

Microwave sintering of cordierite precursor green bodies prepared by starch consolidation

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Abstract

This paper reports on a study of the microwave sintering behavior of green disks prepared by the starch consolidation forming method to produce cordierite-based porous materials. Green disks were formed by thermogelling the aqueous suspensions of talc, kaolin and alumina (29.6 vol.%) and potato starch (11.5 vol.%) at 75 and 85 °C for 4 h, drying and calcining. They were characterized by bulk density and apparent porosity measurements, and SEM. Microwave sintering was carried out at 1300 and 1330 °C for 15, 20 and 25 min, applying 50 °C/min. For purposes of comparison, an analysis of green disks prepared and calcined in the same conditions and conventionally sintered (1330 °C for 4 h) was also made. The materials were characterized by XRD, bulk density and apparent porosity measurements, and microstructurally analyzed SEM. The results were analyzed considering the behavior of starch in aqueous suspension at varying temperatures, and the experimental conditions of consolidation and sintering.

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1. Introduction

Porous ceramic materials are potential candidates for a broad spectrum of technological applications, e.g., catalyst supports, filters, gas sensors, combustion burners, and thermal insulators, among others [1,2]. Several new processing methods have been developed in recent years to meet the high demand of these materials with controlled porous microstructures [1–4].

Research today focuses increasingly on the study of methods that cause low environmental impacts. These methods include a new family of forming techniques (direct consolidation methods) to consolidate ceramic suspensions in nonporous molds (e.g., metal molds) without compaction or removal of water. Thus, a novel non-contaminating low-cost technique was developed, based on the gelling ability of starch aqueous suspension at temperatures of 55–85 °C, for use in the manufacture of porous ceramics. Starch acts as a consolida-

tor/binder of ceramic particles and a pore former after consolidation by burn-out at high temperature [5–8]. During the gelatinization process, starch granules undergo rapid and irreversible swelling by water absorption [9], causing the ceramic particles to stick together and consolidate into a solid body. After applying heat treatments (burning and sintering), a porous material is obtained whose microstructure is dictated by the physical characteristics of starch granules (morphology and size), the amount of added starch and its behavior in warm water (i.e., starch swelling capacity, size and shape of swollen granules) [8,9]. This method has been successfully employed to produce porous bodies of alumina [10], cordierite [5], and mullite [11], among others. Cordierite ($2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2 \cdot 2\text{MgO}$) is a potential candidate for use in thermal insulators, among other applications, because of its low thermal expansion coefficient ($\alpha = 1\text{--}3 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$) and thermal conductivity (1–2 W/mK) [12,13].

The removal of organic components by high-temperature burnout involves long heat treatments (200–700 °C) which must be carried out at very slow heating rates in order to favor the release of high amounts of gases, thus allowing for the

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development of materials with the fewest possible defects. The use of microwave energy to processing materials is a relatively recent development that has been gaining increasing importance in the field of materials research. Its main benefits are faster heating schedules and energy savings compared to conventional heating methods as those found in food processing [14–19]. Microwaves transfer energy directly into the material, where it is converted into heat through the interaction of atoms and molecules with the electromagnetic field, in ionic conduction, dipole relaxation, and photon–phonon processes [14–16,19]. Thus, microwaves provide uniform volumetric heating of materials, allowing for the application of high heating rates and considerably shorter processing times, eliminating numerous difficulties encountered in conventional rapid heating techniques [15,16]. In addition to volumetric heating, other features of microwave processing are the formation of reverse temperature gradients and heat flows compared to conventional heating processes [17]. The direct deposition of energy within the processed material reduces the consumption of energy when compared with refractory furnaces. Therefore, microwave processing offers the advantages of high heating rates, shorter processing times, and significantly lower energy consumption, particularly when compared to high-temperature processes, which involve enormous heat loss with increasing temperature [18]. An important feature that must be considered when microwave-heating a material is porosity, which is the main property of the cordierite under study here. Pore characteristics include the level of porosity, type of porosity, and pore distribution and morphology. Evidence of the interaction of microwaves with pores is clearly visible from the significant increase in loss tangent with increasing porosity in the material. However, it is difficult to quantitatively correlate the increase in loss tangent with porosity, since ceramic bodies may be constituted by other phases and have defects, besides pores [19].

