

Thermal conductivity of traditional ceramics Part II: Influence of mineralogical composition

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Abstract

The thermal conductivity of traditional ceramic materials is known to be a function of their porosity or bulk density. However, the scatter in the thermal conductivity–bulk density data in certain studies, particularly when data from industrially processed brick are involved, suggests that thermal conductivity depends, apart from porosity, on other characteristics such as mineralogical composition, microstructure, humidity, and the presence of soluble salts.

A standard red-firing clay used in brick manufacture has been used in this study with a view to systematising the impact of the different variables that could influence thermal conductivity and mechanical strength. Part I of the study presented the results obtained when the dry bulk density of the pieces and their firing temperature were modified.

Part II examines the influence of the mineralogical composition of the starting raw materials mixture on the thermal conductivity and mechanical strength of clay brick products. The findings suggest that to manufacture traditional ceramics with high thermal insulation and appropriate mechanical properties, it is advisable to use illitic-kaolinitic clays. Large-sized potassium feldspar and quartz particles adversely affect fired mechanical strength. In addition, quartz has high thermal conductivity. The addition of carbonates or the use of calcareous clays has a positive effect on mechanical strength, because carbonate acts as a pore-forming agent and generates crystalline phases during firing that enhance mechanical strength.

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1. Introduction

The thermal conductivity of traditional ceramic materials is known to be a function of their porosity or bulk density [1–4]. However, the scatter in thermal conductivity–bulk density data in certain studies, particularly when data from industrially processed brick are involved, suggests that thermal conductivity depends, apart from porosity, on other characteristics such as mineralogical composition, microstructure, humidity and on the presence of soluble salts [5–9].

In the context of a systematic examination of the different variables that could influence thermal conductivity, a previous paper [10] presented the results obtained when dry bulk density and firing temperature were modified. That study showed that

thermal conductivity does not solely depend on the total porosity of the fired pieces, but that microstructure and pore size distribution need to be considered, which depend on the degree of sintering.

This paper examines the influence of the mineralogical composition of the starting raw materials mixture on the thermal conductivity and mechanical strength of traditional ceramic materials. A standard red-firing clay for brick manufacture was used as starting material into which different raw materials were alternately incorporated to modify its mineralogical composition.

2. Experimental

A red-firing clay (IC) studied in a previous paper [10] was used as starting material. Clay IC mineralogical composition was modified by replacing 15% and 30% by weight of clay IC,

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Table 1
Chemical composition (wt%), true density, and average particle size of the raw materials used.

Oxide	IC	Q	PF	C	D	KC
SiO ₂	58.5 ± 0.3	98.5 ± 0.4	68.3 ± 0.3	0.5 ± 0.1	0.05 ± 0.01	59.2 ± 0.3
Al ₂ O ₃	20.2 ± 0.2	0.50 ± 0.03	17.2 ± 0.1	0.10 ± 0.03	<0.01 ± 0.01	26.5 ± 0.2
Fe ₂ O ₃	7.14 ± 0.05	0.03 ± 0.01	0.10 ± 0.02	0.05 ± 0.01	<0.01 ± 0.01	1.20 ± 0.05
CaO	1.03 ± 0.04	0.33 ± 0.01	0.50 ± 0.02	55.3 ± 0.2	30.3 ± 0.1	0.65 ± 0.02
MgO	1.56 ± 0.09	0.02 ± 0.01	0.08 ± 0.01	0.10 ± 0.01	21.4 ± 0.1	0.25 ± 0.01
Na ₂ O	0.21 ± 0.03	0.01 ± 0.01	2.20 ± 0.10	<0.2 ± 0.2	–	0.05 ± 0.01
K ₂ O	4.35 ± 0.09	0.15 ± 0.01	11.0 ± 0.1	<0.04 ± 0.04	–	0.70 ± 0.02
L.O.I.(1025 °C)	6.09 ± 0.08	0.40 ± 0.03	0.50 ± 0.04	43.5 ± 0.1	45.8 ± 0.1	10.0 ± 0.1
True density (kg/m ³)	2680 ± 15	2650 ± 15	2500 ± 15	2720 ± 15	2850 ± 15	2650 ± 15
d ₅₀ (μm)	6.4 ± 0.1	40 ± 0.8	27 ± 0.5	14 ± 0.3	24 ± 0.5	4.3 ± 0.1

alternately, with each of the following raw materials: quartz (Q, Sibelco SE-6), potassium feldspar (PF, Incusa FK-100), calcite (C, Zaira Calaf C-40), dolomite (D, Quimiamel D-150) and a clay with a higher kaolinite content (KC, Imerys RC-593). Clay KC was included in order to establish the influence of the type of clay mineral. Table 1 shows the chemical composition, true density, and average particle size of the raw materials used.

