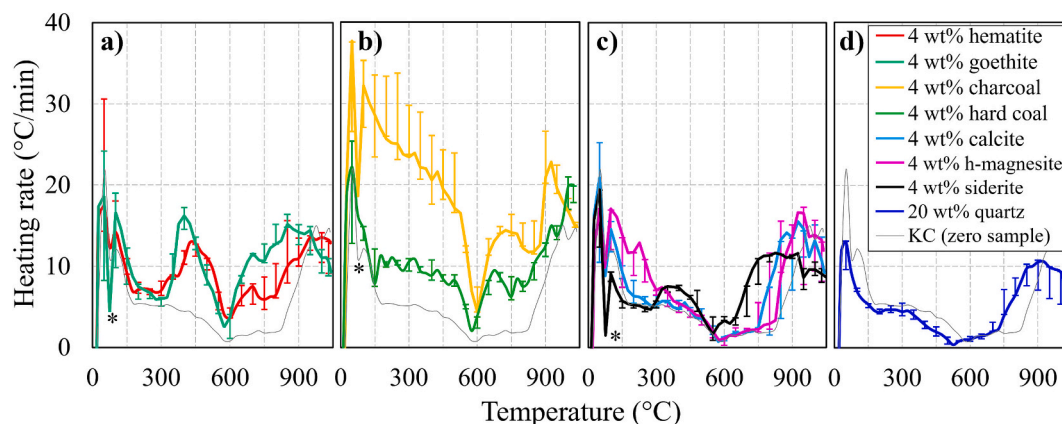


Fig. 6. Heating rate of the core temperature during microwave heating: (a) 4 wt% iron oxides, (b) 4 wt% carbonaceous materials, (c) 4 wt% carbonates and (d) 20 wt % quartz in the zero sample KC; sample mass: ~ 400 g; power: 300 W at 2.45 GHz; for clarity, the error bars for the reference curve KC have been omitted; *evaporation plateau due to water phase change.