This work involved a study of the microwave sintering of cordierite precursor green disks prepared by thermogelling of aqueous ceramic suspensions with potato starch at different temperatures to produce cordierite-based porous materials. The developed microstructures were analyzed in relation to behavior of the aqueous starch suspensions with temperature and experimental conditions of consolidation and sintering used. Moreover, the results were also compared with those obtained by the conventional sintering route.

2. Experimental

2.1. Characterization of the raw materials

A mixture of commercial kaolin (kaolin C-80, Stone Big CORP., Arg.), talc (Talc 40, China) and alumina (A2G Alcoa, USA) powders with particle sizes of $<5\ \mu\text{m}$, was used as a cordierite precursor. Based on a qualitative X ray-diffraction analysis (Philips PW3710, Cu $K\alpha$ radiation at 20 mA and 40 kW) of the raw materials, kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$, File 06-0221) was identified as the main crystalline phase, with traces of quartz (SiO_2 , File 5-0490) and orthoclase (KAlSi_3O_8 , File

31-0966) in the kaolin powder. The main mineral phase in the talc was $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ (File 19-0770), with traces of dolomite ($\text{CaMg}(\text{CO}_3)_2$, File 34-0517) and magnesite (MgCO_3 , File 83-1461), while the only phase identified in the alumina powder was corundum ($\alpha\text{-Al}_2\text{O}_3$, File 42-1468). The cordierite precursor mixture was formulated based on its oxide compositions, which resembled that of stoichiometric cordierite ($\text{SiO}_2 = 51.4\ \text{wt.}\%$; $\text{Al}_2\text{O}_3 = 34.8\ \text{wt.}\%$ and $\text{MgO} = 13.8\ \text{wt.}\%$), but with lower silica content and higher proportions of alumina and magnesia: 37 wt.% of kaolin, 41 wt.% of talc and 22 wt.% of alumina. Based on the global composition, 85.7 wt.% of cordierite and 14.3 wt.% of $\text{Al}_2\text{O}_3\text{-MgO}$ spinel were estimated as the final phases in thermodynamic equilibrium.

Native potato starch commercially available in Argentina was used as a consolidator/binder and a pore forming agent. The real density ($1.47\ \text{g/cm}^3$) was determined by He-pycnometry (Quanta-Chrome, USA). Particle size distribution (Malvern Instruments Ltd., UK) was determined from an aqueous suspension of starch containing a polyacrylic acid (Dolapix CE-64, Zschimmer & Schwarz, Germany) as dispersant and applying ultrasound for 15 min to disperse and stabilize the starch particles. The potato starch presented a bimodal distribution, with a low volume percentage of small granules possibly representing impurities or broken granules, a slightly larger mean particle diameter ($D_{50} = 47.8\ \mu\text{m}$) than the mean diameters of ceramic particles, and a high distribution width ($W = D_{90} - D_{10}/D_{50} = 1.3$; where D_{90} and D_{10} are the granule diameters for 90 and 10 vol.% of granules, respectively). The weight percentage of humidity (14.4 wt.%) was determined by thermogravimetric analysis (Shimatzu, TGA-50) at $10\ ^\circ\text{C}/\text{min}$ up to $120\ ^\circ\text{C}$, in air. An analysis of the morphology of the dry starch by scanning electron microscopy (Jeol JSM-6460) showed granules with smooth surfaces and oval or spherical shapes. The pycnometric density ($\delta_{\text{pic}}^{\text{p}}$) of the precursor mixture determined by pycnometry in kerosene at $37\ ^\circ\text{C}$ was $2.44 \pm 0.06\ \text{g/cm}^3$. The behavior of aqueous starch suspensions as a function of temperature was studied in a previous work [20].

2.2. Preparation and characterization of green bodies

Green disks (13.0 mm diameter; 1.5–3.0 mm thick) were prepared by thermogelling aqueous suspensions of the cordierite precursor mixture (29.6 vol.%) and 11.5 vol.% of potato starch. The suspensions were prepared by: (a) mixing (impeller mixer) the ceramic powders in water (70.4 vol.%) with 1 wt.% of Dolapix CE-64 (Zschimmer & Schwarz, Germany) and 0.5 wt.% of sodium naphthalenesulfonate (both with respect to the ceramic solids content); the ceramic powders were added sequentially, beginning with kaolin, pausing for 24 h, followed by the talc and lastly the alumina; (b) homogenization in a ball mill for 2 h; (c) addition of starch and mixing (impeller mixer) for 3 min; and (d) degassing for 20 min. The suspensions were poured into cylindrical stainless steel molds (which were covered with Teflon tape to reduce water evaporation), heated in air at 75 or $85\ ^\circ\text{C}$ for 4 h

(Memmert, universal oven with forced air circulation) and dried at 50 °C for 12 h. The disks were machined using 600-grit SiC paper to obtain flat and parallel surfaces.

