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Synthesis of Stoichiometric Cordierite Ceramics from Chemically Modified Kaolin by a Controlled Acid Leaching

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Abstract: Cordierite ceramic still attracts great interest due to its interesting properties for applications in the field of modern engineering. It is one of the few ceramics that undergoes extremely low thermal expansion, less than 0.001% in the wide temperature range 25 – 800°C. This work aims to synthesize stoichiometric cordierite ceramics according the following molecular formula: $Mg_2Al_4Si_5O_{18}$, from a single kaolin ($Al_2Si_2O_5(OH)_4$), that is chemically modified by acid leaching. The chemical modification of the kaolin is carried out in a hydrochloric acid solution (6M), at a temperature of 70°C, under magnetic stirring for 2 hours. These optimized acid leaching conditions increase the Si/Al ratio of kaolin, and thus allow a stoichiometric mixture of cordierite, by adding the precipitated magnesium hydroxide. Thermal transformations of the starting mixture in the temperature range 1100 – 1430°C reveals the appearance of several phases, such as sapphirine and enstatite, before the cordierite becomes predominant at 1250°C. These intermediate phases have higher densities than that of cordierite, and thereby their transformations to cordierite phase are accompanied by swelling, which usually causes cracking in the ceramic. Therefore, the densification of structural ceramics is carried out from stoichiometric cordierite powders calcined at 1250°C by solid phase sintering and from the starting mixture by reaction sintering for comparison of the ceramic properties. The results show that in the first case, the maximum densification reaches 92% at 1325°C and then decreases to 78% at 1430°C, due to the formation of a glass phase, and in the second case, the densification reaches 90% at 1325°C, and at 1350°C its becomes very close to the total densification (~95%). The thermal expansion coefficient a (from RT to 700 °C) decreases with increasing sintering temperature; $\alpha = 3.1x10^{-6}\ °C^{-1}$ at 1275°C, and becomes $<1.7x10^{-6}\ °C^{-1}$ at 1400°C.

Keywords: Cordierite, Kaolin, Leaching, Sintering, Ceramic properties

Introduction

Cordierite ceramics still arouses a lot of interest because of its many very interesting properties in the field of modern engineering (Chowdhury et al., 2007a, 2007b). Its thermal expansion is extremely low (<0.001%), and its mechanical strength is high (>245MPa), which gives it excellent resistance to thermal shocks. Its use temperature exceeds 1200°C, cordierite is often found in many refractory products such as burner tubes and firing supports for ceramic industry furnaces with fast firing cycles (Ponomarev et al., 2023; Guangmao Yan et al., 2024; Cheraitia et al., 2020).

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Cordierite also has low dielectric constants (ρ~5) and dielectric losses (tgδ<10^{-3}) over a wide frequency interval, which allows for applications in the electrical industry, electronics in the production of electrical insulators, of substrates, and electronic components (Tekin et al., 2022). Recently studies have shown that cordierite is promising in high frequency applications (Cao et al., 2025; Synkiewicz et al, 2021), and in heat storage (Deng et al, 2024), (Boutaleb et al, 2024).

The stoichiometric cordierite is well defined by the SiO2-Al2O3-MgO ternary diagram system with the following centesimal chemical composition: 51.36%SiO2.34.86%Al2O3.13.78%MgO, and a SiO2/Al2O3 ratio = 1.47. The conventional method of synthesis of pure cordierite involves reacting at high temperature a mixture of oxides of SiO_2, MgO, and Al_2O_3 corresponding to the chemical composition. These synthetic and pure oxides make it possible to produce a highly densified ceramic at a very high temperature (~1430°C) close to the melting temperature of theoretical cordierite (1470°C) (Kobayashi et al., 2000). Also many attempts at synthesis of cordierite are developed from available and less expensive natural materials, such as kaolin (Kobayashi et al, 2000), talc [16], sepiolite (Zhou et al, 2011), stevensiste (Bejjaoui et al., 2010), and many other materials carrying the constituent elements of cordierite. Often, these starting mixtures must be composed of several ingredients (kaolin, talc, silica, magnesium compounds...) to succeed in a stoichiometric cordierite. The acid leaching of kaolin ($R_{SiO2/Al2O3}$<1,20) is ideally suited to modify $R_{SiO2/Al2O3}$, by hydrolyzing kaolinite ($Al_2O_3.2SiO_2.2H_2O$) into soluble aluminium and amorphous silica (Al-Harahsheh et al., 2023; Peng et al, 2021; Edama et al, 2014). An optimized chemical treatment will synthesize pure cordierite by adding a suitable magnesium compound.

