



# Using marble and granite rejects to enhance the processing of clay products

A.M. Segadães<sup>a,\*</sup>, M.A. Carvalho<sup>b</sup>, W. Acchar<sup>c</sup>

<sup>a</sup>University of Aveiro, Department of Ceramics and Glass Engineering (CICECO), 3810-193 Aveiro, Portugal

<sup>b</sup>Polytechnic Institute of Viana do Castelo, School of Technology, 4900-348 Viana do Castelo, Portugal

<sup>c</sup>Federal University of Rio Grande do Norte, Department of Physics, 59072-970 Natal-RN, Brazil

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## Abstract

The heterogeneity of traditional clay-based materials accommodates a variety of waste products compositions with little sacrifice of the final product properties and a much welcome relief on waste disposal concerns. Hence, the incorporation of industrial wastes or sub-products in bricks and tiles is becoming common practice. However, the continued depletion of natural resources throws new light on the potential use of some industrial wastes and natural sub-products as full-fledged alternative ceramic raw materials.

This work shows the changes in properties of an industrial red-clay-based mixture, already in use in the production of floor tiles, due to additions of an ornamental stone cutting reject (marble and granite). Samples containing up to 30 wt.% reject were uniaxially pressed and sintered in air in an electric furnace (1100–1150 °C, for 2 h). The results obtained (X-ray diffraction and fluorescence, thermal analysis, firing shrinkage, water absorption and mechanical strength), explain the effect that the marble and granite reject additions have on the clay mixture behaviour and show how the properties of the sintered red-clay products can be improved, with the possibility of using lower firing temperatures.

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## 1. Introduction

Every human activity, in general, and industrial activity, in particular, has some impact on the environment, positive or negative, more or less

difficult to ameliorate and minimize, and is affected back by the environment. Economic and demographic growth, with the adoption of higher and healthier living standards, demands the control and management of this delicate balance, which is a formidable challenge that society must meet. The industrial activity, needing to continuously improve living standards, drags with itself two environmental difficulties: (1) it ultimately feeds on natural non-renew-

\* Corresponding author. Tel.: +351 234 370 236; fax: +351 234 425 300.

E-mail address: [segadaes@cv.ua.pt](mailto:segadaes@cv.ua.pt) (A.M. Segadães).

able resources that are getting scarce and will reach exhaustion sooner or later; and (2) it produces increasing amounts of waste materials.

Generally speaking, industries of all kinds have already looked for alternative, less expensive, natural raw materials and have optimised their processes in order to make the most of what is available to them with the simultaneous benefit of producing less waste.

Waste is any thing or substance whose owner or producer wants or intends to dispose of and is regarded as having zero cost. However, even when a given waste material is considered generally inert and non-hazardous, it cannot be simply discarded and must be disposed of at a landfill site, at the very least. It is becoming frequent that landfill sites dedicated to the disposal of urban solid waste do not accept industrial waste and the industry becomes responsible for the conditioning and keeping of the waste.

Thus, industry faces two new concerns: (1) natural raw materials are getting more expensive; and (2) waste materials are becoming a burden with a rising price tag on it. Recycling waste materials is nothing new, particularly in energy-intensive industries (e.g. iron and steel, aluminium, glass, paper). When it comes to consuming or reusing other industries wastes, the ceramic industry, particularly the sector devoted to the fabrication of building products, stands at the top of the list. Natural raw materials used in the fabrication of clay-based ceramic products show a wide range of compositional variation and the resulting products are very heterogeneous. For this reason, such products can tolerate further composition fluctuations and raw material changes and the ceramic industry is very capable of incorporating a variety of waste materials (Anderson and Jackson, 1983; Alleman, 1989; Bazadjiev et al., 1991; Dominguez and Ulmann, 1996; Perez et al., 1996; Dondi et al., 1997; Silva et al., 1998; Caligaris et al., 2000; Crespo and Rincón, 2001; Piscicella et al., 2001; Pereira et al., 2004; Monteiro et al., 2004). Even if this is done in small amounts, the high production rates of such ceramic products translate into significant consumption of waste materials. Moreover, given the reasonably high firing temperatures generally used ( $>1000$  °C), an effective incorporation of the material into the ceramic matrix is accomplished, which is particularly interesting when dealing with the inertization of hazardous wastes.

What has been overlooked too often is the fact that some wastes are similar in composition to the natural raw materials used nowadays, and often contain materials that are not only compatible but also helpful in the fabrication of ceramics (Santos, 1997; Thomas and Peer, 2001). Within this scenario, upgrading industrial wastes to alternative raw materials becomes interesting, both technically and economically, for a wide range of applications.

Raw materials for the ceramic industry can be one of three major kinds, given the distinctive roles they play during processing: plastic components (e.g. clays), fluxing components (e.g. feldspars) and inert components (e.g. quartz sand). Aimed at a possible use as alternative raw materials, wastes have been classed (Dondi et al., 1997) as fuel wastes, which contain high level of organic or carbon-rich substances (high calorific added-value); fluxing wastes, which promote better sinterability of the ceramic body (glassy phase former); and plasticity-controlling wastes, which affect mostly green body preparation (shrinkage controller, both during drying and firing). Waste materials that might be easy to up-grade to alternative raw materials for the ceramic industry are those that are inert, non-dangerous, and produced in high quantities. Each waste material will have a dominant effect on the fabrication process but can also contribute to the roles played by the other raw materials.