The average green densities (δ_v) were determined by immersion in Hg (Archimedes method) and the disk porosities (% P_v) were calculated as $100 \times (1 - \delta_v/\delta_{pic}^P)$. A microstructural analysis of the green disks obtained by thermogelling potato starch at the two temperatures was carried out by SEM (Jeol JSM-6460).

2.3. Thermal treatments and characterization of final materials

Disks prepared at both consolidation temperatures and dried were calcined in a conventional furnace for 2 h at 650 °C, using a heating rate of 1 °C/min to minimize the formation of cracks during the burnout of starch caused by the high heating rates employed in microwave treatments.

After calcination, the disks were sintered in a microwave furnace (multi-mode cavity) at 2.45 GHz (Cober Electronics, MS6K) using susceptor material as a heating aid. Details of this material have been reported in a previous work [18]. Microwave sintering was carried out at different temperatures (1300, 1325 and 1330 °C) and times (15, 20 and 25 min), at a heating rate of 50 °C/min and a cooling rate of 30 °C/min. For purposes of comparison, an analysis was made of conventionally sintered green disks prepared and calcined in the same conditions. These disks were sintered at 1330 °C for 4 h in an electric furnace equipped with SiC heater elements, applying a heating rate of 3 °C/min, followed by cooling to 25 °C at rate of 5 °C/min [21].

Phase evolution as a function of sintering temperature and time was analyzed by XRD (Siemens, D500, Cu K_α radiation, at 30 mA and 40 kW).

Microwave and conventionally sintered disks were characterized based on bulk density (δ_s) and apparent porosity (% P_s) measurements taken by immersion in water (Archimedes

Table 1
Density (δ_v) and porosity (% P_v) of green disks.

T_c (°C) ^a	δ_v (g/cm ³)	% P_v
75	1.27 ± 0.04	48 ± 1
85	1.20 ± 0.08	51 ± 2

^a T_c , consolidation temperature.

method). Polished surfaces (up to 1 μ m) were analyzed microstructurally by SEM (Jeol JSM 6460). Before polishing, porous samples were vacuum impregnated with epoxy resin and dense samples were embedded in polyester resin. The equivalent diameter, which was calculated as the mean value of the diameters passing through the center of each individual pore measured with a step of 2°, was selected as the parameter to estimate the pore size. These diameters were determined from an analysis of the SEM micrographs using image analysis software (Image-Pro Plus, Media Cybernetics).

3. Results and discussion

3.1. Starch consolidation

3.1.1. Characterization of green disks

The values of density (δ_v) and porosity (% P_v) of the green compacts are shown in Table 1.

The tendency for porosity in the green disks prepared at the two consolidation temperatures can be explained by the fact that these microstructures are influenced by the behavior of starch in aqueous suspensions at different temperatures (swelling capacity and characteristics of the swollen granules). A previous study [20] indicated that native potato starch presented the highest swelling capacity in water in the range of temperatures of the gelatinization process. Thus, a high proportion of swollen granules can be considered at both consolidation temperatures, a proportion that increases at the higher consolidation temperature (85 °C). The presence of a

Table 2
Crystalline phases in microwave sintered (MS) and conventionally sintered (CS) materials.

T_c (°C) ^a	Sintering route	T_s (°C) ^b	t_s (min) ^c	Crystalline phases
75	MS	1300	15	Cordierite, spinel, alumina, n.i. phases
			20	
			25	
		1325	15	Cordierite, spinel, alumina
			20	
			25	
		1330	15	Cordierite, spinel, alumina
			20	
			25	
85	CS	1330	240	Cordierite, spinel, (alumina) ^d
	MS	1300	15	Cordierite, spinel, alumina, n.i. phases
			15	
			15	
	1330	1330	15	Cordierite, spinel, alumina
240				

^a T_c , consolidation temperature.