In order to prepare the test pieces, clays IC and KC were dry milled in a hammer mill to a particle size fraction below 0.5 mm. Compositions of clay IC, in which 15% and 30% by weight of clay IC were alternately replaced with each raw material, were prepared in a lab mixer. The resulting powder was then moistened to 0.055 kg water/dry solid kg and test pieces were pressed at 25 MPa. These pieces were dried and then fired in an electric kiln at a heating rate of 5 °C/min to a peak temperature of 1000 °C, with a 60-min dwell at peak temperature. Cooling was performed by natural convection.

Dry and fired bulk density was determined by the mercury displacement method. The mechanical strength of the fired pieces was determined by three-point bending in a mechanical testing machine with bar-shaped test pieces, 80 mm long, 20 mm wide, and 6 mm thick.

The thermal conductivity measurement tests were conducted according to an adapted experimental procedure of international standards ISO 8301:1991: thermal insulation – determination of steady-state thermal resistance and related properties – heat flow meter apparatus, and ISO 8302:1991: thermal insulation – determination of steady-state thermal resistance and related properties – guarded hot plate apparatus.

Table 2
Characteristics of the test pieces.

Composition (wt%)	ρ_s (kg/m ³)	ρ_c (kg/m ³)	λ (W/(m K))	σ (MPa)
100% IC	1955 ± 2	1973 ± 3	0.56 ± 0.01	18.0 ± 0.4
85% IC + 15% Q	1974 ± 2	1942 ± 2	0.57 ± 0.01	11.1 ± 0.6
70% IC + 30% Q	1983 ± 4	1920 ± 2	0.58 ± 0.01	7.0 ± 0.2
85% IC + 15% PF	1978 ± 2	1938 ± 2	0.52 ± 0.01	12.2 ± 0.3
70% IC + 30% PF	1940 ± 2	1890 ± 1	0.50 ± 0.01	8.6 ± 0.5
85% IC + 15% C	1979 ± 2	1771 ± 2	0.51 ± 0.01	16.3 ± 0.4
70% IC + 30% C	1992 ± 2	1621 ± 4	0.46 ± 0.01	13.2 ± 0.3
85% IC + 15% D	2006 ± 2	1814 ± 3	0.52 ± 0.01	15.3 ± 0.3
70% IC + 30% D	2065 ± 3	1676 ± 3	0.49 ± 0.01	11.5 ± 0.2
85% IC + 15% KC	1974 ± 2	1982 ± 2	0.50 ± 0.01	19.9 ± 0.3
70% IC + 30% KC	1959 ± 3	1960 ± 2	0.47 ± 0.01	19.1 ± 0.3

Finally, the porous texture of the pieces was observed in a scanning electron microscope.

3. Results

3.1. Influence on bulk density

Table 2 details the raw materials contained in each composition, in addition to the dry bulk density (ρ_s), fired bulk density (ρ_c), thermal conductivity (λ), and mechanical strength (σ) of the test pieces.

The table shows that the introduction of the different raw materials in the compositions modifies dry and fired test piece properties. The differences in ρ_s are related to the particle size distribution of the different raw materials, as well as to differences in their true density (Table 1). The raw materials with the highest true density are dolomite (2.85 g/cm³) and calcite (2.72 g/cm³), correlating with the higher ρ_s values for the pieces containing these raw materials.

The bulk density values of the fired test pieces are plotted versus the clay IC content alternately replaced with each respective raw material in the starting composition in Fig. 1. The figure shows that ρ_c decreases in all the compositions with respect to clay IC, except in the kaolinitic clay-containing composition, which yields similar values. The differences observed between the test compositions may be attributed to

