

## Research Article

# Sol-Gel Synthesis of Mullite Starting from Different Inorganic Precursors

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Using silicotetraetilortosilicate (TEOS) mixed with aluminum tri-sec-butoxide (TSBAI) or aluminum cloaures mullite ceramics were created by the sol-gel method. The quantities used of each substance were those that led to obtain stoichiometric mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ). The experimental methodology used for obtaining mullite consisted in: sol-gel synthesis of precursor materials, isothermal treatment of those materials, and characterization of resulting materials. In order to determine the advance of reactions during mullite formation, isothermal treatments between  $300^\circ\text{C}$  and  $1600^\circ\text{C}$  were performed, keeping the samples at each temperature during 4 h. From XRD results, it may be said that precursor powders originally amorphous start to crystallize in  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  at  $1200^\circ\text{C}$ , and the mullite formation starts at  $1200^\circ\text{C}$ , with being completed at  $1600^\circ\text{C}$ . The use of TSBAI favors the formation of mullite crystals at lower temperature. From SEM observations a microstructure that presents primary mullite with randomly oriented grains of secondary mullite with acicular shapes and sizes that range between 1.25 and  $1.50\ \mu\text{m}$  long may be determined.

## 1. Introduction

Materials from the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$  system play an important role in the development of traditional and advanced ceramics. Mullite, a material obtainable at atmospheric pressures, is part of this system. Mullite is a rare mineral not found in abundant quantities in nature; although most of traditional ceramics have it as part of their final composition for mineral aluminosilicates are regularly used for its manufacturing. The importance of mullite lies in its good mechanical, thermal, chemical, and electrical properties which remain under elevated temperatures (about  $1500^\circ\text{C}$ ) [1–3]. Conventionally, mullite is produced by high temperature calcination of mixtures of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  [4]. The activation of energy for ion diffusion that takes place through the network of energy requires high temperatures; therefore, high sintering temperatures are required ( $>1700^\circ\text{C}$ ) to obtain dense bodies of mullite [5]. Moreover, the sol-gel process enables the production of amorphous and polycrystalline materials with special characteristics starting from submicron powders of

high purity [6–8]. Its usefulness lies in the fact that it requires low temperatures to obtain dense bodies compared to traditional manufacturing methods by fusion [6–8]. One of the many uses of mullite takes place in the electronics industry where it is used as substrate [1, 3]. A substrate is the support on which an electronic circuit, consisting of conductors, dielectrics, and integrated components, is mounted. The choice of substrate depends on the thermal, mechanical, and electrical characteristics of the circuit. Based on the above, the aim of this research is the development and characterization of mullite ceramics by the sol-gel method, in search of its applications as a substrate in electronics.

## 2. Experimental

The following experimental methodology for obtaining mullite consisted in the following steps.

- (i) Sol-gel synthesis of the mullite precursor materials.

TABLE 1: Reagents and their function in obtaining mullite.

Function	Reagents	Formula	Trademark
Precursor	Silicon tetraethyl orthosilicate	(TEOS)	Sigma-Aldrich
Precursor	Aluminum alkoxide, Aluminum tri-sec-butoxide	(TSBAI)	Sigma-Aldrich
Precursor	Aluminum chloride	(AlCl <sub>3</sub> )	Sigma-Aldrich
Thinner	Isopropanol	(2-propanol)	JT Baker
Hydrolyzing agent	Deionized water	H <sub>2</sub> O	JT Baker
Catalyst	Hydrochloric acid	(HCl)	

- (ii) Heat treatment of these materials for producing mullite.
- (iii) Characterization of both materials: precursors and heat-treated.

**2.1. Synthesis of Precursor Materials.** The synthesis of precursor materials was carried out using the reagents indicated in Table 1. The role of each is also mentioned. Considering that the TEOS-TSBAI precursors were used for obtaining sample 1 and for obtaining sample 2, a mixture of TEOS-AlCl<sub>3</sub> precursors was used.

The composition of mullite precursors was aimed at the stoichiometric relation: 3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>. Sample 1 was prepared using aluminum alkoxide (TSBAI) as a precursor of alumina, and sample 2 was prepared using aluminum chloride (AlCl<sub>3</sub>) also as precursor of alumina. In both samples the silica precursor was tetraethyl orthosilicate (TEOS).

