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Recovering inorganic wastes

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Abstract

The aim of this work is to recover wastes by uniaxial pressing and injection moulding. A previous morphological, chemical and mineralogical identifications of different waste disposal batches guaranteed its reproducibility and revealed that in some cases they should be suitable to be treated by those conformation processes. Different tests regarding the chemical and structural evolution with temperature were performed with uniaxial pressed samples, which revealed that in all the cases there are formation of mullite and glassy phases. The presence of these phases contributes to a good physical and chemical behaviour of the fired parts. Moreover, the tests performed in a plastograph equipment to evaluate the rheological characteristics of different mixtures of waste powders with a commercial binder (function of temperature and applied load) show a good performance of these feedstocks to be injected. The typical thickness of injected products were less than 4.8 mm. After injection moulding or uniaxial pressing followed by sintering the final products present density and porosity values close to the theoretical ones. The Weibull modulus obtained with waste products is in the range of those of technical ceramics. These wastes have chemical and structural characteristics after firing that are interesting to the manufacturing of different ceramic products.

Keywords: Inorganic wastes; Uniaxial pressing; Powder injection moulding; Recovery

1. Introduction

All around the world, millions of tons of inorganic wastes are produced everyday in rock quarries and industries, in cutting and polishing rocks for building applications. In order to get profit with these wastes, it was intended to use them as raw material to manufacturing ceramic products. This is advantageous due to the reproducibility of chemical composition and particle size distribution of these wastes. Thus, it could be a good alternative because it will conduct to a significantly decrease of waste storage. Two different process of conformation was selected, uniaxial pressing and injection moulding.

Uniaxial pressing followed by sintering can be used for manufacturing parts with simple geometry [1]. However, to produce great series of components with complex design, injection moulding is the appropriate technology. In this process the ceramic powder is blended with a binder in order to obtain a homogeneous mixture, which allows it to be injected into a mould and to produce the desired shape and dimensions of the part. Subsequently, the binder vehicle is removed, usually by slow heating, before sintering [2,3].

The optimal ceramic content in the feedstock and a suitable mixing technique are the key to achieve adequate rheological properties for injection moulding and homogeneous mixtures, which originate the best properties after injection, debinding and sintering processes [2–6].

In order to get high solids loading without a dramatic increase in viscosity the powders must have spherical and small particles with a multimodal particle size distribution [3,7]. The powder used in this study presents particles with a lamellar morphology and a mean diameter of 8.5 μm. These characteristics are not the most suitable for injection moulding. However, using slate wastes as material, it was shown that the optimisation of slate wastes content in mixture and feedstock preparation are crucial to overcame the problem concerning the high value of mean particle size [8]. Depending of the mixing technique there are slate/binder mixtures, which present adequate rheological properties for injection moulding, even with high content of solids [9]. In fact, as showed in these studies [8,9], mixtures with slate contents 50 and 53 vol.% optimised by the loading curve or torque rheometry, did not originate products with quality, after debinding and sintering; on the contrary, a mixture of 60 vol.%

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showed a good performance during the injection moulding process and after the heating treatments.

In the present work it will be compared the performance of both conformation process as function of end products properties, maintaining the sintering temperature.

2. Materials and experimental details

2.1. Materials

The powder used in this study were slate wastes (surface area 7.3 $m^2\,g^{-1}$ and density 2900 $kg\,m^{-3}$), with a chemical composition of SiO_2 = 54%, Al_2O_3 = 24%, FeO = 8–10%, other oxides = 9% and a combustion loss = 5%. The mineralogical composition was evaluated by X-ray diffraction (*Philips* diffractometer model Xpert) and is constituted by chlorite, mica quartz and small quantities of accessories minerals. The powder particle size distribution, evaluated with a Coulter LS 130 equipment, is represented in Fig. 1, being the mean particle diameter (d_{50}) 8.5 μm. The powder of slate reflects the lamellar cleavage of the bulk rock and present particles with morphology in lamella shown in SEM microphotograph (Fig. 2). The binder used was an industrial polymeric mixture (Hostamont TP EK 583, *Höechst*).

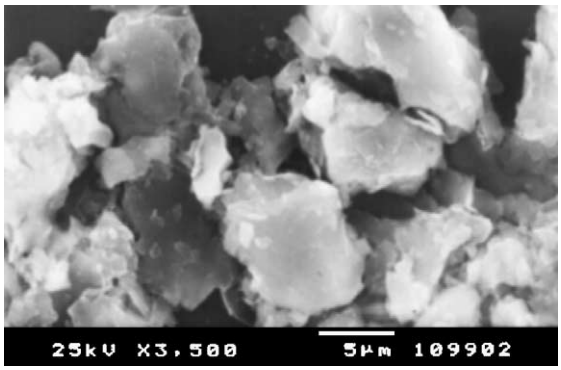

Fig. 2. SEM microphotograph of slate wastes particles.