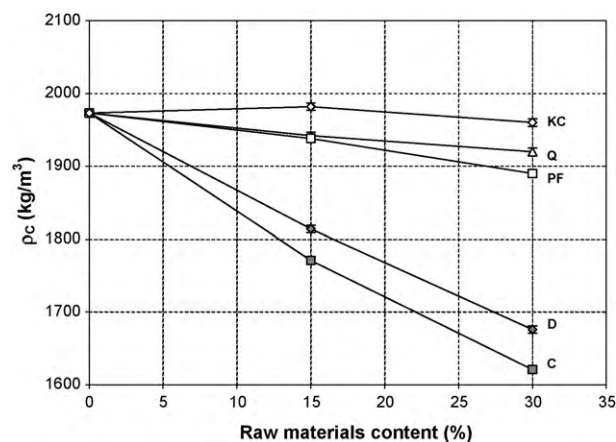


Fig. 1. Evolution of fired bulk density with the raw materials contained in the compositions.

changes in the loss on ignition of the raw materials (Table 1) and to the refractory nature of some minerals at working temperature (1000 °C). Thus, the compositions with calcite and dolomite provide the lowest fired bulk density owing to calcite and dolomite decomposition at temperatures above 700 °C [11]. The bulk density values for the compositions containing quartz and feldspar are similar and close to those of clay IC. Quartz and feldspar reduce the loss on ignition of the mixture and act as fillers at the firing temperature tested, as will be seen below.

3.2. Influence on thermal conductivity

Fig. 2 shows how the thermal conductivity of the fired pieces evolves with the different raw materials in the compositions. The partial replacement of clay IC with the respective raw materials reduces thermal conductivity, except when quartz is incorporated into the composition: this decrease is greater for the kaolinitic clay and calcite, and slightly lower for potassium feldspar and dolomite. In contrast, quartz raises thermal conductivity.

One reason for the change noted is the thermal conductivity of the different phases in the fired pieces. These phases come from minerals (quartz and feldspar) that do not fuse or dissolve during firing, from the arising crystalline phases (calcium and magnesium silicates and aluminosilicates), and from the presence of amorphous and glassy phases resulting from the dehydroxylation and subsequent melting of some clay minerals [12–14]. In addition, differences in thermal conductivity can also stem from changes in the bulk density and microstructure of the fired pieces as a result of the loss on ignition and particle size of the raw materials.

Fig. 3 shows the scanning electron micrographs of polished cross-sections of fired test pieces corresponding to the 100% IC composition and to the 70% IC compositions with 30% quartz, calcite, and feldspar, respectively. The piece made from clay IC (Fig. 3(a)) shows quartz (1) and feldspar (2) particles in a clayey matrix with interconnected pores resulting from incipient vitrification. Some highly densified agglomerates (3) are present, which come from clay agglomerates in the IC clay.

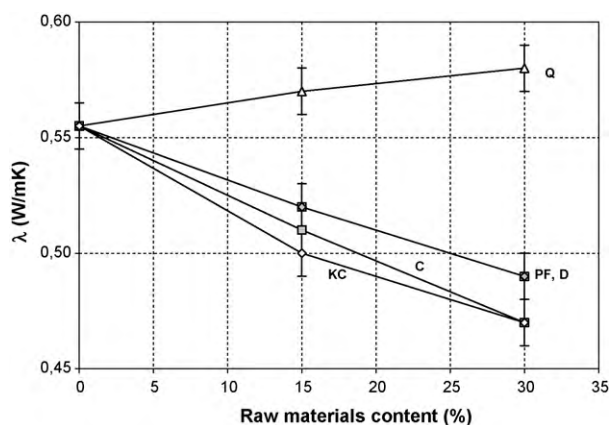


Fig. 2. Evolution of thermal conductivity with the raw materials contained in the compositions.

The introduction of 30% quartz (Fig. 3(b)) reduces the clay matrix content and increases the number and size of quartz particles. This test piece contains larger pores than the IC composition (Fig. 3(a)), while quartz particles have not yet begun to dissolve at 1000 °C. The microstructure of the piece with 30% PF (Fig. 3(d)) is very similar to that of the quartz-containing piece, the only difference being feldspar particle size, which is smaller than quartz particle size (Table 1). It may also be observed that, just as in the quartz-containing piece, the feldspar particles have not dissolved or fused at 1000 °C. Despite the similarity of the microstructure of the pieces with 30% quartz and 30% potassium feldspar, their conductivity is different, which evidences the effect of the crystalline phases on conductivity ($\lambda = 7.7$ W/(m K) for the quartz and $\lambda = 2.3$ W/(m K) for the potassium feldspar [15]).