Among such waste materials, marble and granite rejects produced in the cutting process of ornamental stones (e.g. marble and granite) are becoming a worrying factor for industry owners and environmentalists alike, due to the growing amount of rejected mud that is continuously discarded into rivers and lagoons, likely leading to their necrosis. Marble is a crystalline metamorphic limestone, basically containing calcite ( $\text{CaCO}_3$ ) and, maybe, dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ), and granite is an igneous rock, with feldspar, quartz and mica as major components. As a rule, marble and granite rejects show non-plastic behaviour and, like the great majority of traditional ceramic products, their major constituents, expressed as oxides, are silica ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ), followed by lime ( $\text{CaO}$ ) and alkaline oxides ( $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ ). Iron oxide contents can be significant, due to the sawing process, and will promote a dark colouring of the final ceramic

products, but this oxide should not play such an important role during ceramic processing, in air, at low temperatures.

The objective of this work is to study the prospective use of marble and granite rejects as additives and their effect on the sintering/densification behaviour and mechanical properties of a clay-based ceramic material, seeking a lower temperature sintering material and a means to ameliorate and minimize the cutting mud negative impact on the environment.

## 2. Experimental procedure

A typical clay mixture used in industry (where it is fired at 1150 °C for 90 min) and a dry marble and granite reject, not beneficiated in any way (used as collected from the stone-cutting industry) were selected as raw materials and characterized. The characterization included chemical composition (X-ray fluorescence (XRF), Philips PW 1400), mineralogical composition (X-ray diffraction (XRD), Rigaku Geigerflex D/max-Series), thermal behaviour (dilatometry, differential thermal analysis (DTA) and thermogravimetric analysis (TGA), LINSEIS STA, 10 °C/min up to 1150 °C), and particle size distribution (Coulter LS 230). Selected mixtures containing 0, 10, 20 and 30 wt.% of reject were prepared and homogenized for 4 h in a planetary mill with alumina grinding balls. The powders were uniaxially pressed into test bars ( $50 \times 4 \times 4 \text{ mm}^3$ ) under a load of 20 MPa. Powder compacts were sintered at temperatures between 1100 and 1150 °C for 2 h, with a heating rate of 500 °C/h. The heating rate and the sintering time used in this work were so chosen to simulate the actual sintering process used in the ceramic industry.

The mechanical strength of the sintered samples (average of five specimens for each value) was measured with a universal testing machine (Shimadzu AutoGraph-25TA) in three-point bending tests at a constant cross-head speed of 0.5 mm/min. Apparent density and water absorption were determined by using the Archimedes water displacement method, as specified by the ISO 10545-3 Standard, and the crystalline phases present after sintering were identified by X-ray diffraction. Fracture surfaces were observed by

Scanning Electron Microscopy (Hitachi S-4100, at 25 kV, after carbon coating) and qualitative EDS was used to identify the various phases observed.

## 3. Results and discussion

Table 1 gives the chemical composition of the clay mixture and the marble and granite reject. The clay mixture shows the expected typical composition: rich in silica and alumina (minor contents of Mg, Ti, Ca, Na and K oxides), accompanied by a significant amount of iron oxide, which will be responsible for a dark colouring of the fired pieces. The loss on ignition (6.38%) is within the usual range for red-clay mixtures, and is most likely associated with volatile components, organic matter burn-off and/or carbonate decomposition (Santos, 1997). In terms of oxides, the reject material consists basically of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and CaO, with minor contents of MgO, Fe<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O and Na<sub>2</sub>O. The loss on ignition (LoI) is slightly higher than what would be expected if all CaO present resulted from the decomposition of calcite, suggesting that dolomite might also be present. In terms of chemical composition, the major differences between the two materials (hence, those that might induce some changes in the clay mixture behaviour, upon the reject additions) lie in the higher alkaline-earth oxide content (particularly CaO) and the LoI value.

Fig. 1 shows the X-ray diffraction patterns of the clay mixture and the reject. It can be seen that the clay mixture contains quartz and kaolinite, and minor amounts of mica and iron oxide (Fe<sub>3</sub>O<sub>4</sub> and/or hematite, whose major peaks lie in the same  $2\theta$  range). The reject also contains quartz, together with potash feldspar, dolomite, calcite, plagioclase and mica. The crystalline phases identified by XRD are in agreement with the results obtained by XRF (Table 1).