^b T_s , sintering temperature.

^c t_s , sintering time.

^d (alumina) = alumina in very low amount.

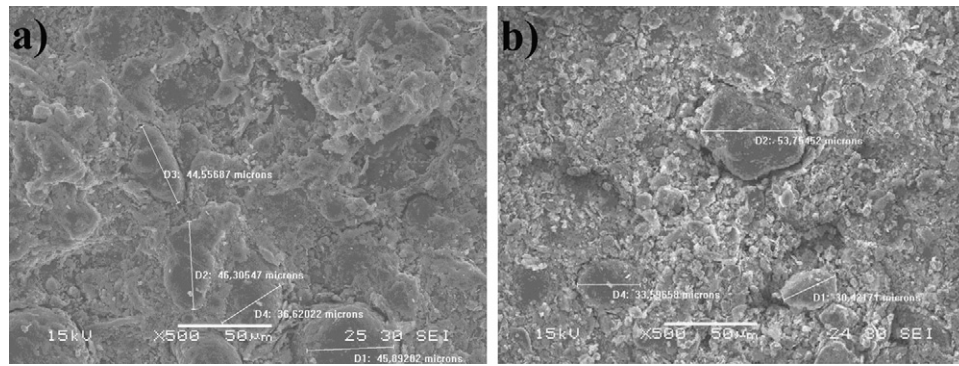


Fig. 1. Green microstructures of disks consolidated for 4 h at (a) 75 °C and (b) 85 °C.

greater proportion of swollen granules at 85 °C with slightly larger sizes than the ceramic particles may explain the slight difference between porosities obtained in green disks [22].

SEM micrographs of the green compacts consolidated at 75 and 85 °C are shown in Fig. 1. Both images show swollen starch granules and original granules that did not swell, which form the gel structure together with leached components from gelatinized granules. These findings were confirmed by an elemental carbon analysis (EDAX) [22]. In both materials, the sizes (between 30 and 60 µm) and morphology of the granules observed by SEM fell within the range of values determined for dry and gelatinized starches [20,22]. The slight differences in porosity were not visible by SEM.

3.2. Microwave sintering

3.2.1. Analysis of the final materials

Table 2 shows the phases (identified by XRD analysis) in the materials prepared by starch consolidation at 75 and 85 °C and microwave sintered in different experimental conditions, together with the results obtained from materials sintered by the conventional route.

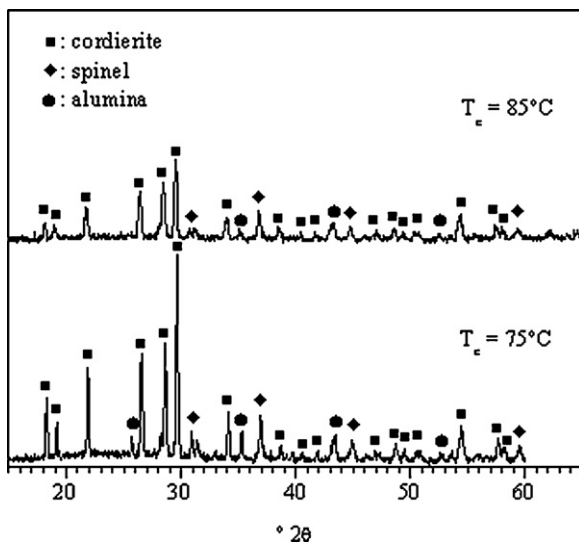


Fig. 2. X-ray diffraction patterns of materials consolidated at 75 and 85 °C, and microwave sintered at 1330 °C for 15 min.

The materials prepared at the two consolidation temperatures and sintered by the conventional route showed cordierite as the main crystalline phase ($\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$, File 13-293), together with spinel ($\text{Al}_2\text{O}_3\cdot\text{MgO}$, File 5-0672) and a very minor amount of alumina as corundum ($\alpha\text{-Al}_2\text{O}_3$, File 42-1468). None of the peaks characteristic of raw materials was identified. It was also observed that the baseline rose in the range 20–30° 2θ, which corresponds to the region of maximum diffraction peaks of silicate crystalline phases. The presence of a glassy silicate phase originating from impurities in the raw materials and from the decomposition of kaolinite was inferred based on this finding. In both cases, low melting point silicates were generated, which reduce the temperature of liquid formation of the $\text{SiO}_2\cdot\text{Al}_2\text{O}_3\cdot\text{MgO}$ ternary system (1350 ± 5 °C) [23].