**2.2. Hydrolysis Condensation.** The synthesis was carried out as follows.

- (i) The solutions were prepared at room temperature; they were kept separately and stirred magnetically continuously for fifteen minutes for homogenization.
- (ii) A three-necked flask was placed in a thermostatic bath and heated at a constant temperature of 80°C.
- (iii) Solution 1 was added in the three necked flask and mechanically stirred at 600 rpm during 20 minutes.
- (iv) Afterwards, the solution was added drop by drop to the hydrolyzing flask, while the stirring continued, till the end; the solution was stirred for 10 more minutes.
- (v) Subsequently, the solution was divided into 5 equal parts. Each part was added to the three-necked flask every 5 minutes.
- (vi) After the last addition of the solution to the three-necked flask, the solution was kept in constant agitation for 10 minutes.
- (vii) Upon completion of the reaction time, the solution was poured into a polypropylene container and was hermetically sealed for gelation.

**2.3. Aging.** The gelation process in the hermetically sealed container made of polypropylene consisted in reviewing and withdrawing the expelled liquid exudates (water, alcohol and acid) daily. This was done for 20 consecutive days, noting that

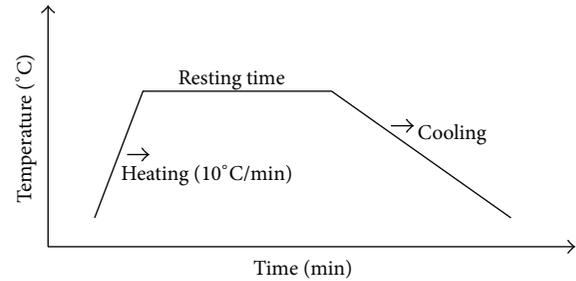


FIGURE 1: Heat treatment cycle.

during the last days the release of the remaining liquid was zero.

**2.4. Drying.** After the aging step, containers were partially opened very slowly for the solvent to evaporate and for the gel to dry up to a constant weight. This process was carried out at room temperature. Evaporation of the solvents was done very slowly in order to obtain powders. Finally, mullite precursor materials were dried at a temperature of 120°C for 8 days more.

**2.5. Isothermal Treatment.** Heat treatments for the study of the transformation phase of precursors to mullite materials were conducted in an electric furnace. The samples were placed in a sample holder of sintered high purity alumina. Figure 1 shows the isothermal process that was undergone by mullite precursor materials.

Figure 1 shows three zones.

- (i) Zone 1: stage of heating at a rate of 10°C/min to reach desired temperature (300°C to 1600°C).
- (ii) Zone 2: resting time of the material within the furnace (4 and 8 hours).
- (iii) Zone 3: furnace cooling period to room temperature with a cooling rate of 10°C/min.

**2.6. Samples Characterization.** To perform the characterization of the samples the following techniques were used: X-ray diffraction (XRD), infrared spectroscopy by Fourier transform (FT-IR), and scanning electron microscopy (SEM). These techniques were used in order to gather data about their chemical composition (physicochemical, thermal, and microstructural analyses).

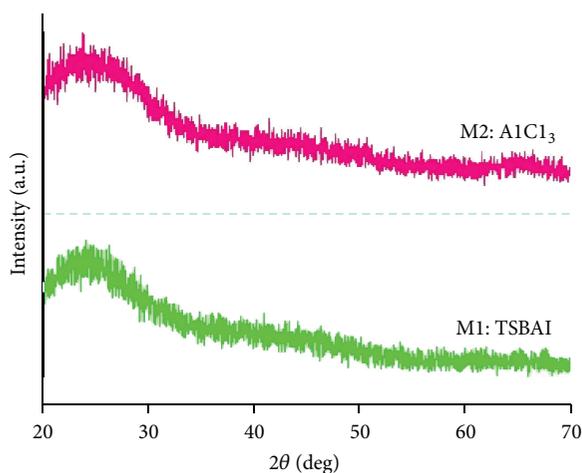


FIGURE 2: X-ray diffraction spectra of mullite precursor materials synthesized with TSBAI and  $\text{AlCl}_3$  and dried at  $110^\circ\text{C}$ .