Fig. 2 shows the particle size distribution of the clay mixture and the marble and granite reject. The

Table 1  
Chemical composition of the raw materials, as determined by XRF (wt.%)

	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	MgO	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO	LoI
Clay	19.59	63.11	6.11	0.23	0.26	1.69	1.44	0.73	0.17	0.05	6.38
Reject	12.62	47.93	2.97	12.58	2.27	2.33	4.90	0.45	0.27	0.05	13.11

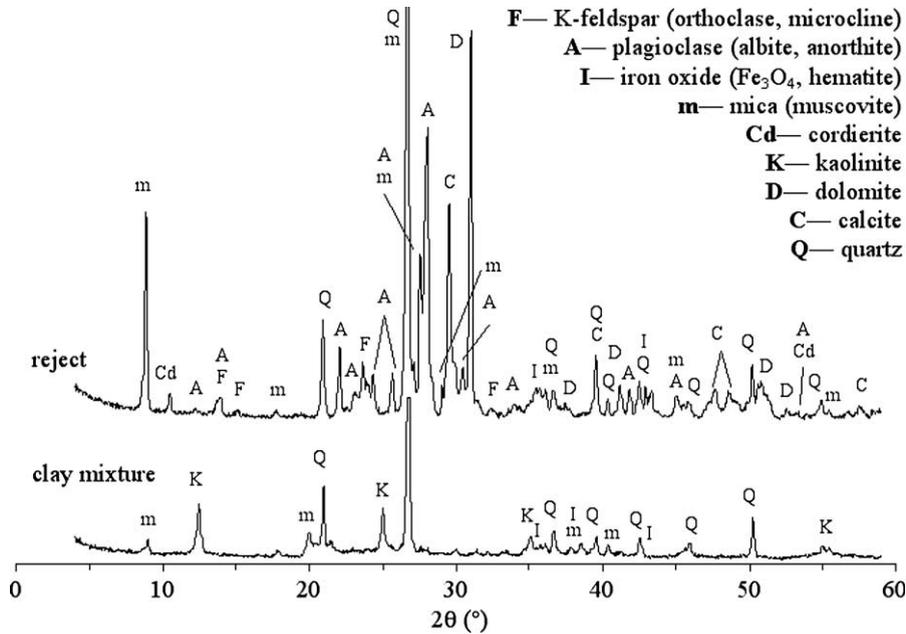


Fig. 1. X-ray diffraction patterns of the raw materials.

distributions are rather similar and the major feature to note is the generally larger particle sizes in the reject (average particle size of 23  $\mu\text{m}$ , whereas in the clay mixture the average particle size is 11  $\mu\text{m}$ ). Although green processing was not the objective of this work, one must bear in mind that differences in particle size distribution and the non-plastic character of the reject, particularly for high reject contents, might introduce some difficulties in the shaping process. Although any mixture of particles of different sizes should reach a higher packing density than that of the individual components (Zok and Lange, 1991), the non-plastic character of the reject might give rise to lower green density values. On the other hand, drying shrinkage

might be improved (reduced), although dried bodies might loose some mechanical strength.

Fig. 3A shows the thermal behaviour of the two materials. The thermogravimetry (TG) of the reject shows a small smooth weight loss from 40 to 600  $^{\circ}\text{C}$  (adsorbed water and other volatile materials), followed by a sharp weight loss ( $\sim 10\%$ ) that can be attributed to the carbonates decomposition. Endothermic peaks in the DTA curve (respectively at  $\sim 100$  and  $\sim 900$   $^{\circ}\text{C}$ ) correspond to both weight losses. The DTA curve for the reject also shows an exothermic peak at  $\sim 970$   $^{\circ}\text{C}$ , suggesting the formation of crystalline phases (probably more anorthite, given that mullite is not a compatible phase for this composition). Compared to those of the reject, the clay DTA and TG curves show a much larger adsorbed and chemically bonded water loss and there are no obvious signs of carbonates. There is a clear exothermic peak at  $\sim 1000$   $^{\circ}\text{C}$  (i.e. in the same temperature range as the exothermic peak observed for the reject), which might correspond to the formation of crystalline mullite.

Fig. 3B depicts the results obtained from the dilatometric study carried out. The clay mixture shows the characteristic  $\alpha$ -to- $\beta$  quartz inversion at 573  $^{\circ}\text{C}$  and the typical clay behaviour (Santos, 1997),

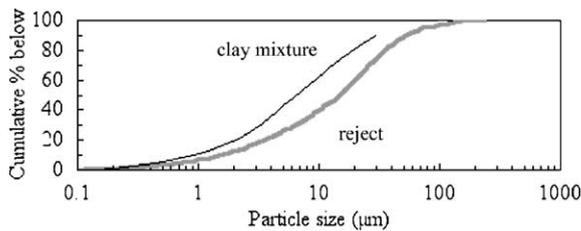


Fig. 2. Particle size distribution of the raw materials.