The materials prepared at 75 °C and microwave sintered at the lowest temperature (1300 °C) for 15, 20 and 25 min, as well as those prepared at 85 °C and sintered at the same temperature for 15 min, showed crystalline phases identified by XRD as cordierite (File 13-293), spinel (File 5-0672) and alumina (as received material) as corundum (File 42-1468). The presence of non-identified crystalline phases was also determined. In the materials prepared at 75 and 85 °C, an increase of the sintering temperature from 1300 to 1330 °C, regardless of sintering time, caused the non-identified phases to disappear, spinel and alumina to decrease, and cordierite to increase, indicating that increasing the sintering temperature favored the reaction. In addition, in the case of materials consolidated at 75 °C, the microwave sintering time at 1325 and 1330 °C did not affect the reaction significantly (Table 2). The presence of a glassy silicate phase was also inferred from the XRD analysis (the baseline rose in the range of 20–30° 2θ) in all the microwave sintered materials.

The materials prepared at 85 °C and sintered at 1325 and 1330 °C for 15 min showed a decrease in the intensity and a slight increase in the width of the diffraction peaks corresponding to the cordierite phase (Fig. 2) indicating the formation of cordierite with a slightly lower degree of crystallinity than that of the sintered materials prepared at 75 °C. This result demonstrates that the cordierite phase crystallinity not only depends on the sintering temperature but also of the consolidation temperature. Therefore, for each consolidation temperature, the green microstructure developed

after calcination influences the crystallinity of the majority phase (cordierite).

Table 3 lists the values of bulk density (δ_s) and apparent porosity (% P_s) of the materials prepared at the two consolidation temperatures and microwave sintered at 1325 and 1330 °C, temperatures that cause a significant reaction advance degree. The results obtained by CS are also given in this table.

After microwave sintering, the samples consolidated with potato starch at 75 °C showed significantly higher porosity than those consolidated with starch at 85 °C, regardless of sintering temperature and time. Samples consolidated at 75 °C and sintered at 1325 and 1330 °C for 15 min showed an apparent porosity higher than 30%, while the same material consolidated at 85 °C and sintered at 1325 and 1330 °C for 15 min was significantly less porous (P_s lower than 8%). Increasing the sintering temperature to 1330 °C favored densification of the samples consolidated at 75 °C, and the highest decrease in porosity occurred in 20 min of sintering. In contrast, the disks sintered by conventional heating did not show changes in porosity (55%) in response to the higher consolidation temperature.

Gelatinized starch granules with different characteristics (i.e., particularly size and morphology) were developed at two consolidation temperatures because of their distinct behavior in aqueous suspensions at these temperatures. Thus, after calcination, a particular pore morphology was developed in the green materials for each one of the consolidation temperatures

Table 3

Density and porosity values of disks processed by microwave (MS) and conventional (CS) sintering.

T_c (°C) ^a	Sintering route	T_s (°C) ^b	t_s (min) ^c	δ_s (g/cm ³)	% P_s	
75	MS	1325	15	1.58 ± 0.03	38	
			20	1.55 ± 0.01	40	
			25	1.91 ± 0.02	25	
		1330	15	1.70 ± 0.3	35	
			20	2.05 ± 0.03	18	
			25	2.05 ± 0.02	17	
85	CS	1330	240	1.20 ± 0.1	55	
		MS	1325	15	2.15 ± 0.05	8
			1330	15	2.20 ± 0.05	2
	CS	1330	240	1.20 ± 0.04	55	

^a T_c , consolidation temperature.

^b T_s , sintering temperature.

^c t_s , sintering time.