### 3. Results and Discussion

**3.1. Chemical and Crystalline Structure of Mullite Precursor Materials.** The mullite precursor materials were obtained in powder. These powders were compacted and heat-treated for conversion to mullite. Figure 2 shows the spectra of X-ray diffraction of the mullite precursor material synthesized with TSBAI and  $\text{AlCl}_3$  and dried at  $110^\circ\text{C}$ . Figure 2 shows that mullite precursor materials have a short-range structure, independently of the type of aluminum precursor used. The broad peak with a maximum located at  $\sim 24^\circ$  in 2 theta, represents the presence of vitreous silica ( $\text{SiO}_2$ ).

Figure 3 shows the FT-IR spectra of the synthesized mullite precursor materials with TSBAI (M1) and  $\text{AlCl}_3$  (M2), dried at  $110^\circ\text{C}$ . In M1 sample, a shoulder can be observed at very low absorption intensity at  $1161\text{ cm}^{-1}$  which corresponds to the formation of mullite. The formation of silica is evidenced by the appearance of a band at  $1090\text{ cm}^{-1}$  corresponding to the Si-O-Si bond. Two absorption bands are assigned to the Si-O-Al bond, observed at  $820$  and  $791\text{ cm}^{-1}$ , suggesting that the aluminum forms a covalent-ionic bond with the Si-O bond during the hydrolysis-condensation reaction of the silicon alkoxides such as aluminum, forming Si-O-Al bonds in the presence of alumina and silicon oxide in the synthesized material. The band at  $729\text{ cm}^{-1}$  was assigned to the group of Al and/or Si tetrahedral,  $[\text{AlO}_4]$  and/or  $[\text{SiO}_4]$ , by its vibration mode. The absorption band derived from Al-O bond was observed at  $635\text{ cm}^{-1}$  [9–11]. In M2 sample, the absorption band observed at  $1060\text{ cm}^{-1}$  was assigned to the Si-O-Si bond; this band is seen shifted to a lower wave number, with respect to the  $1080\text{ cm}^{-1}$  of  $\text{SiO}_2$ ; this is due to the structure formed from the different precursors. The tetrahedral Al-O bond of lengthening was observed at  $922\text{ cm}^{-1}$  and the absorption band observed at  $779\text{ cm}^{-1}$  was assigned to the bending vibration of the tetrahedral Al or Si,  $[\text{AlO}_4]$  and/or  $[\text{SiO}_4]$  [9–11].

**3.2. Chemical and Crystalline Structure of Sintered Samples.** Figures 4 and 5 show the X-ray diffraction spectra of

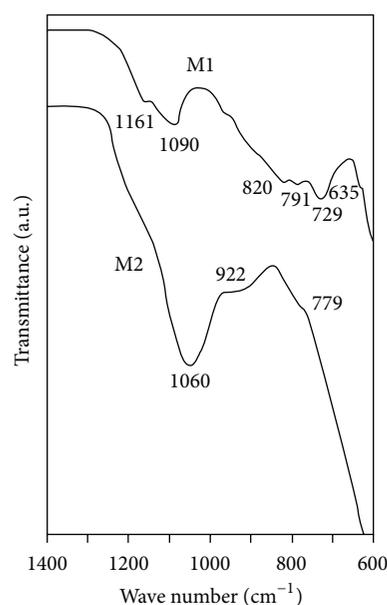


FIGURE 3: FT-IR spectra of mullite precursor materials synthesized with TSBAI (M1) and  $\text{AlCl}_3$  (M2).

mullite precursor samples synthesized with TSBAI and  $\text{AlCl}_3$ , respectively, and thermally treated in the range of  $300^\circ\text{C}$  to  $1000^\circ\text{C}$ . Figure 4 shows that in the temperature range of  $300^\circ\text{C}$  to  $600^\circ\text{C}$  the material retains its short-range structure. By increasing the temperature to  $900^\circ\text{C}$ , the material starts to crystallize, which is indicated by the presence of small diffraction peaks: two of these are located at  $23^\circ$  and  $46^\circ$  in two theta; and correspond to characteristic peaks of kyanite ( $\text{Al}_2\text{SiO}_5$ ); a third peak is located at  $67^\circ$  in two theta, and this refers to the peaks of stishovite ( $\text{SiO}_2$ ). It is observed that at  $1000^\circ\text{C}$  the intensity of the peaks continues to increase, which means that the increase in temperature favors the phase crystallization. In this spectrum four peaks in particular are highlighted: one at  $22.3^\circ$  that corresponds to cristobalite ( $\text{SiO}_2$ ), two more localized at  $37.6^\circ$  and  $46^\circ$  corresponding to kyanite, and a fourth peak located at  $67.6^\circ$  corresponding to stishovite.