On the other hand, the densification of ceramics remains insufficient due to the presence of impurities which lead to the formation of vitreous phases at high temperatures and thus lower densification. In addition, the crystallization of cordierite in these cases occurs from intermediate phases such as mullite, sapphirine, clinoenstatite, and spinel (Redaoui et al., 2018; Cameruccia et al., 2003; Khattab et al, 2021). These phases have densities much higher than that of cordierite, their transformations into cordierite can generate microcracks in the ceramic, which requires a delicate and complex thermal cycle to achieve a densification without defects. To avoid this handicap, Ogiwara et al developed highly densified ceramics from calcined cordierite powder using pure oxides, but such studies remain few (Ogiwara et al., 2010).

This research studies the possibility of synthesizing stoichiometric cordierite from a single kaolin which is chemically modified by an acid leaching, and to perform a comparison of the ceramic properties of cordierites prepared by reaction sintering from the starting mixture, and those prepared from previously calcined cordierite powders.

Method

Raw Materials

The kaolin used in this study is taken from a deposit located in eastern Algeria in the region of Djebel Debbagh. This kaolin is known for its black coloration which is caused by a manganiferous mineral; todorokite (Mn,Ca) Mn_5O_{11}n4H_2O) (Senoussi et al., 2016; Boulmokh et al., 2007; Debbakh et al, 2020). To increase the clay mineral content and decrease the sand content, the kaolin DD is sieved by liquid process, under 20 microns and the obtained fraction is designated by DD00. The magnesium is prepared in the form of hydroxide ($Mg(OH)_2$) by precipitation from a solution of magnesium chloride and ammonium hydroxide, with commercial reagents.

Controlled Acid Leaching Method

The controlled acid leaching tests are carried out in a flat-bottomed flask, fitted with a thermometer and a refrigerant, which is installed on a heating plate equipped with magnetic agitation. The same conditions are maintained for all experiments; a solid/liquid ratio is 1:5, molarity of 6M HCl, rotation speed of 450rpm, and duration of 6 hours. The leaching is stopped with a large quantity of distilled water, and the solid is washed several times with distilled water until a neutral pH is obtained, then dried at 100°C in an oven to constant weight. Five experiments are conducted at temperatures of 25°C, 50°C, 60°C, 70°C, and 90°C; the fractions of processed DD00 are designated DD25, DD50, DD60, DD70, and DD90 respectively.

Preparation of Cordierite Ceramic Samples

Pellets (ϕ:13x2,5mm) are prepared by hydraulic pressing (50MPa) from the leached kaolin having the optimal SiO_2/Al_2O_3 ratio for obtaining a stoichiometric cordierite, adding the precipitated magnesium hydroxide. The starting mixture is previously crushed by attrition in ethyl alcohol in the presence of zirconia beads (2 to 3mm) during 120mn. Sintering of pellets are performed at different temperatures, from 1100°C to 1430°C to determine the optimal temperature of cordierite synthesis, and from calcined powder which is ground in the same way as previously.

Characterization

The chemical compositions of the raw materials and of the leached kaolin were carried out using a Rigaku ZSX Primus X-ray fluorescence spectrometer. The identification of crystalline phases of sintered pellets was performed by XRD analysis using a Philips X'Pert Pro (CuK = 1,54056Å) diffractometer, with a continuous scan from 5 to 80°. The characterization is continued by the simultaneous thermal analysis DSC-TGA (STA Netszch 409PC luxx), and the dilatometer (Nezscth 409PC), with a heating rate of 10°C/min between room temperature and 1400°C. The microstructural observations of the ceramics were performed by a scanning electron microscope (SEM) of the type JSM-6360LV. The bulk density (dap), water absorption (Abs), and bulk specific gravity (dsp) were determined using a method based on the principle of Archimedes recommended by the American company for testing and determining the specifications of materials ASTM C373 (ASTM Specification C373). The dielectric characteristics are performed on metallized ceramics using an Agilent HP4192A impedance analyser over a frequency range of 100Hz to 8MHz.