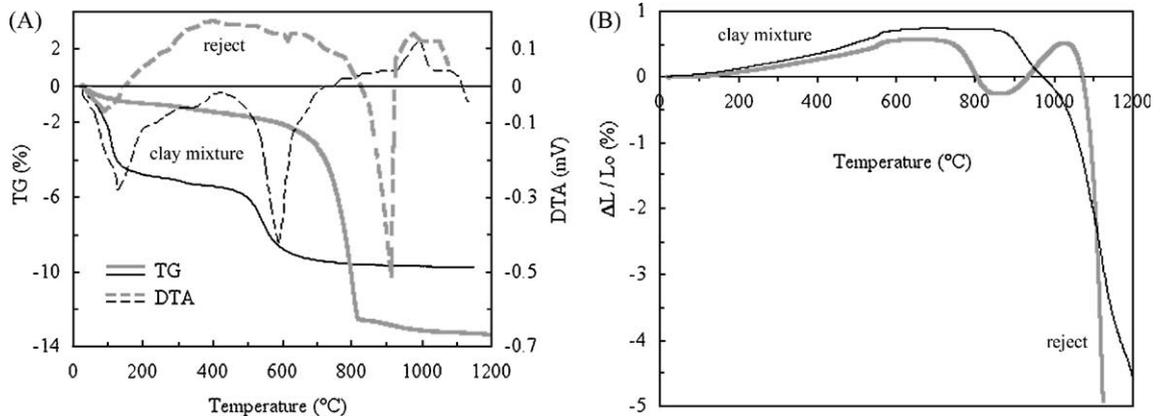


Fig. 3. Thermal behaviour of the raw materials: (A) DTA and TG, and (B) dilatometry.

characterised by uniform expansion up to  $\sim 900$  °C, followed by a strong shrinkage. On the other hand, the reject, that also shows the characteristic  $\alpha$ -to- $\beta$  quartz expansion, shows a strong shrinkage at  $\sim 800$  °C, which is likely due to a microstructural rearrangement occurring during the carbonate decomposition, with a large loss of carbon dioxide, and the simultaneous formation of a liquid phase, due to the presence of the alkaline oxides (Table 1), as predicted by the phase equilibrium diagram and discussed later. However, upon the decomposition of the carbonates, the expansion resumes at  $\sim 900$  °C, as the resulting CaO

reacts with the other oxides present, to form the new crystalline phases already detected in the thermal analysis (Fig. 2). This is most likely related to the expansive formation of anorthite. Similar behaviour was reported in the literature for clay materials with high calcium oxide content (Assal et al., 1999; Thomas and Peer, 2001), whereas clay materials containing low CaO content do not show any expansion (Neves, 2002).

Fig. 4 illustrates the differences between the X-ray diffraction patterns obtained for the sintered samples. Upon sintering at  $1140$  °C, both the clay mixture and

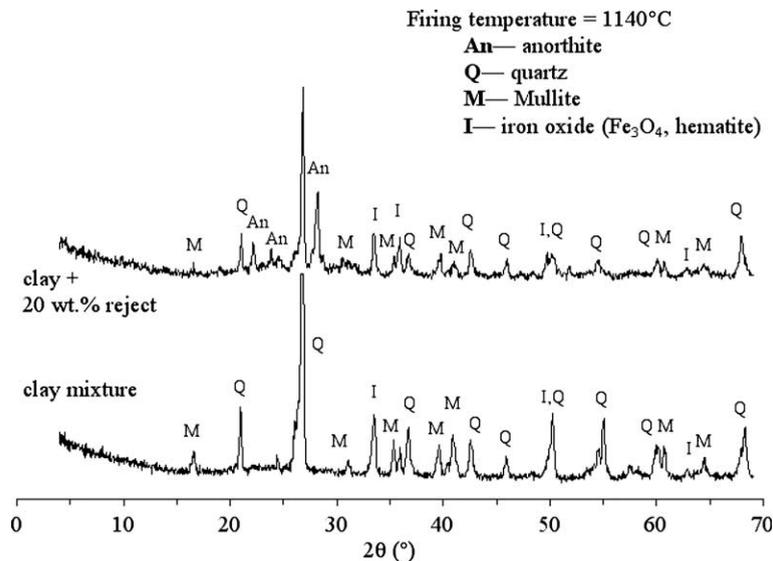


Fig. 4. Typical X-ray diffraction patterns of samples sintered at  $1140$  °C.

the reject-added compositions contain quartz and iron oxide. The presence of mullite is the contribution of the clay mixture whereas the presence of anorthite is the contribution of the reject, as suggested by the corresponding DTA (Fig. 3A) and dilatometry curves (Fig. 3B). In comparison with the fired clay mixture, the anorthite content of the fired reject-added compositions increases at the expense of the mullite content, as will be discussed later.

Fig. 5 illustrates the fracture surface of sintered samples, as observed by SEM. The sintered clay mixture still displays large quartz grains (strong peak in the XRD in Fig. 4) and a significant amount of porosity, even at the highest firing temperature investigated. On the contrary, the samples containing reject additions show clear signs of vitrification and a higher degree of closed porosity.