(75 and 85 °C). Granules gelatinized at 75 °C increased in size compared to their initial size, presenting an oblong geometry with well-defined borders and little deformation. Granules gelatinized at 85 °C, on the other hand, were slightly larger (they continued to swell at 85 °C), but some showed less perfect edges [20] and an irregular morphology (deformed granules), and some even broke up into smaller fragments. The apparent porosity of the green disks consolidated at both temperatures was fairly similar (Table 1), as was mentioned in Section 3.1. Therefore, the notable difference in the final porosities of sintered materials may have resulted from the

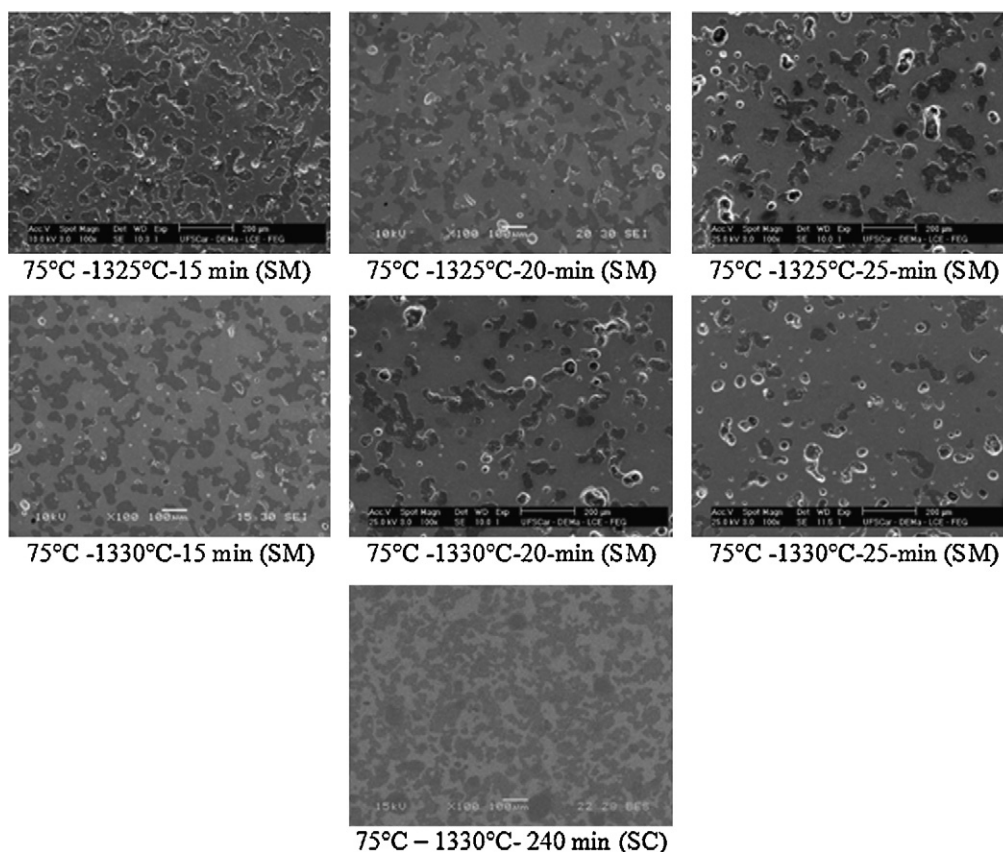


Fig. 3. Microstructures of the samples consolidated at 75 °C and sintered by microwave and conventional routes.

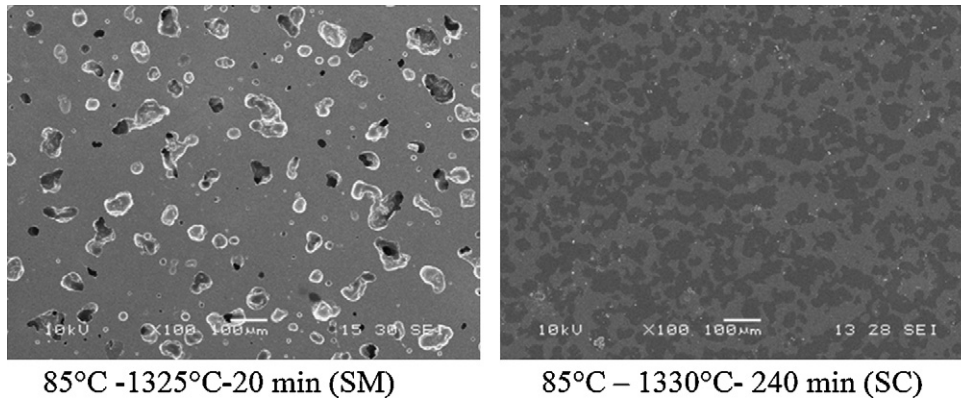


Fig. 4. Microstructures of the samples consolidated at 85 °C and sintered by the microwave and conventional routes.