Results and Discussion

The Chemical Modifications of the Kaolin DD

Table 1 shows the chemical compositions of treated DD kaolin samples, prepared magnesium hydroxide (HMP), and synthesized cordierite (CR). It is noted that below 50°C, SiO_2/Al_2O_3 remains unchanged, but at higher temperatures this ratio increases sharply, and for >90°C this ration is multiplies by 3, which shows that more than 50% of kaolin is dissolved. It is clear that temperature is a predominant parameter in the leaching of kaolin (Edama et al., 2014; Al-Harahsheh et al., 2023). The halloysite network undergoes leaching through the release of soluble Al^{3+} and insoluble amorphous silica (White et al., 2018).

It is also noted that MnO strongly decreases under the effect of acid action, certainly because of the leaching of the todorokite which results in the reduction of Mn^{4+}, and Mn^{3+} into Mn^{2+} which are perfectly soluble in acidic solutions (Manish Kumar et al., 2019; El Hazek et al., 2006). According to these results, it is deduced that the conditions that allow for having the optimal SiO_2/Al_2O_3 (1,48) for a stoichiometric mixing of cordierite are those of DDT70, with 86.3%DDT70 + 13.7%HMP.

Table 1. Chemical compositions of kaolin samples treated at different temperatures

	DD00	DDT25	DDT50	DDT70	DDT90	HMC	CR
SiO_2	52,52	54,40	54,42	58,57	75,50	0,18	50,90
Al_2O_3	41,61	44,30	44,60	39,56	23,25	0,00	34,36
MnO	3,41	0,14	0,11	0,18	0,02	0,00	0,16
MgO	0,65	0,45	0,21	0,25	0,20	98,15	13,62
K_2O	0,17	0,12	0,08	0,08	0,09	0,06	0,07
Na_2O	0,25	0,05	0,04	0,08	0,05	0,48	0,07
SO_3	0,48	0,15	0,17	0,08	0,13	0,38	0,12
CaO	0,45	0,22	0,16	0,14	0,09	0,15	0,07
Fe_2O_3	0,15	0,11	0,09	0,10	0,10	0,17	0,12
NiO	0,20	0,01	0,01	0,12	0,03	0,00	0,01
Others	<0,10	<0,10	<0,10	<1,00	<0,50	<0,50	<0,50
SiO_2/Al_2O_3	1,26	1,23	1,22	1,48	3,25	----	----

Cordierite Synthesis

The XRD diffractograms performed on ceramics prepared from the optimized starting mixture are reported in Figure 1. At 1100°C, several phases are identified such as sapphirine (00-011-0607), quartzβ (01-089-8946), spinel (01-086-0084), and clionenstatite (01-084-0652). At 1150°C, only sapphirine and cordierite persist with a ratio of 75%:25%. At 1200°C, we detect the appearance of a reflection at 10.60° which may be related to cordierite (96-900-5806), with many reflections that may be related to sapphirine (00-021-0549). At 1250°C, the majority of the reflections can be attributed to cordierite (00-009-0326), or indialite (01-082-1884), with some reflections that reveal the presence of sapphirine (01-071-2398), and the mass ratio becomes 90% :10% respectively. At 1275°C, only cordierite (01-089-1485) is identifiable whereas sapphirine has certainly completely disappeared in favor of cordierite, the presence of cristobalite (01-082-1410) (<1%) is noted. Beyond this temperature, cordierite becomes predominant, but at 1400°C, we notice the presence of a significant glass phase, certainly due to the presence of fluxing elements that are always present in the kaolin.

Simultaneous thermal analysis (DSC-TGA) of the optimized starting mixture shows that dehydroxylization of HMP occurs around 376°C, and that of halloysite around 512°C yielding MgO and $Al_2Si_2O_7$ (metakaolin) respectively (Figure 2). The formation of an exothermic peak at 932°C confirms the interaction between Magnesium oxyde and metakaolin to form new phases like cordierite, sapphirine ($Mg_6Al_6.5Si_1.5O_{20}$), and clinoenstatite ($MgSiO_3$). A second exothermic peak occurs around 1200°C which translates the crystallization of orthorhombic cordierite from the other phases, in accordance with the XRD analysis (Figure 1).

The thermodidimensional analysis of the pressed starting mixture shows many linear shrinkages, (1) of 400-800°C due to dehydroxilation of halloysite, (2) of 800-1000°C accompanying chemical reactions of new compounds, and (3) of 1000-1150°C probably to sintering of these phases (Figure 3). A swelling is remarkable from 1150°C to 1300°C, this thermodidimensional feature can be attributed to the crystallization of cordierite from phases such as sapphirine, and clinoenstatite. These crystalline phases have higher densities (>3g.cm^{-3}) than that of cordierite (2.52g.cm^{-3}), their transformation certainly leads to the increase in the volume of the ceramic. An unsuitable thermal cycle can lead to microcracking in ceramics.