Figure 5 shows that the material synthesized with  $\text{AlCl}_3$  retains its short-range structure when it is treated at temperatures between  $300^\circ\text{C}$  and  $600^\circ\text{C}$ , which was also observed in materials synthesized with TSBAI. At temperatures of  $900^\circ\text{C}$  to  $1000^\circ\text{C}$  the formation of small localized diffraction peaks at  $23$ ,  $46$ , and  $67^\circ$  is shown. The first is characteristic of the presence of cristobalite, the second refers to kyanite, and the latter corresponds to stishovite. Comparing this sample with the previous one, it may be said that the phase formation takes place at a much lower temperature.

Figures 6 and 7 show the XRD spectra mullite precursor samples synthesized with TSBAI and  $\text{AlCl}_3$ , respectively, and thermal-treated in the range of  $1200^\circ\text{C}$  to  $1600^\circ\text{C}$ . Figure 6 shows that the sample prepared with TSBAI presents a crystal structure, with well-defined peaks corresponding to the temperature of  $1200^\circ\text{C}$ , where the mullite formation is observed by the appearance of faint peaks at  $26.2$ ,  $26.7$ ,  $31.3$ ,

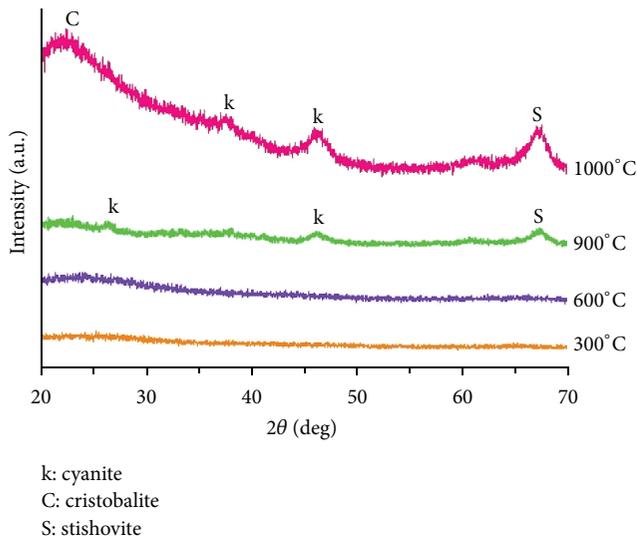


FIGURE 4: XRD spectra of synthesized samples with TSBAI and thermally treated in the range of 300°C to 1000°C.

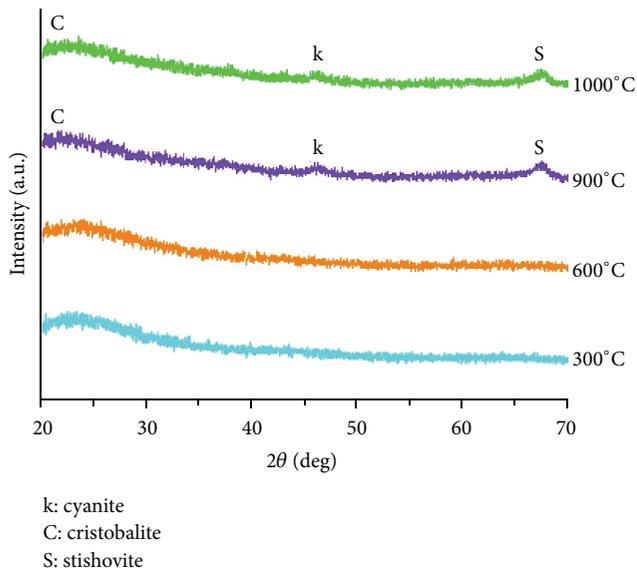


FIGURE 5: XRD spectra of synthesized samples with  $\text{AlCl}_3$  and thermally treated in the range of 300 to 1000°C.