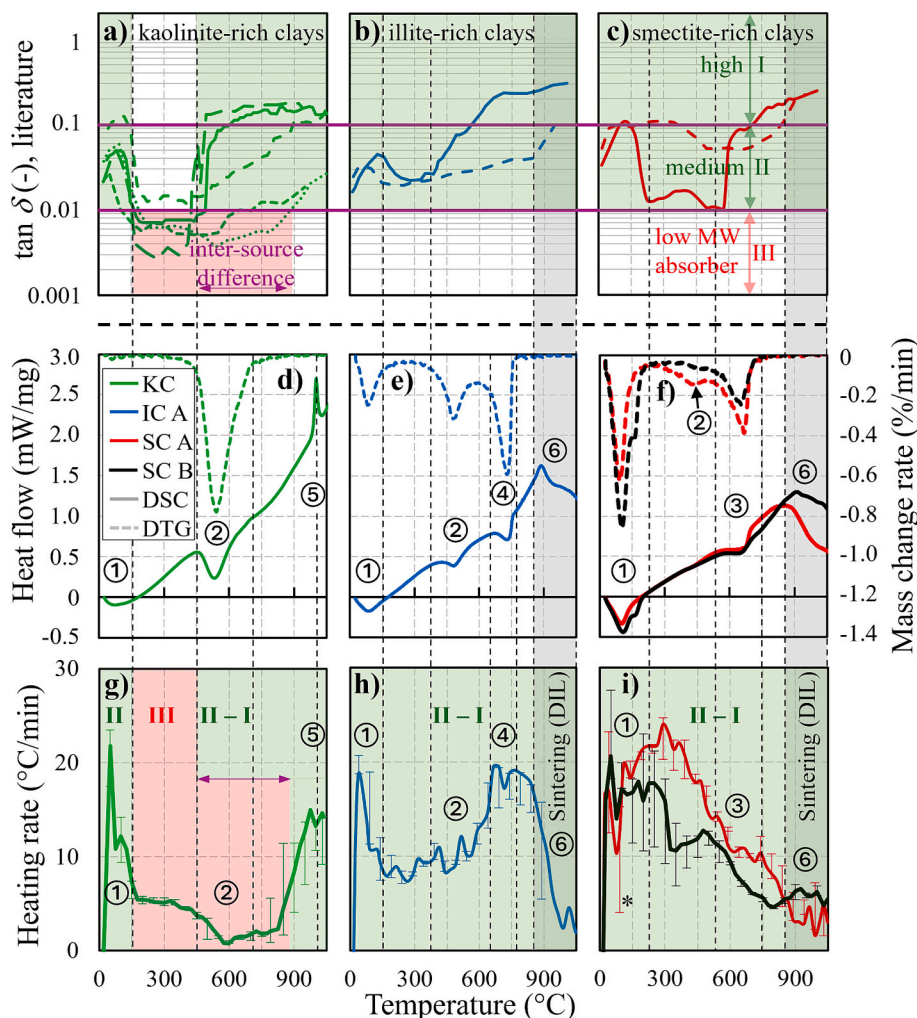


Fig. 8. Comparison of thermal and dielectric analyses with heating rates: (a–c) $\tan \delta$ from literature, (d–f) measured DSC and DTG curves, (g–i) measured heating rates; left column kaolinite-rich clays, middle illite-rich and right smectite-rich; colored areas (Vermeiren et al., 2024): red = weak (III), green = moderate (II) to good (I); (1)–(6): (1) dehydration, (2) dehydroxylation of kaolinite and/or illite, (3) dehydroxylation of smectite, (4) decomposition of calcite, (5) Al—Si spinel formation and (6) sintering (DIL); *evaporation plateau due to water phase change.

exothermic the formation of Al—Si spinel (Jasmund and Lagaly, 1993; Salmang et al., 2022). Similar behavior has been observed previously (Reinosa et al., 2019): despite the measured increase in dielectric losses at the beginning of dehydroxylation, a significant increase in microwave heating only occurred after the completion of dehydroxylation (in this case, at 650 °C), which indicates the dominant role of reaction enthalpy.

The heating rate of IC A (Fig. 8h) was also high until ~150 °C and remained significantly higher than that of KC, even after drying. This corresponds to literature values indicating a moderate continuing loss tangent (Fig. 8b), which for illite-rich clays can increase from around 350 °C. At this temperature, illite also begins to dehydroxylate slowly, peaking for IC A and IC B at 500 °C and ending slowly, which transitions into the decomposition of calcite (Karunadasa et al., 2019) at 640 °C (Fig. 8e). The dehydroxylation of illite is significantly less endothermic than that of kaolinite (cf. (2) in Fig. 8d and e). This is consistent with the observed heating rate, which rises slightly during the dehydroxylation of IC A and IC B instead of decreasing, as was the case with KC (Fig. 8g). Upon completion of the dehydroxylation of illite, the heating rate rose sharply, reaching a maximum during the endothermic calcite decomposition up to 850 °C (Fig. 8h). With the onset of sintering of IC A and IC B at ~850 °C, $\tan \delta$ increases sharply, while at the same time the DSC shows an endothermic shift and the heating rate drops sharply. Another contributing factor to the decreasing heating rate is discussed later in this chapter.

Due to their high content of water and interlayer water, SC A and SC B exhibited the highest initial loss tangent (Fig. 8c) and correspondingly high initial heating rates (Fig. 8i). As dehydration progressed, absorption decreased, resulting in a significant drop in the heating rate. SC B showed a strong decline due to its higher mass loss up to about 200 °C. Meanwhile, SC A exhibited similar behavior, with an increased heating rate. During dehydroxylation (Fig. 8f), the heating rate continued to decrease and fell further as the DIL showed strong expansion for SC A and strong shrinkage for SC B at ~850 °C with an endothermic shift in DSC.

These mechanisms are also reflected in clay mixtures. Kaolinite-rich mixtures lead to a pronounced minimum in the heating rate during dehydroxylation, while illite-rich mixtures contribute to a slow increase during and a sharp increase after dehydroxylation. Smectite enhanced the initial microwave absorption and increased the heating rates in the low-temperature range.