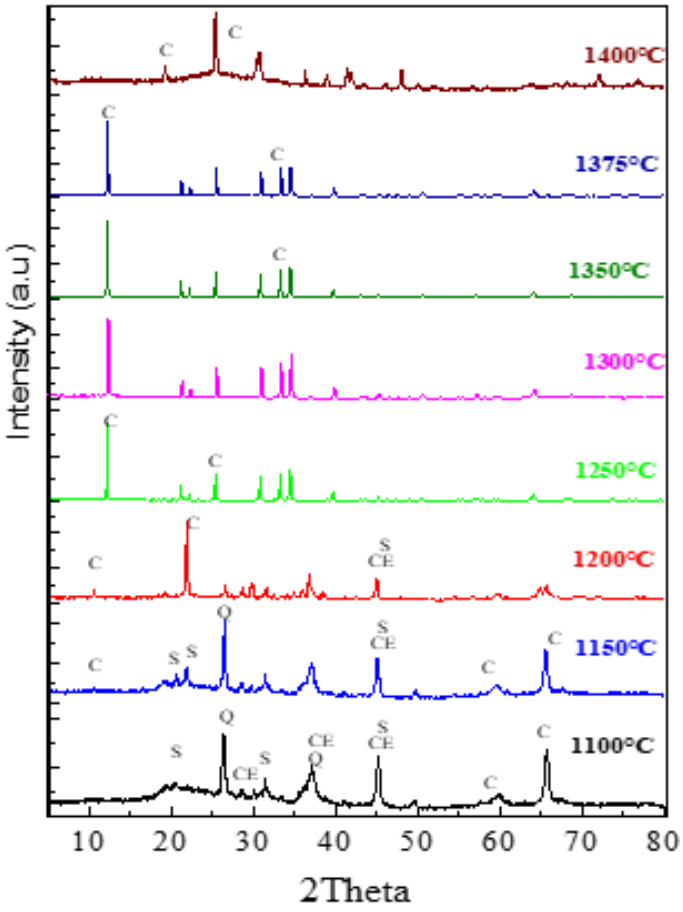

Figure 1. XRD patterns of calcined starting mixture at different temperature
(c) cordierite, (CE) clinoenstatite, (S) sapphirine, (Q) quartz,