The test piece with 30% calcite (Fig. 3(c)) is much more porous than the other test pieces, pore size (dark areas in the figure) being similar to that of the initial calcite particles (~ 15 μ m). This suggests that CaO reacts at the calcite grain boundaries with Al_2O_3 and SiO_2 from clay mineral dehydroxylation to form calcium crystalline phases. The high porosity stemming from calcite particle decomposition leads to low thermal conductivity.

Fig. 4(a) shows the microstructure of the piece with dolomite, in which dark coarse particles can be observed. According to EDX analysis, these particle cores are very rich in magnesium while the grain boundaries contain high concentrations of calcium and magnesium, in addition to silicon and aluminium. When these particles are more closely examined (Fig. 4(b)), small crystals and fine pores can be observed. Particle appearance and EDX analysis suggest that CaO diffuses towards the particle boundary during firing and reacts with silica and alumina from the clay minerals, producing crystalline phases, namely calcium aluminosilicates, which also contain magnesium. Once all the CaO has reacted, the surplus magnesium remains unreacted. A core of MgO crystals with pores thus develops inside the initial dolomite particles. In contrast, the microstructure of the calcite-containing piece differs completely. In this case, all the CaO reacts, producing a large centre pore surrounded by calcium silicates and aluminosilicates.

The differences in porous texture between the fired pieces whose starting composition contained either calcite or dolomite hardly affect thermal conductivity. These results indicate that total porosity is the main factor influencing thermal conductivity in these compositions.

Fig. 5 shows the relationship between thermal conductivity and fired bulk density. The figure also includes data for clay IC fired at 1000 °C, in which pressing pressure was modified (the data are presented in Part I [10]).

The raw materials that raise test piece thermal conductivity lie above the clay IC line, while those below this line have the opposite effect. Quartz, and to a lesser degree calcite and dolomite, raise thermal conductivity, while potassium feldspar and particularly the kaolinitic clay lower thermal conductivity.

The composition with quartz displays the highest thermal conductivity, which is related to the high thermal conductivity of quartz (7.7 W/m K). Quartz particles do not fuse or dissolve

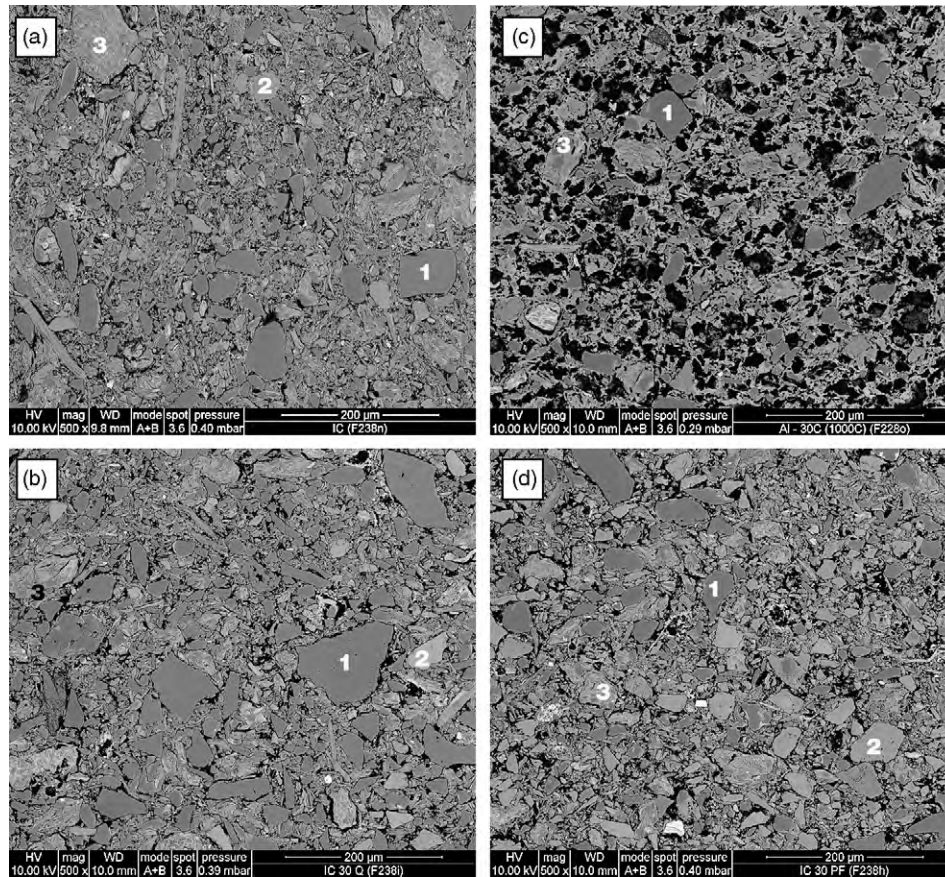


Fig. 3. Micrographs of cross-sections of the fired pieces: (a) 100% IC, (b) 70% IC + 30% Q, (c) 70% IC + 30% C, (d) 70% IC + 30% PF; 1, quartz particles; 2, potassium feldspar particles; 3, clay agglomerates.