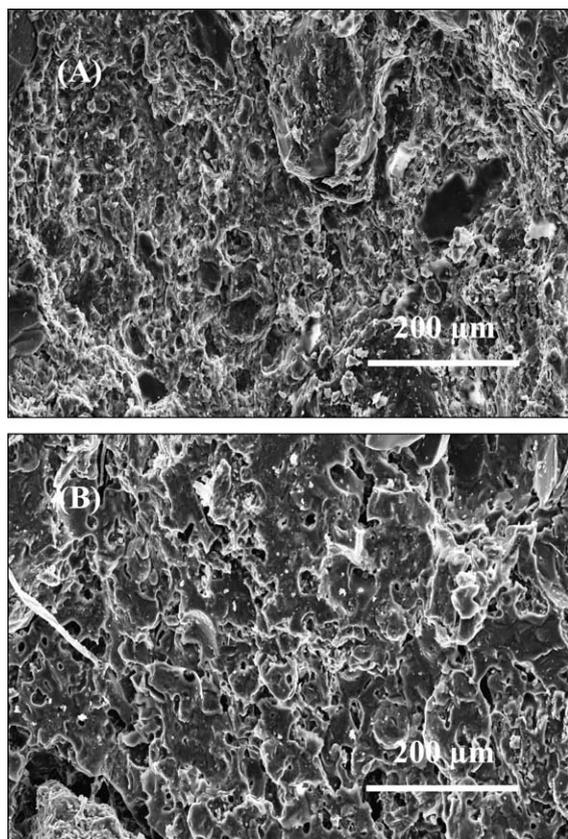


Fig. 5. SEM micrographs of the fracture surfaces of samples sintered at 1140 °C: (A) clay mixture; and (B) clay mixture+20 wt.% reject.

The differences that are observed in the properties of the sintered samples, upon reject addition, are mostly due to changes in the initial melting temperature and the amount of liquid phase present at the sintering temperature, together with changes in the crystalline phases present. Table 1 shows that mixtures of the two materials, specially those with high reject contents, will show significant composition differences, namely higher alkaline and alkaline-earth oxide content (particularly CaO) and lower iron oxide content. Also, during firing, the high LoI of the reject (Table 1, Fig. 2) might not be entirely overcome by the densification process and increased porosity might result. The fluxing effect of the third oxide on the alumina+silica compositions can be best understood using the phase equilibrium diagram of the relevant system. In the real system, with all the minor components present, the dominant oxides effects will remain, but always occur at lower temperatures.

The effect of iron oxides, in air, can be discussed using the  $\text{Al}_2\text{O}_3\text{--SiO}_2\text{--Fe}_3\text{O}_4$  phase diagram. Fig. 6 shows the relevant compatibility triangle (silica–mullite–spinel), containing both the clay mixture and the reject compositions (hence, all the mixtures thereof). In both, the first-melting tie-triangle (shaded area in Fig. 6) is also the same and melting begins at ~1400 °C. Additions of reject to the clay mixture will actually reduce the amount of both the liquid phase and mullite formed at that temperature (calculated using the lever rule).

The effect of the alkaline oxides ( $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$ ) can be discussed using the  $\text{Al}_2\text{O}_3\text{--SiO}_2\text{--K}_2\text{O}$  phase diagram (rather similar systems,  $\text{K}_2\text{O}$  being the strongest flux). Again, both the clay mixture and the reject compositions lie in the same compatibility triangle (silica–mullite–potash feldspar), in which melting begins at 985 °C, as shown in Fig. 7, and share the same first-melting tie-triangle (shaded area in Fig. 7). The higher alkaline oxide contents brought in by the reject additions translate into higher amounts of liquid phase formed during initial melting but the amount of mullite decreases.

As mentioned earlier, the CaO content will be the major change in the mixture chemical composition. Fig. 8 shows the relevant part of the  $\text{Al}_2\text{O}_3\text{--SiO}_2\text{--CaO}$  phase diagram and the position of the clay mixture and the reject compositions in terms of their