2.2. Experimental details

2.2.1. Techniques

The diagram corresponding to the uniaxial pressing and powder injection moulding processes is represented in Fig. 3.

(a) Uniaxial pressing

The slate wastes as aspirated powder was uniaxial pressed in a metallic mould with dimensions of 100 mm × 50 mm × 5 mm at 40 MPa. The samples were sintered in static atmosphere with heating rate of 10 °C min^{-1} and a holding time at 1170 °C of 30 min followed by cooling at room temperature in furnace.

(b) Powder injection moulding

A slate/binder mixture 60:40 vol.% was processed using a continuous mixer (*Farrel Continuous Mixer* CP 23). In this technique the mixture was premixed in a twin kneader (140 °C and 140 rpm) and after was extruded at 60 rpm with a constant barrel temperature of 150 °C, in a continuous process. After, the extruded mixture was granulated.

Disks with 3.8 mm of thickness were produced by injection moulding the granulated material, using a injection machine ARBURG model All Rounder 220/150 E. After injection the disks were cutted in parallelepiped bars (67 mm × 6 mm × 3.8 mm). The injection parameters are presented in Table 1.

A chamber furnace was used to heat at least 10 parallelepiped bars in dynamic air, in order to remove the binder. The debinding cycle was a continuous heating at 0.5 °C min^{-1} from room temperature to 700 °C. Subsequently the bars were cooled in the furnace to room

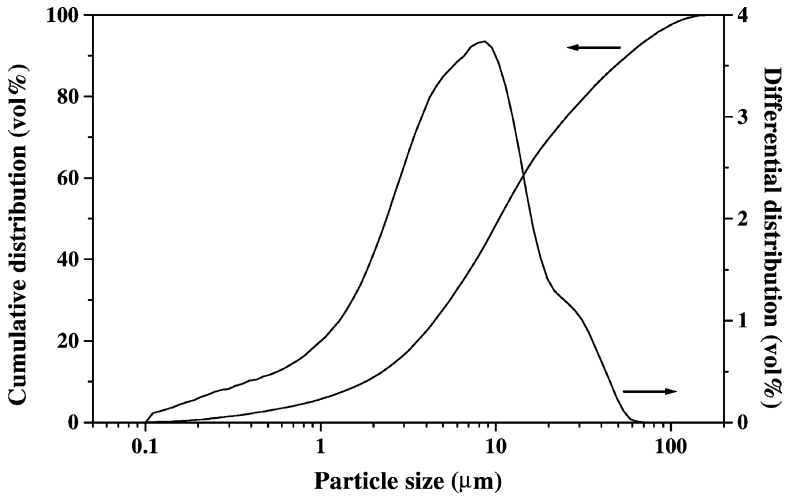

Fig. 1. Slate wastes particle size distribution.

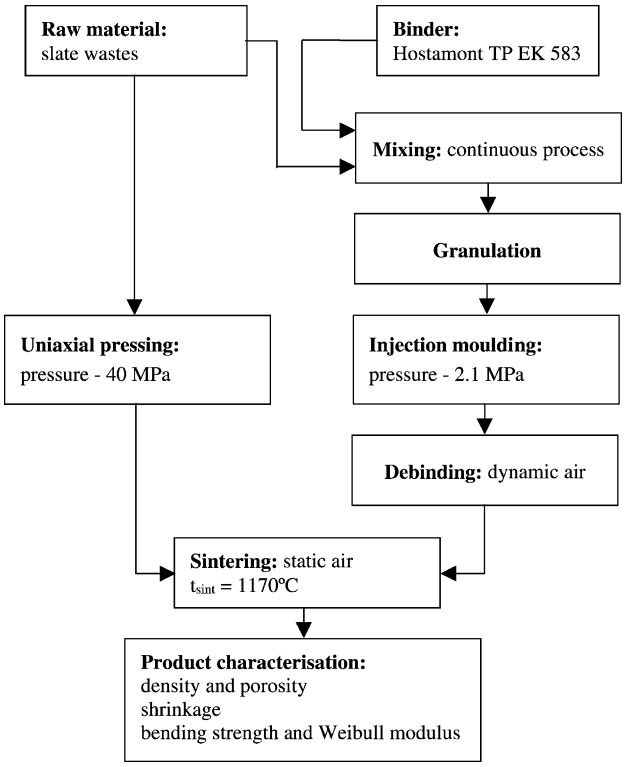

Fig. 3. Diagram of uniaxial pressing and injection moulding processes.

temperature. After debinding, the parallelepiped bars were sintered in static air. The heating rate was 10 °C min^{-1} to 600 °C and after 1 °C min^{-1} to 1170 °C with a holding time at this temperature of 30 min followed by cooling in the furnace to room temperature.