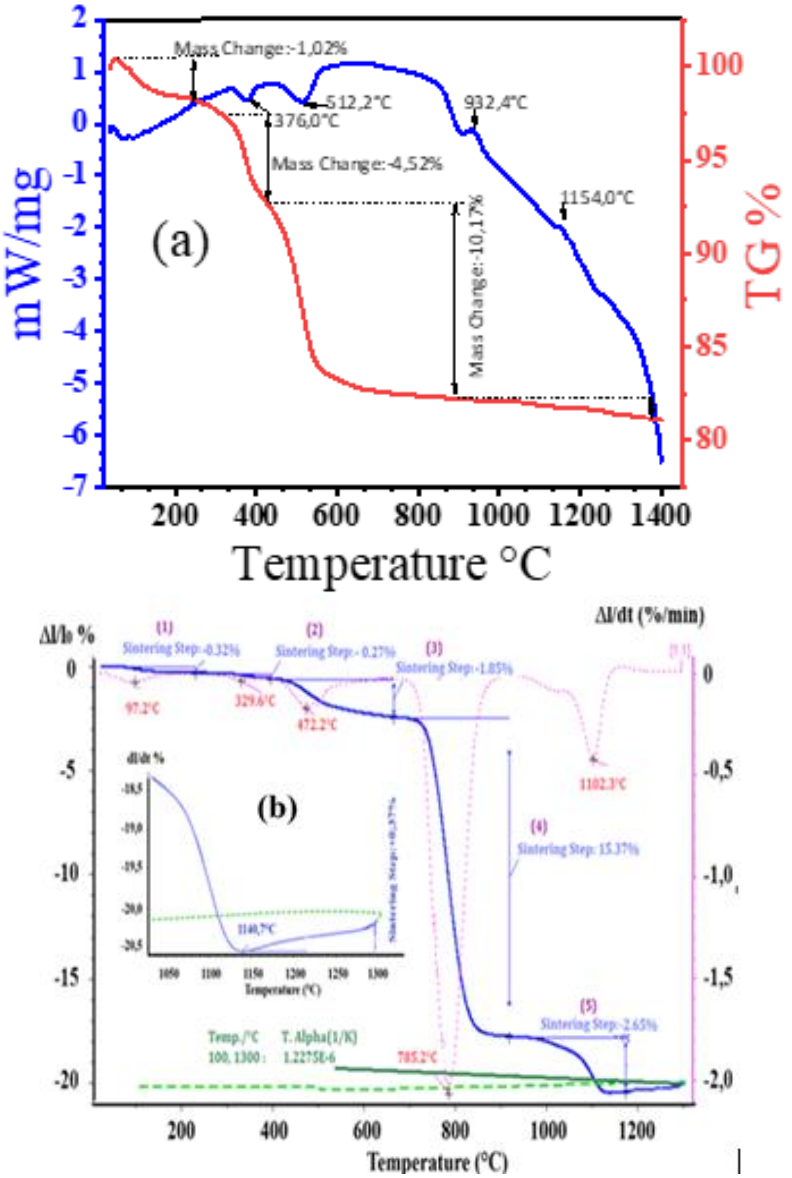

Figure 2. (a) DSC-ATG, and (b) Thermo-dimensional analysis of starting mixture

Sintering

The densification results of the starting mixture and of calcined cordierite powder are presented in figures 3. In the first case, at 1100°C densities (dap, dsp) higher than the theoretical density of cordierite (2.52g/cm3) are observed, this result confirms the results of the DRX analysis (Figure 1). The ceramic is composed mainly of sapphirine, spinel, and clinoenstatite which have higher densities. From 1200 to 1350°C, the bulk density decreases more significantly than the specific density which remains unchanged (~2.50g/cm3), this is the temperature range of cordierite crystallization. From 1400°C, the two densities decrease strongly, and the water absorption increases strongly, and 1425°C dap < 2.00g/cm^3. certainly because of the increase in porosity, and the formation of vitreous phases.

In the case of calcined powder of cordierite (indirect sintering), the bulk density increases with temperature, it reaches a maximum value of 2.35g/cm^3 at 1425°C, with 5% water absorption. While the specific density which is that of prepared cordierite decreases slightly with temperature, certainly because of the glassy phases of impurities.