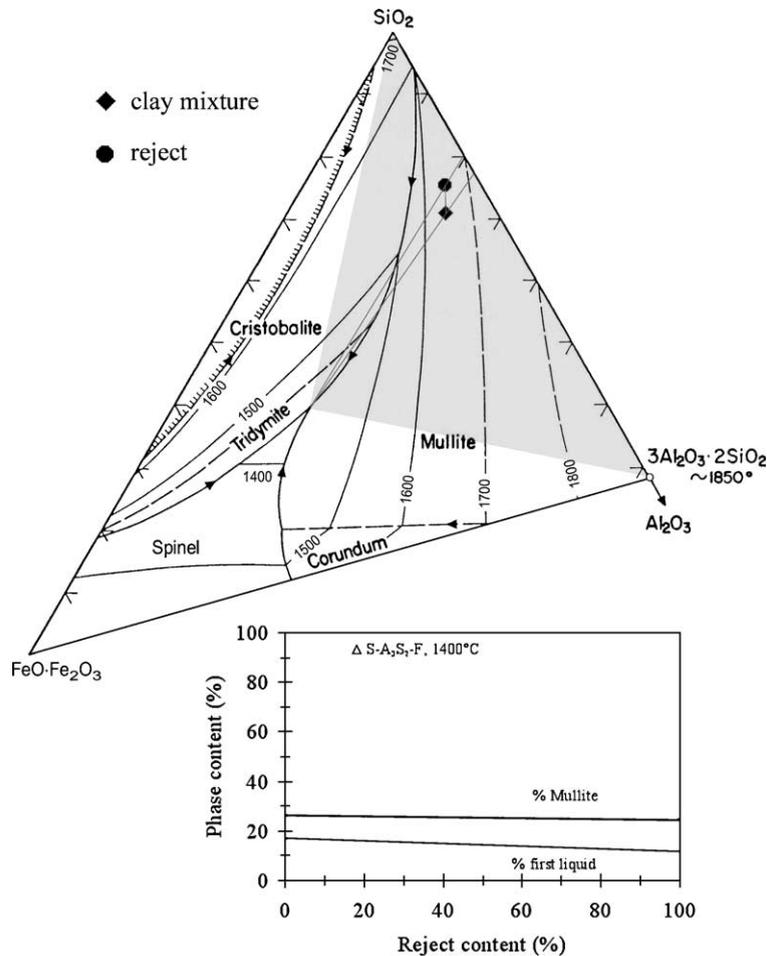


Fig. 6. Silica–mullite–spinel compatibility triangle in the  $\text{Al}_2\text{O}_3$ – $\text{SiO}_2$ – $\text{Fe}_2\text{O}_3$  system (in air) containing both the clay mixture and the reject compositions, showing the effect of reject additions on the amount of phases present at the initial melting temperature (shaded area is the first-melting tie-triangle, used to calculate the amount of phases by the lever rule).

$\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$  and  $\text{CaO}$  contents (black marks). To begin with, the clay mixture and the reject lie in different compatibility triangles with different initial melting temperatures, namely, silica–mullite–anorthite at  $1345^\circ\text{C}$ , and silica–anorthite–wollastonite at  $1170^\circ\text{C}$ , respectively. Reject additions above  $\sim 62$  wt.% would be needed to take the mixture composition across to the lower melting compatibility triangle, where mullite is replaced by wollastonite. This does not give much credit to the fluxing effect of  $\text{CaO}$ , unless the synergetic effect of the presence of the other fluxing oxides is taken into account. When the compositions are represented in terms of their “equivalent  $\text{CaO}$ ” content ( $\text{CaO} + \text{MgO} + \text{Fe}_2\text{O}_3 +$

$\text{Na}_2\text{O} + \text{K}_2\text{O}$ ), their location in the diagram changes (grey marks). The clay mixture and the reject still lie in the same different compatibility triangles, but reject additions of  $\sim 9$  wt.% suffice for the mixture composition to cross over to the lower melting compatibility triangle. The amount of anorthite is maximum in this mixture and decreases henceforth. For reject contents above  $\sim 9$  wt.%, melting begins at  $1170^\circ\text{C}$  and the amount of first liquid steadily increases (up to reject contents of  $\sim 70$  wt.%).

It is expected that the properties of sintered samples that depend on the presence of a liquid phase, follow the trend depicted in Fig. 8. Also, given the differences in density between mullite and

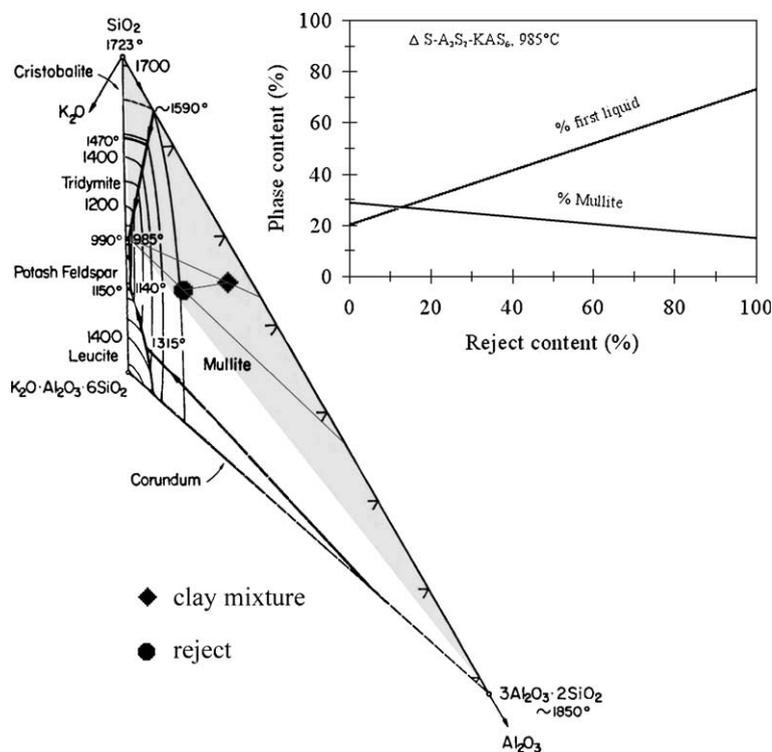


Fig. 7. Silica–mullite–potash feldspar compatibility triangle in the  $\text{Al}_2\text{O}_3$ – $\text{SiO}_2$ – $\text{K}_2\text{O}$  system containing both the clay mixture and the reject compositions, showing the effect of reject additions on the amount of phases present at the initial melting temperature (shaded area is the first-melting tie-triangle, used to calculate the amount of phases by the lever rule).

anorthite ( $3.06$  and  $\sim 2.73 \text{ g cm}^{-3}$ , respectively), the detrimental effect of the expansive formation of anorthite is expected to be mostly noticeable in compositions containing  $\sim 9 \text{ wt.}\%$  reject. When comparing the experimental values, one must bear in mind that the starting green body apparent density was not necessarily kept constant. As mentioned before, the non-plastic character of the reject might give rise to lower green density values for the reject-added compositions, given that a constant compaction pressure was used throughout the work.