during firing at 1000 °C [10], thus leaving large-sized particles that enhance the heat flow. This finding is consistent with Erker's studies on the thermal conductivity of industrial bricks [6].

Calcite and dolomite reduce clay IC thermal conductivity when the process variables are held constant (Table 2). This is because calcite and dolomite decompose at temperatures above 700 °C, which greatly raises porosity, as Figs. 3(b) and 4(a) show. At the same fired bulk density (Fig. 5), however, calcite and dolomite increase thermal conductivity. In these composi-

tions, calcium and magnesium silicate and aluminosilicate crystalline phases form at temperatures above 860 °C [18].

Certain studies [17] indicate that, in materials with the same chemical composition, those with crystalline phases exhibit higher thermal conductivity than those containing amorphous or glassy phases. Calcite compositions need to be fired above the crystalline phase formation temperature (860 °C) to reach the required properties (strength, moisture expansion, etc.) [16]. At lower temperatures thermal conductivity decreases due

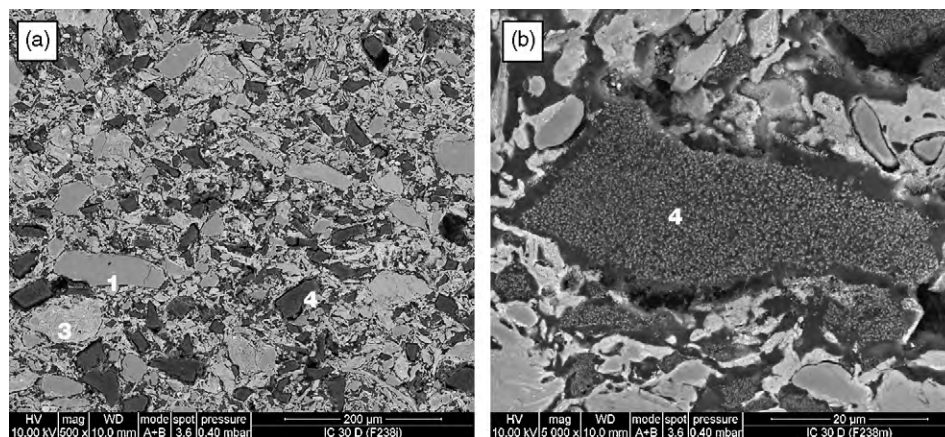


Fig. 4. Micrographs of cross-sections of the fired pieces: (a) 70% IC + 30% D, (b) partly reacted dolomite particle, surrounded by calcium–magnesium crystalline phases; 1, quartz particle; 3, clay agglomerate; 4, magnesium-rich particle.

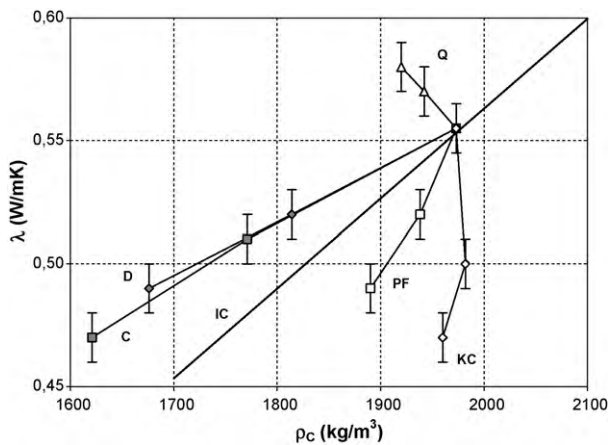


Fig. 5. Evolution of thermal conductivity with fired bulk density.

to the high content of amorphous phase but other properties as moisture expansion will rise.

Other papers report similar findings [1,5,6,19] in samples taken from fired industrial bricks and from fired laboratory compositions. These studies indicate that calcium carbonate-containing compositions display higher thermal conductivity than those without calcium carbonate.