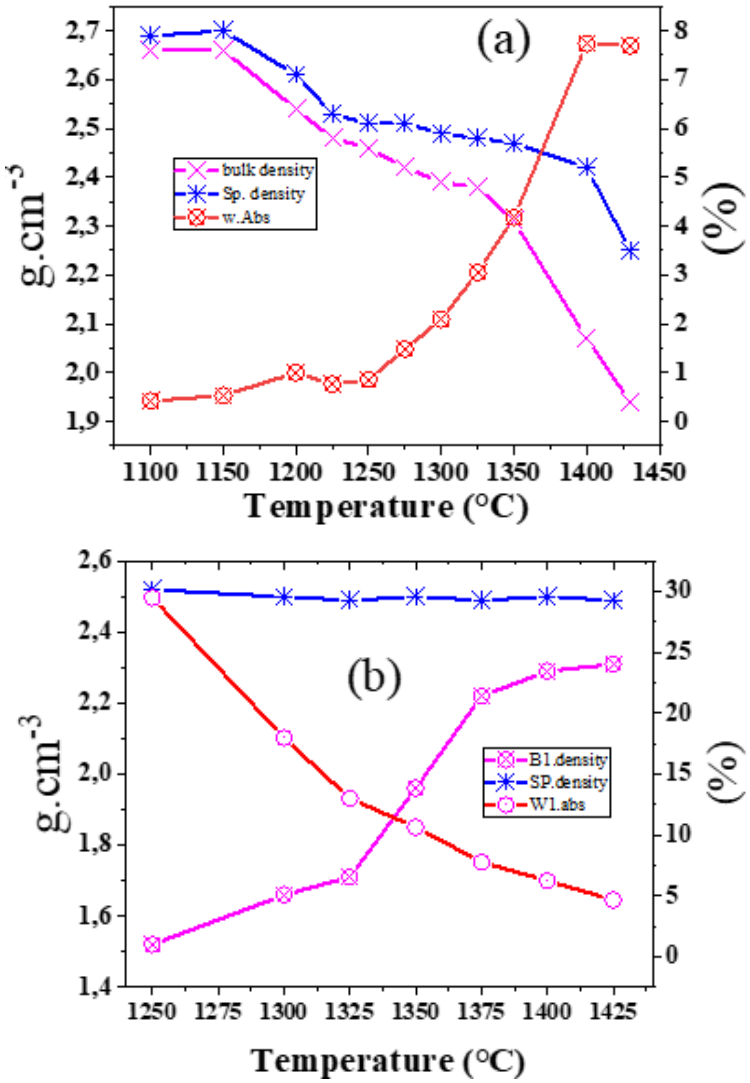

Figure 3. Physical properties of ceramic as a function of sintering temperature
(a) Direct sintering (b) Indirect sintering

Dilatometric Curves of Cordierite

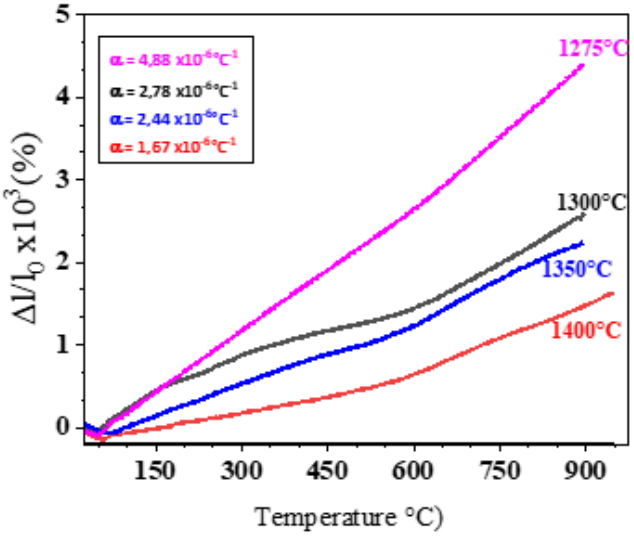

Figure 4. Dilatometric curves of cordierite sintered at different temperatures

Figure 4 shows the expansion curves of sintered cordierite ceramics at increasing temperatures. It is remarkable that the coefficient of linear expansion (TEC) decreases significantly with the increase in sintering temperature. This result seems to confirm that the maturation of cordierite always occurs at a temperature close to fusion of cordierite (~1450°C), (Chowdhury et al., 2007a, 2007b).

Dielectric Properties

The results of dielectric characterizations are reported in Figure 5. The same behaviour is observed in both cases of ceramics prepared by direct sintering (Figure 5a) and those prepared by indirect sintering (Figure 5b). The dielectric constants (ε_r) vary little significantly over a wide frequency interval, while the dielectric losses ($tg\delta$) decrease markedly with frequency. The values of ε_r and $tg\delta$ are consistent with the values of industrial cordierite ceramics 5 and 0,025 at 1MHz respectively.

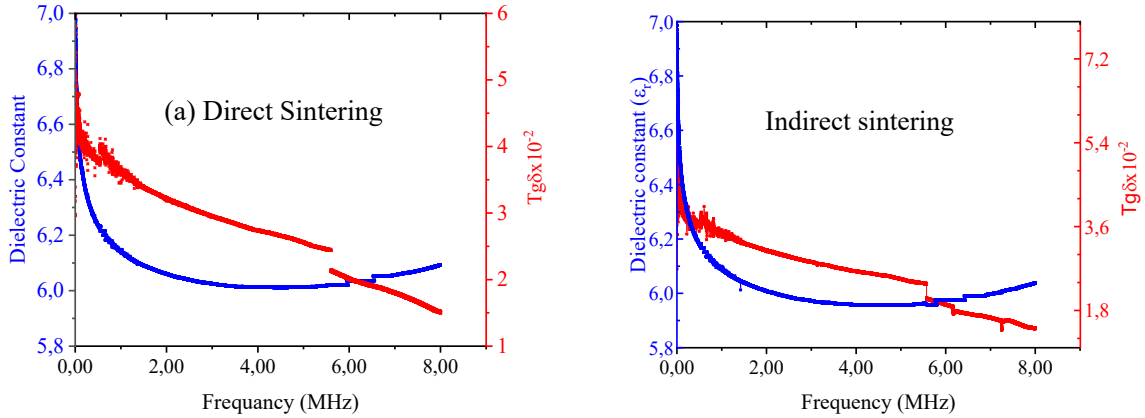

Figure 5. Dielectric constants and losses as a function of frequency (a) direct sintering, (b) indirect sintering

Microstructure

The microstructural observations of sintered ceramics at 1430°C show, in the case of direct sintering (Figure.6a,b), a greater presence of pores than in the case of indirect sintering (Figure.5d,e). In both cases, the vitreous phase is clearly apparent.