Fig. 9 shows the experimentally observed changes in such properties, as a function of the reject content and the sintering temperature. As expected, at each firing temperature, the reject acts as a fluxing agent during firing (promoting the formation and development of a liquid phase), increasing firing shrinkage and reducing open porosity (hence, water absorption). This effect is particularly noticeable upon the first reject additions ( $10 \text{ wt.}\%$ ), becoming less

intense afterwards, as suggested by Fig. 8. Although equilibrium was not fully established during firing, as shown by the presence of small mullite peaks in the X-ray diffraction patterns of the reject-added compositions (Fig. 4), the formation of liquid at a much lower temperature seems to be the dominant factor. When the reject content is higher than  $\sim 9 \text{ wt.}\%$ , both linear shrinkage and water absorption tend to remain unchanged, for all temperatures investigated. At any given reject content, the temperature effect on linear shrinkage and water absorption follows the trend observed for the clay mixture alone.

On the contrary, apparent density values tend to steadily decrease with increasing sintering temperature, even for the clay mixture alone, and also with increasing reject content. In terms of increasing reject content, if the green density does decrease due to the reject additions, mixtures with higher reject contents are expected to reach lower fired densities too, at any given firing temperature. But the

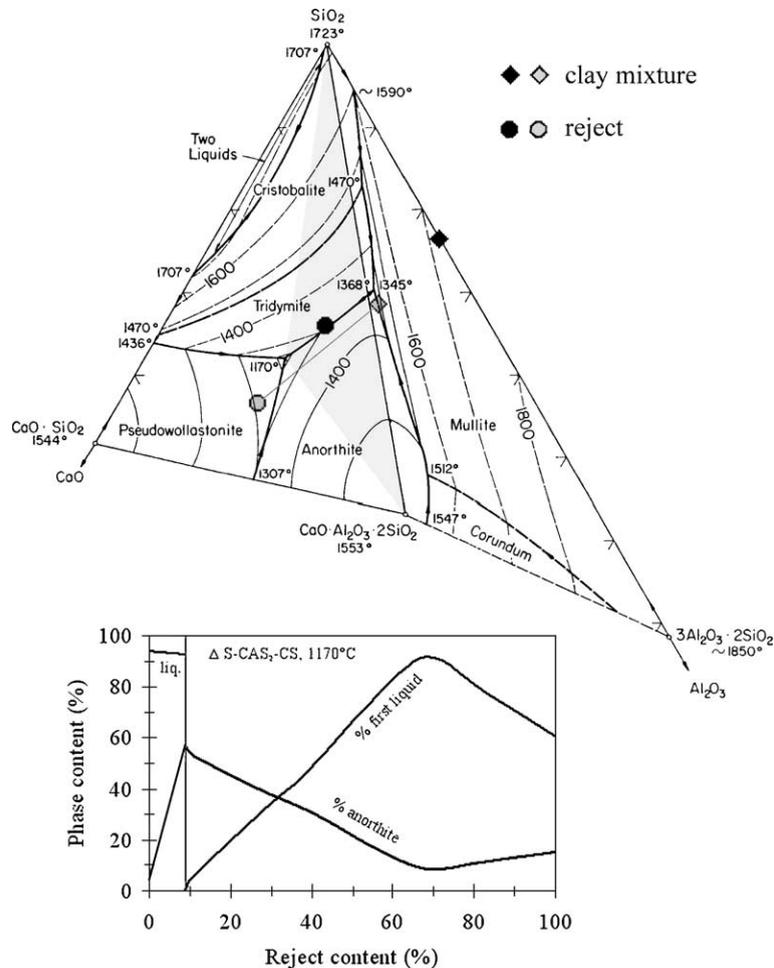


Fig. 8. Compatibility triangles in the  $\text{Al}_2\text{O}_3$ – $\text{SiO}_2$ – $\text{CaO}$  system containing the clay mixture (silica–mullite–anorthite) and the reject (silica–mullite–wollastonite) compositions, showing the effect of reject additions on initial melting temperature and the amount of phases present at that temperature (shaded area is the first-melting tie-triangle, used to calculate the amount of phases by the lever rule). Grey marks represent the location of compositions in terms of their “equivalent CaO” (i.e.  $\text{CaO} + \text{MgO} + \text{Fe}_2\text{O}_3 + \text{Na}_2\text{O} + \text{K}_2\text{O}$ ) content.

clear trend observed in Fig. 9 suggests the effect of a stronger variable. Given that open porosity generally decreases with the first reject addition and remains roughly constant afterwards (water absorption in Fig. 9), decreasing density can be the combined result of the replacement of a solid phase (mullite) by another of lower density (anorthite), as suggested by Fig. 8, and/or of a higher percentage of closed porosity. The onset of melting is expected to occur within the same temperature range as the decomposition of the carbonates (900–1000 °C), and gas evolution during the early sintering stages is higher in mixtures with higher reject contents. At the lower firing temper-

atures (1100 and 1125 °C), when the less abundant liquid phase has a weaker counteracting effect and equilibrium is more difficult to reach, gas evolution will mostly translate into open porosity, as seen in the slightly increasing water absorption values in Fig. 9. As the firing temperature increases, the liquid becomes more abundant but, despite the liquid pull towards densification, some existing pores might get sealed (balance between the liquid capillary drag and the gas pressure inside the pores) (Assal et al., 1999).