different characteristics (morphology and sizes) of the pores produced by burning potato granules gelatinized at 75 and 85 °C. Energy absorption is known to vary, depending strongly on the dielectric loss factor of the material to be heated [24], and the dielectric properties are influenced by the material's porosity. Thus, the dielectric inhomogeneity of porous ceramics enhances the electric field on the pores' internal surfaces. Local enhancement of the electric field improves material flux at the surfaces and affects the local driving force for densification [25]. The morphology and fragmentation of granules in the material consolidated at 85 °C probably generated pores whose characteristics favored higher absorption of microwave heat, leading to selective heating that promoted the flow of material and hence the sintering kinetics. In addition, the lower crystallinity of the cordierite phase generated in microwave sintered materials prepared at 85 °C may also have favored the densification process, and consequently a lower porosity.

After sintering, the disks consolidated at 75 °C and microwave sintered at 1325 and 1330 °C at the various times, as well as the conventionally sintered disks, displayed higher levels of porosity than the starch content and a volumetric shrinkage (9% by microwave and 30% by the conventional route). This behavior is consistent with the swelling of some of the granules during the consolidation process prior to sintering.

For cordierite-based materials, the highest porosities (~40%) obtained by microwave sintering (1325 °C, 15 or 20 min) were lower than those obtained by the conventional route (55%). However, the microwave sintering time was significantly shorter than in conventional sintering.

Figs. 3 and 4 show SEM micrographs of final materials developed from green materials consolidated at 75 and 85 °C and processed by microwave and conventional sintering, respectively. The microstructures of green materials consolidated at 75 °C followed by microwave sintering showed considerably higher porosity than those consolidated at 85 °C and also microwave sintered. However, microstructures with the highest porosity were developed in conventionally sintered materials prepared at both consolidation temperatures. The main microstructural feature found in the materials consolidated at 75 °C and microwave sintered, as well as in those consolidated at both temperatures and conventionally sintered, was the presence of a

network of interconnected pores surrounded by a denser ceramic matrix. This rather complex porosity showed cavities highly interconnected by tortuous channels. This type of morphology made it difficult to determine the pore size, except in materials whose pores showed a more spherical morphology. The materials consolidated at 75 °C and sintered by microwave showed diminishing porosity as the sintering temperature increased from 1325 °C to 1330 °C. Moreover, upon increasing the sintering time, these materials showed a strong decrease in channel tortuosity and the presence of isolated pores with average sizes of 48 and 30 µm in materials sintered at 1325 and 1330 °C for 25 min, respectively. These facts were more marked in materials sintered at 1330 °C. In contrast, materials consolidated at 85 °C and sintered by microwave showed little porosity, isolated pores with a more spherical morphology, and average pore size of 35 µm. On the other hand, materials consolidated at 75 and 85 °C, and sintered by the conventional route showed highly tortuous channels (highest degree of interconnection).

4. Conclusions

Porous cordierite-based materials, with potential applications as thermal insulators, were prepared by starch consolidation and microwave sintering. These materials are potential candidates for use as thermal insulators. The resulting microstructure, particularly the volume of porosity and pore characteristics, was strongly influenced by the green microstructure developed after calcination which depended on consolidation temperature. Porous materials were only obtained by microwave sintering, in certain experimental conditions of sintering temperature and time, from green materials consolidated at 75 °C. However, the material obtained by microwave sintering prepared by thermogelling at 85 °C, presented very low porosity, independently of the used sintering temperature. In contrast, the materials obtained by conventional sintering prepared at each one of the consolidation temperatures used did not show different porosities. The porous materials developed from green disks prepared at 75 °C and microwave sintered at 1325 and 1330 °C for 15 min and conventionally sintered reached high levels of porosity, with pores interconnected by channels. The channels in the microwave sintered materials were less tortuous and this

tortuosity decreased in response to increasing sintering temperature. Increasing the sintering temperature of the materials consolidated at 75 °C and 85 °C led to higher densification and favored the evolution of the reaction. Although the pore volume achieved by microwave sintering of materials prepared at 75 °C was lower than that obtained by the conventional route, the use of microwave radiation allowed for a considerable reduction of the time required to promote reaction-sintering and to produce high porosity.

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