The increase in $\tan \delta$ with temperature in clays has been attributed to the mobilization of interlayer cations during dehydroxylation and densification-related improvements in charge transport pathways (e.g., (DTI, 2017)), as well as defect formation and thermally activated charge carriers (Reinosa et al., 2019). Another plausible explanation, particularly for Fe-rich illitic systems, could be the redistribution of structural iron during thermal transformation. Under oxidizing conditions, previous studies with long dwell times have shown that structural iron

released during the breakdown of Fe-rich illite can form poorly ordered iron oxide phases at lower temperatures and hematite near $\sim 900^\circ\text{C}$ (Murad and Wagner, 1996). Since hematite—especially defect-rich hematite—exhibits significantly higher microwave absorption than typical clay minerals (Pickles et al., 2005), the formation of such Fe-oxide phases could contribute to the observed increase in electromagnetic coupling. Additionally, higher contents of fluxing oxides (e.g., Fe_2O_3 and K_2O) may promote the formation of liquid phases at higher temperatures (Abdel-Kader et al., 2024). These phases tend to have increased ionic mobility, which can lead to greater dielectric losses and microwave energy absorption. However, this interpretation remains qualitative. Direct confirmation would require frequency-resolved dielectric measurements combined with temperature-dependent mineralogical characterization of clays with different iron contents.

Volumetric heating with microwaves can lead to an increased temperature in the center of a sample (Wagner et al., 2022). During the dehydroxylation of kaolinite (Fig. 9a, left and middle), some of the irradiated energy in KC is used for this reaction, which temporarily reduces the temperature gradient within the sample. Once the center is completely dehydroxylated, effective dissipation increases again, causing the core temperature to rise faster than in areas that are still in the endothermic reaction state. This feedback loop can lead to increasing thermal inhomogeneity, which is further intensified by the temperature-dependent $\tan \delta$ and reinforced by the exothermic peak. This results in higher temperatures inside KC compared to the surface, as can be seen in the photos taken after the experiment at a core temperature of 1050°C —similar for kaolinitic brick clay BC C (Fig. 9a, right).

Illite-rich clays, on the other hand, exhibit a different behavior (Fig. 9b, left and middle). The reaction areas are distributed over a broader temperature range and are predominantly endothermic. At the same time, the absorption level remains in the medium-to-high range, so pronounced weak absorption states do not occur. This results in smaller local differences in energy absorption, which contributes to a more homogeneous temperature distribution during microwave heating. Smectite-rich clays behave similarly to illite-rich systems and are therefore not discussed separately here.

The heating process balances out when heat losses at the surface (due to radiation, conduction, and convection) equal the absorbed microwave power (National Research Council, 1994). Local maxima or minima may also arise from temporary changes in microwave power

absorption, particularly when combined with endothermic or exothermic structural transformations. In illite-rich samples, a more uniform temperature distribution results in the entire sample being heated. In contrast, kaolinite-rich samples exhibit temperature-dependent absorption behavior: between very low and high. The formation of local inhomogeneities can lead to increased energy dissipation in the sample center while keeping the surface comparatively cool. This effect can yield to thermal-dielectric feedback, which promotes a rapid increase in the core temperature (Wagner et al., 2022). This means that the heating rate may appear to rise quickly, but the overall heating rate across the sample may not increase at the same rate. This interpretation may need to be reconsidered for larger sample sizes, as the behavior of the sample is expected to change when the microwaves' penetration depth becomes a governing factor.

The cracking observed in several samples can be attributed to a combination of rapid volumetric expansion, or bloating, and thermal gradients within the samples. In illite- and Fe-rich systems, bloating leads to internal stresses that exceed the material's mechanical strength. Additionally, inhomogeneous microwave heating can generate temperature gradients between the core and surface, which further promotes crack formation. Thus, these cracks are considered a consequence of process conditions rather than an inherent limitation of the material system.