As for the flexural strength of sintered samples, Fig. 9 clearly shows that low additions of reject (again

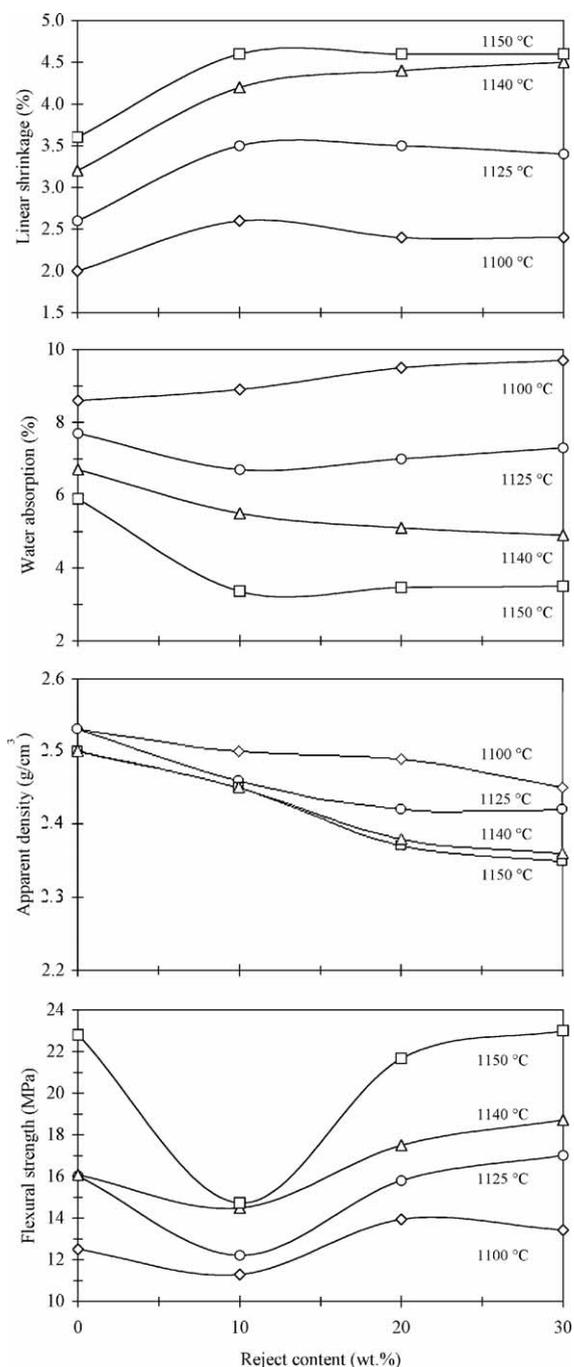


Fig. 9. Changes in the most relevant properties (linear shrinkage, water absorption, apparent density and flexural strength) of sintered samples, as a function of reject content and sintering temperature.

~10 wt.%) are the most detrimental. The fluxing effect of the reject, for the short firing times used, might be overruled by gas evolution and the expansive formation of anorthite (maximum at ~9 wt.% reject, predicted by the phase diagram), regardless of the firing temperature. Higher reject additions lead to more abundant liquid (up to ~70 wt.% reject, as predicted by the phase diagram), which will mostly remain in the sintered body as a bonding glassy phase. Hence, the flexural strength improves, again for all firing temperatures investigated.

Considering that, in the industry using the particular clay mixture studied in this work, firing is carried out at 1150 °C, these results show that comparable mechanical properties and lower water absorption can be obtained, at the same temperature, with the addition of ~30 wt.% reject (i.e. an upgrade from class IIb to class IIa of ISO 13006 standard, at the cost of a slightly higher firing shrinkage). Moreover, with the addition of ~30 wt.% reject, firing can be carried out at significantly lower temperatures, with small sacrifice of firing shrinkage, open porosity and flexural strength (i.e. the final product remains within the original class IIb). Despite the increased consumption of raw materials brought about by the higher firing shrinkage, it must be reminded that the reject is a waste product (nominally, zero cost) and the lower firing temperature will, undoubtedly, translate into significant energy savings.

#### 4. Conclusions

The results obtained in this work show that high contents (~30 wt.%) of a particular non-beneficiated, fine grained and low iron, marble and granite reject can be added to an industrial clay mixture, already in use in the production of floor tiles, not only with no major sacrifice of the properties of the final product (which remains within the original class IIb or might even be up-graded to class IIa of ISO 13006 standard), but also, and more importantly, with the possibility of using lower firing temperatures. A lower firing temperature will, undoubtedly, translate into significant energy savings, and the large use of the marble and granite reject into a much welcome relief on waste disposal concerns.

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