As Fig. 5 shows, the partial replacement of clay IC with potassium feldspar reduces the thermal conductivity of the IC composition. This effect may stem from the reduction in initial quartz content of the clay IC composition when clay IC (which contains quartz) is partly replaced with feldspar. Potassium feldspar thermal conductivity is 2.3 W/(m K), which is much lower than that of quartz (7.7 W/(m K)).

The replacement of clay IC with kaolinitic clay (KC) leads to a marked decrease in thermal conductivity. This could be due to a change in clay matrix porosity, which reduces the continuity of the solid phase. The microstructure of the agglomerates in the 100% IC composition and in the 70% IC + 30% KC composition is shown in Fig. 6. The presence of illite in clay IC leads to dense agglomerates with elongated pores (Fig. 6(a)). In contrast, the clay KC agglomerates, which contain a larger quantity of kaolinite, exhibit very little

Table 3

Values of coefficients b_0 , b_1 , and b_2 .

Raw material	b_0	$b_1 \times 10^4$	$b_2 \times 10^3$	r^2
IC	−0.172	3.68	−	0.995
Q	−0.172	3.68	1.54	0.994
PF	−0.170	3.66	−0.89	0.995
C	−0.166	3.66	1.26	0.994
D	−0.172	3.68	1.51	0.996
KC	−0.161	3.62	−2.82	0.979

densification and keep the small pores (Fig. 6(b)). The absence of dense areas reduces thermal conductivity [20]. This suggests that, in addition to total porosity, pore size distribution and, hence, the continuity of the solid phase are determining factors in thermal conductivity.

Thermal conductivity depends on fired bulk density and on the mineralogical composition of the raw materials mixture used. In order to relate thermal conductivity to both variables, a multi-parametric fit was derived for each composition using fired bulk density (ρ_c) and raw materials content (M), as a percentage, as independent variables. Data for clay IC fired at 1000 °C, using different pressing pressures [10], in addition to the values of the compositions with 15% and 30% of each raw material, were used to obtain these fits. The fitting equation was as follows:

$$\lambda = b_0 + b_1 * \rho_c + b_2 * M$$

The coefficients obtained for each series of compositions are presented in Table 3. In all the studied compositions, the values of coefficients b_0 and b_1 remain practically constant, whereas that of b_2 changes. The value of b_2 shows how each raw material affects the thermal conductivity of the respective mixtures with clay IC, independently of any changes in fired bulk density.

Quartz and dolomite raise thermal conductivity the most, with b_2 values close to 1.5, followed by calcite with a b_2 value of 1.26. Potassium feldspar and, in particular, kaolinitic clay reduce thermal conductivity.

Finally, Fig. 7 shows the thermal conductivity values calculated with the fitting equations versus the experimental values for all the compositions. Note that the calculated values

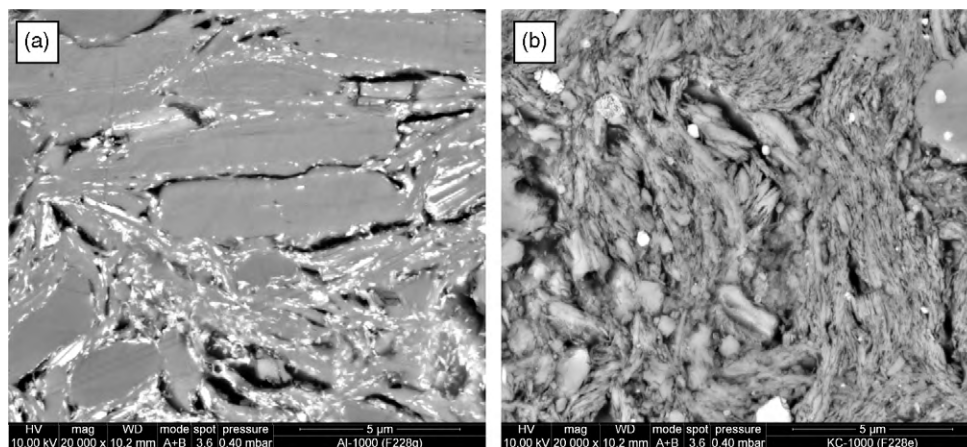


Fig. 6. (a) Micrograph of clay agglomerates in the 100% IC composition; (b) micrograph of clay agglomerates in the 70% IC + 30% KC composition.