4.2. Additive effects on microwave heating

The carbon-containing trials exhibited high initial heating rates due to their strong microwave absorption (Bhoi et al., 2019). However, this effect decreased with increasing temperature (Fig. 10a). This is due to the progressive combustion of the carbon and the associated reduction in microwave absorbent material.

The influence of iron oxides (Fig. 10a) on the heating rate is initially low and increases with rising temperature. Hematite's electrical conductivity also shows a similar upward trend (Engel and Tuller, 2014). Dissipation via the magnetic component of microwaves is negligible for hematite across the entire range because the magnetic component is very low compared to the dielectric component (Hotta et al., 2010; Peng et al., 2012). The transformation ranges of the iron oxide phases appear to significantly influence the heating rate. In goethite (Pickles et al., 2005), dehydroxylation leads to the formation of defective hematite or hydrohematite, causing a marked increase in heating behavior due to the significantly increased loss tangent—In addition, defective hematite exhibits a higher loss tangent than well-crystallized hematite. Siderite also showed an increase in heating rate, which correlates with its conversion to hematite and the associated increase in loss factors around

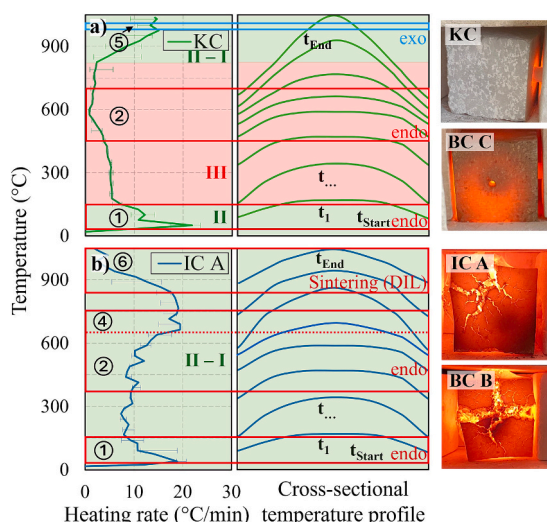


Fig. 9. Assumed heating profile through the cross-section: a) KC and b) IC A; heating rate (left), assumed heating profile (middle), and final sample pictures of KC & kaolinitic brick clay BC C (right top) as well as IC A & illitic brick clay BC B (right bottom); colour coding: red is weak (III), green is medium (II) to good (I); (1)–(6): (1) dehydration (2) dehydroxylation of kaolinite and/or illite (4) decomposition of calcite (5) Al–Si spinel formation (6) sintering.

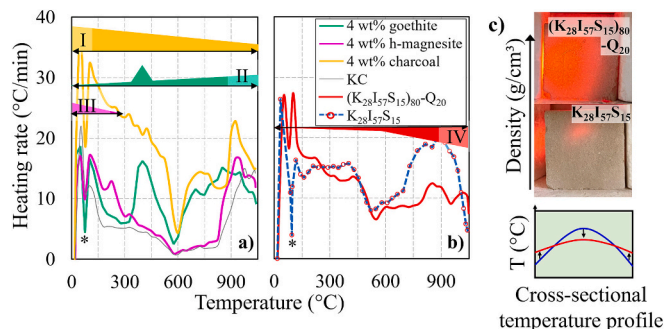


Fig. 10. Influence of additives on microwave heating behavior: (a) qualitative comparison of heating rates for carbonaceous (I), iron-bearing (II), hydrated phases (III); (b) increased density caused by quartz (IV); and (c) effect of quartz/density on surface luminosity and inferred cross-sectional temperature profile, illustrating the impact of bulk density on heating homogeneity; colored bars indicate relative changes over temperature; for clarity, the error bars for all samples have been omitted; *evaporation plateau due to water phase change.

400 °C (Lovás et al., 2010). However, the relative effect depends strongly on the initial composition. While iron oxides can significantly increase absorption in low absorbing KC, their additional effect is less pronounced in mixtures that already have higher absorption (Fig. 7b).