2.2.2. Product characterisation

The true and apparent densities of some sintered samples were measured in a helium picnometer (*Micromeritics* Accupyc 1330) and by water immersion using a sealant (Archimedes method), respectively. The porosity of some samples was also measured by mercury porosimetry (*Micromeritics* Poresizer 9320).

The dimensional variations of slate parallelepiped bars, before and after sintering, were measured, in order to calculate the shrinkage value.

Table 1
Injection parameters

	Mixture 60:40 vol.%
Mould temperature (°C)	40
Barrel temperature (°C)	100/140
Nozzle temperature (°C)	150
Injection pressure (MPa)	2.1
Plasticating pressure (MPa)	0.7
Injection speed	2.5
Screw speed (rpm)	160
Cooling time (s)	60
Holding time (s)	5

Table 2
Density of the products

	Uniaxial pressed	Injection-moulded
True density (g cm^{-3})	2.60	2.60
Apparent density (g cm^{-3})	2.56	2.60

A *Gabbrieli* CRAB 424 testing machine was used to test at least 10 sintered bars, in order to estimate the quality of pressed and injected products, respectively.

3. Results and discussion

3.1. Density and porosity

Table 2 illustrates the different densities of slate products according to the manufacturing process. The comparison of apparent density and true density of the sintered samples shows the presence of pores in pressed slates. However, samples densify easily for densities up to close of the true density.

The porosity of some samples after sintering was evaluated by mercury porosimetry. The equipment allows the determination of pores with dimensions between 360 μm and 6 nm. In the present study, due to the small pore size, it was not possible to quantify the open porosity. In fact, from Table 2 it can be concluded that for pressed products there is some difference between the true and the apparent densities that could be due to close porosity.

3.2. Shrinkage

Other important parameter for the processing of ceramics is the dimensional variations of samples before and after sintering associated to sintering efficiency. Besides the shrinkage due to sintering, two kinds of shrinkage can be present after the sintering process—one resulting of the ceramic powder irreversible phase transformations and another due to the lost of the binder (the last one in the case of injection-moulded products). Table 3 shows the dimensional variations in length (l), width (w) and thickness (t) of pressed and injection-moulded bars after sintering.

The low packing density of slate particles, due to its lamellar shape, induces a high shrinkage value in thickness direction in both conformation processes. The shrinkage value in the other dimensions is similar, for each of the conformation technique. However, higher values were determined

Table 3
Shrinkage measurements in parallelepiped bars after sintering

Dimension	Uniaxial pressed	Injection-moulded
Length ± standard deviation (%)	10.2 ± 0.1	13.8 ± 0.8
Width ± standard deviation (%)	10.2 ± 0.2	12.0 ± 1.2
Thickness ± standard deviation (%)	15.8 ± 0.9	14.7 ± 1.3

Table 4
Flexural strength and Weibull modulus

Physical characteristic	Uniaxial pressed	Injection-moulded
Bending strength (MPa)	92	95
Weibull modulus	25	12

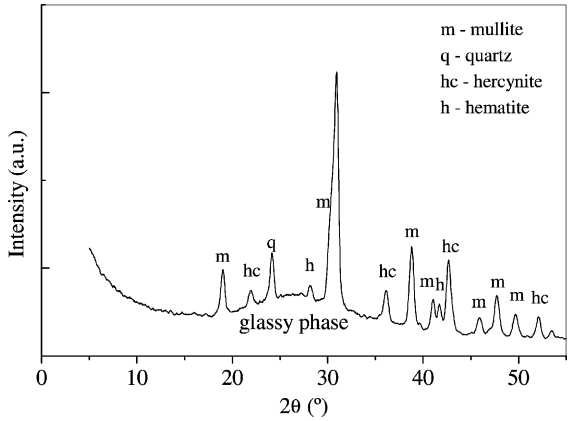

Fig. 4. X-ray difractogram of slate wastes after sintering.

for the injection-moulded samples due to the presence of binder.

3.3. Bending strength

Table 4 shows the bending strength of pressed and injection-moulded parallelepiped bars and the Weibull modulus (*m*). It can be concluded that the conformation process does not influence significatively the bending strength. The Weibull modulus should be highlighted, particularly for pressed samples, especially when considering the Weibull modulus of 10, which is the goal for what called advanced ceramics [10,11]. The lower value obtained for the injection-moulded samples reveals the presence of more defects.

The low porosity and the values of bending strength can be related to the phase transformations that occurs in slate wastes with heat treatments, which give rise to: glassy phase and mullite (Fig. 4). In fact, the glassy phase that fill into the pores available between the granules of the material allows a good mechanical performance.

4. Conclusions

Taking into account the experimental results it can be affirmed that both conformation processes induce a final product with a good quality. All of the evaluated properties reveal that the new material have a wide range of applications and are highly competitive with most traditional ceramics presently on the market.

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