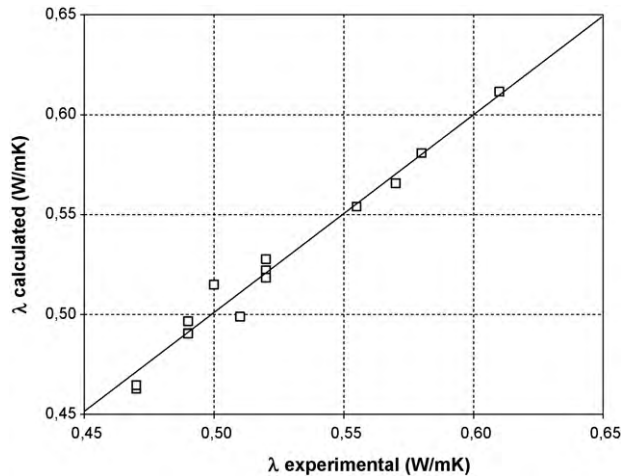


Fig. 7. Calculated versus experimental thermal conductivity values.

lie very close to the experimental ones, thus validating the values of the fitting equation coefficients.

3.3. Fired mechanical strength

The evolution of fired mechanical strength with raw materials content is plotted in Fig. 8. The figure shows that the partial replacement of clay IC with the respective test raw materials reduces fired mechanical strength except when clay KC is introduced, in which case fired mechanical strength does not change significantly.

Mechanical strength decreases because of increased fired porosity and microstructural flaw (crack) size, which occurs when the raw materials particle size is large enough [21]. To distinguish both effects, the values of fired mechanical strength are plotted versus fired bulk density in Fig. 9. Data for clay IC from [10] have also been included.

The results show that the raw materials can be grouped according to their effect on clay IC fired strength when fired bulk density is kept constant. Thus a first group, comprising calcite and dolomite, raises fired mechanical strength, which rises as the calcite and dolomite content in the IC composition increases. The literature suggests that this improvement is due

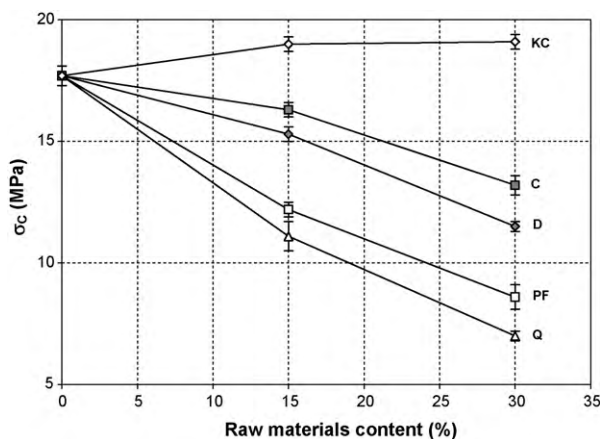


Fig. 8. Evolution of fired mechanical strength with raw materials content.

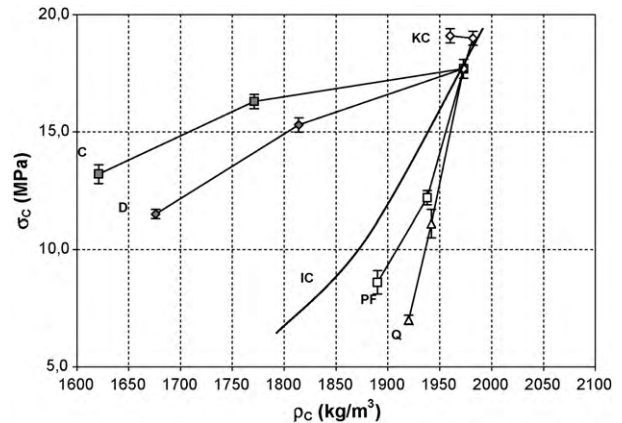


Fig. 9. Evolution of mechanical strength with fired bulk density.

to a homogenous distribution of the grains and pores [14], but Fig. 3(b) shows, however, that the increased fired mechanical strength stems from the formation of new crystalline phases during firing. Calcite provides higher mechanical strength than dolomite, owing to the presence of unreacted MgO.