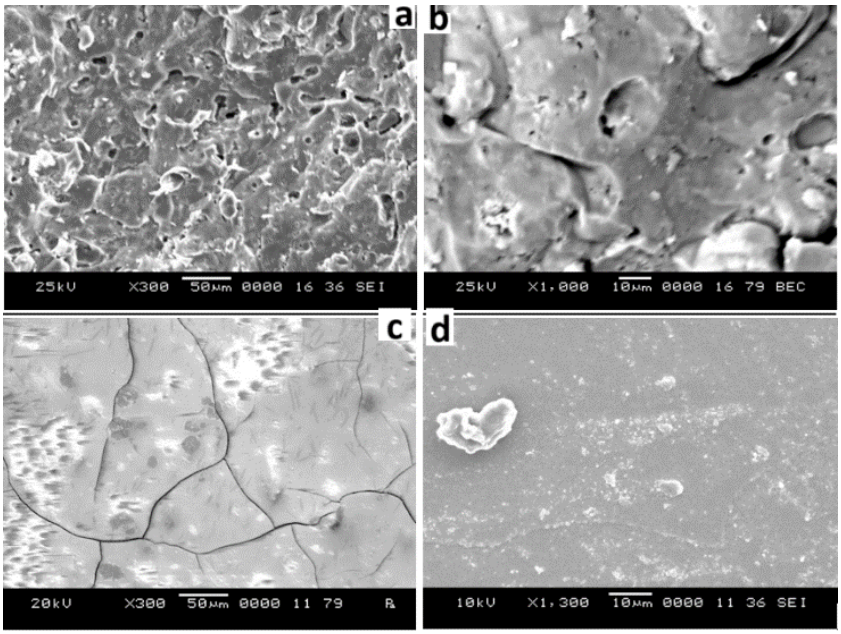

Figure 6. Microstructures of sintered ceramics at 1430°C
(a,b) direct sintering, (c,d) indirect sintering

Table 2 summarizes the physical and dielectric properties of sintered ceramics at 1430°C, obtained according to both methods. It is noted that the reaction sintering method does not allow obtaining highly dense ceramics due to the presence of melting elements contained in the kaolin, whereas the calcined powder sintering method allows for increasing the density of the ceramics to 2,38g.cm^{-3}, which is equivalent to a densification rate of ~95%.

Table 2: Comparison of the ceramic properties of the 2 methods

Properties	Starting mixture Direct sintering	Calcined mixture Indirect sintering
Sintering temperature (°C)	1430	1430
Bulk density (g/cm^3)	2,00	2,38
Specific density	2,15	2,50
Water absorption	2,28	5,37
Open porosity	4,92	11,46
Thermal expansion coefficient (TEC) x10^{-6}°C^{-1} (RT to 1000°C)	1,67	1,72
Dielectric constant (1MHz)	6,06	6,09
Loss tangent (1MHz)	0,032	0,034
Dielectric constant (8MHz)	6,09	6,04
Loss tangent (8MHz	0,025	0,020

Conclusion

This study allowed the synthesis of stoichiometric cordierite from a single kaolin that is chemically modified by controlled acid leaching. The optimal conditions of leaching with HCl (6M), a temperature of 70°C, and a duration of 120 minutes allowed to modify the SiO_2/Al_2O_3 ratio and thus succeed in a stoichiometric mixture of cordierite, by adding precipitated magnesium hydroxide.

The study of thermal transformations of the starting mixture reveals the appearance of several phases, such as sapphirine and enstatite, before cordierite became predominant at 1250°C. These intermediate phases have higher densities than that of cordierite, and consequently their transformations in the cordierite phase are accompanied by swelling, which generally causes cracks in the ceramic.

The comparison of the physical properties of ceramics sintered from the starting mixture (direct sintering) and from cordierite powder pre-calcined at 1250°C (indirect sintering) shows that in the case of direct sintering, densification cannot reach that of theoretical cordierite (d_{th}) due to the formation of glass phases. In the case of indirect sintering, densification approaches d_{th} and reaches 94% at 1430°C.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest

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