The hydromagnesite (Fig. 10a: h-magnesite) exhibited dehydration and increased heating rates (Fig. 10a) up to 340 °C. Neither calcium carbonate nor the dehydrated magnesite increased the heating rate in this mixture, which confirms the loss-free behavior reported in the literature (DTI, 2017; Lovás et al., 2010; Znamenackova et al., 2014).

The reduced heating rate of the quartz-containing mixtures (Fig. 10b) at low temperatures is consistent with the low absorption of quartz (DTI, 2017). The greater decrease at high temperatures may be related to increased sample density and an improved thermal energy balance, as evidenced by the more homogeneous luminance distribution (Fig. 10c). The cross-sectional temperature profile schematically illustrates the assumed internal temperature distribution within the sample.

4.3. Brick clays

The heating behavior of brick clays is very complex due to their mineralogical diversity and cannot be predicted with absolute certainty based on the results of this study. Nevertheless, relative trends in heating behavior can be identified. All brick clays exhibited a minimum heating rate during kaolinite dehydroxylation. In the case of illitic compositions (such as BC B), a maximum also occurred after the illite dehydroxylation. Due to the significant chemical and mineralogical variability within the smectite and illite groups, their impact on microwave-induced heating behavior remains unclear. Comparing the two smectites SC A and SC B revealed clear differences in the absolute heating rates. At the same time, natural brick clays often contain other minerals, such as chlorite, or alternating clay mineral layers which could not be evaluated in this study.

In addition, brick clays often contain iron-bearing phases that increase overall microwave absorption. Organic components lead to additional heating, particularly at lower temperatures, as reflected in the initial behavior of BC B and BC C compared to BC A—due to exothermic reactions or microwave coupling with carbonaceous components.

4.4. Conclusion

This work demonstrates that mineralogical composition and their thermal behavior significantly influence microwave heating behavior. Clays, rich in illite and smectite, exhibited stronger overall heating than kaolinitic clays. Kaolinite-dominated materials tended to heat non-homogeneously, while illitic and smectitic clays heated more uniformly due to their higher loss tangent in the complete temperature range. Additionally, illitic clays tend to bloat in a microwave field.

At lower temperatures, smectite-rich systems exhibited the highest initial heating rates, followed by illite-rich clays, while kaolinite-rich clays showed the lowest microwave energy absorption. During dehydroxylation, the heating rate decreased in all systems, most significantly in kaolinite and least significantly in illite. After completion of dehydroxylation, illite-rich systems exhibited a significant increase in the heating rate. In contrast, the onset of sintering was associated with a significant decrease in the heating rate. Kaolinite-rich clays exhibited an increase in heating rate at the center of the sample at higher temperatures. However, sintering of kaolinite would require temperatures higher than those examined here.

Additional additive phases primarily affect the intensity of microwave absorption. Carbonaceous components and iron oxides significantly increase heating rates. Quartz reduces heating intensity, but it may contribute to a more homogeneous temperature profile due to increased sample density.

While the behavior of complex brick clays is difficult to predict, their microwave response can be reasonably estimated based on the dominant

clay mineralogy and composition of additives. This study advances the understanding of the interplay between mineralogy, thermally induced reactions, and microwave heating in clays. It provides a basis for adapting electrified thermal treatment processes to mineralogical characteristics.

CRediT authorship contribution statement

Andreas Blüml: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Markus Hainthaler:** Project administration, Funding acquisition. **Karl-Christian Thienel:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors gratefully acknowledge the financial support provided by Projektträger Jülich (PtJ) within the project [03EN1082B]. The authors also thank Schlagmann Poroton GmbH & Co. KG for providing a wide range of clay materials and access to their laboratory facilities. The authors further acknowledge the Technology Campus Teisnach (TCTS) for their support in the development and implementation of the temperature measurement system. The work was also supported by the joint academic partnership 'Infrastructure, Building and Urban Development' of the Bavarian Academic Forum – BayWISS.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.clay.2026.108307>.

Data availability

Data will be made available on request.

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