A second group, comprising potassium feldspar and quartz, reduces fired mechanical strength, this reduction being most pronounced when quartz is introduced in the IC composition. These raw materials provide the mix with large particles that do not fuse or dissolve during firing: instead they increase microstructural flaw size (Fig. 3). During cooling, the larger particle size of quartz and the allotropic transformation of $\beta \rightarrow \alpha$ quartz around 573 °C further reduce mechanical strength. At this temperature, quartz particles shrink significantly and separate from the surrounding clay matrix, raising microstructural flaw size.

The third group, consisting of kaolinitic clay, slightly raises fired mechanical strength in the IC composition. The larger quantity of small clay particles in the mixture stemming from the introduction of clay KC does not increase microstructural flaw size, so that fired mechanical strength is held.

3.4. Thermal conductivity–mechanical strength analysis

A key aim of the ceramic industry is to improve the thermal insulation of ceramic products without impairing their mechanical properties. This section analyses the thermal conductivity and mechanical strength of test pieces obtained under the same conditions (pressing pressure of 25 MPa and peak firing temperature of 1000 °C).

Mechanical strength is plotted versus thermal conductivity of the test compositions in Fig. 10. The intersection of the dashed lines corresponds to clay IC. The position of the experimental data in relation to the dashed lines allows four areas to be identified, which classify the effect of each raw material on the respective compositions.

Area 1, which identifies compositions that raise both mechanical strength and thermal conductivity, contains no test composition. Area 2, which identifies compositions that reduce mechanical strength and raise thermal conductivity, contains the compositions with quartz: quartz is therefore the most

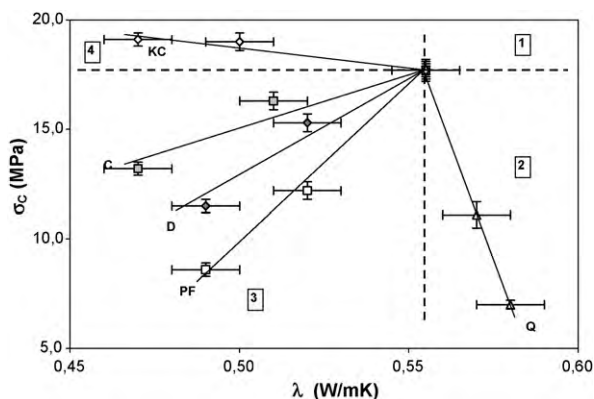


Fig. 10. Fired mechanical strength versus thermal conductivity.

unsuitable tested mineral because it worsens thermal insulation and impairs the mechanical strength of the studied ceramic body. This is due to the high thermal conductivity of quartz and the increase in microstructural flaw size.

Most of the test compositions are located in Area 3, which identifies a simultaneous decrease in thermal conductivity and mechanical strength. The compositions containing calcite and dolomite reduce thermal conductivity significantly, but only slightly reduce mechanical strength: the reduction in thermal conductivity is caused by the porosity that develops with carbonate decomposition, while the crystallisation of calcium and magnesium silicates and aluminosilicates strengthens the pieces (offsetting the increased porosity), particularly when calcite is used. The inert behaviour of potassium feldspar at 1000 °C raises fired porosity and flaw size, thus simultaneously reducing both properties, particularly mechanical strength, since no strengthening mechanism develops in these minerals.

Finally, the compositions with kaolinitic clay lie in Area 4. This raw material reduces thermal conductivity while slightly raising fired mechanical strength. These effects result from the small particle size and refractoriness of clay KC, which generate a large quantity of small pores, reduce the volume of the densified areas, and do not increase microstructural flaw size in the pieces. This is therefore the most suitable tested raw material for enhancing the heat insulation capacity and fired mechanical strength of the IC composition.

4. Conclusions

This study examines the influence of the mineralogical composition of a brick body on thermal conductivity and mechanical strength. The findings suggest that to manufacture lightweight ceramics with high thermal insulation and appropriate mechanical properties, it is advisable to use clays that contain kaolinite and illite. Kaolinite gives rise to a porous matrix with small pores, which reduce thermal conductivity without impairing fired mechanical strength.

The study shows that, in the tested compositions, large-sized potassium feldspar and quartz particles adversely affect fired mechanical strength. In addition, quartz has high thermal conductivity. In contrast, the addition of carbonates or the use of calcareous clays has a positive effect on mechanical strength,

particularly when the compositions contain calcite, because carbonate acts as a pore-forming agent and generates crystalline phases during firing that enhance mechanical strength.

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