33.6, 35.5, 39.6, 41.2, 42.9, 54.3, 57.8, 61, 64, and 66.8° in two theta. Moreover, the presence of three phases such as, cristobalite with characteristic peaks at 23° and 68.4°, silicon oxide with a peak at 38.1°, and finally corundum ( $\text{Al}_2\text{O}_3$ ) with characteristic peaks at 25.8, 43.6 and 52.9°. At 1300°C all above mentioned peaks are observed, and peak intensity increases steadily which is a characteristic of cristobalite at 23° in two theta. At 1400°C and 1500°C increments in peak intensity are observed. However, at 1600°C the disappearance of peaks of cristobalite, corundum, and silicon oxide is observed, because cristobalite ( $\text{SiO}_2$ ) and silicon oxide react with corundum ( $\text{Al}_2\text{O}_3$ ) favoring mullite formation, so those peaks characteristic of mullite are more intense at 1600°C.

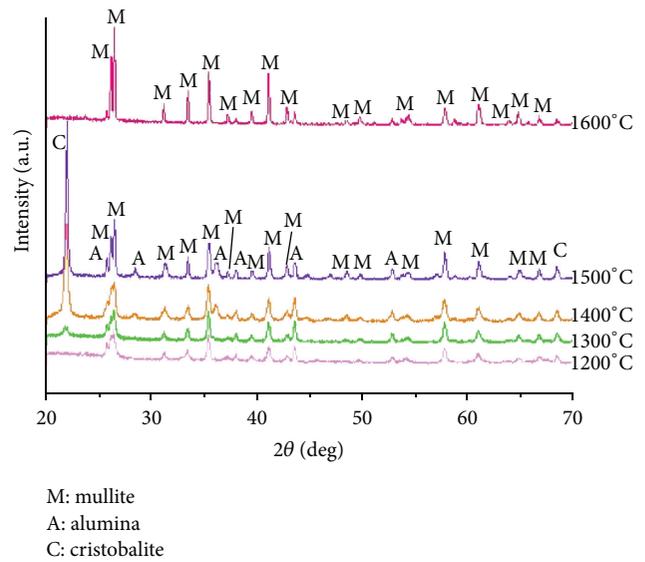


FIGURE 6: XRD spectra of synthesized samples with TSBAI and thermally treated in the range of 1200°C to 1600°C.

Figure 7 shows the sample prepared with  $\text{AlCl}_3$  and thermal treated in the range of 1200°C to 1600°C. The spectrum at 1200°C in Figure 7 shows low intensity and broad diffraction peaks similar to the samples treated at 900°C and 1000°C (Figure 6) unlike the sample prepared with TSBAI and treated at 1200°C which presents well-defined peaks. It still presents very low intensity peaks; four correspond to the formation of kyanite with characteristic peaks in 26, 35.5, 57.8, and 67.4° and two peaks at 43.7° and 46° in two theta, corresponding to the presence of stishovite (allotropic form of  $\text{SiO}_2$ ). At 1300°C, the formation of the characteristic peaks of mullite is observed; however, these peaks present low intensity. At 1400°C and 1500°C the mullite peaks increase their intensity. At 1400°C the cristobalite peak at 22° in 2 theta reaches its greatest intensity, and at 1500°C it begins to decrease. The reason for this is that cristobalite ( $\text{SiO}_2$ ) reacts with corundum ( $\text{Al}_2\text{O}_3$ ) favoring the formation of mullite. Therefore, at 1600°C total formation of mullite is shown. In this material the presence of mullite was evident at 1300°C indicating that the use of  $\text{AlCl}_3$  as a precursor of the  $\text{Al}_2\text{O}_3$  source delays the formation of mullite in 100°C unlike when an alkoxide of Al is used.

FT-IR spectra of the M1 sample synthesized with TSBAI in Figure 8 show the evolution of the formation of mullite from the sample without heat treatment to the sample heat-treated at 1600°C. In this figure, two absorption bands with maximums at 886 and 1014  $\text{cm}^{-1}$ , corresponding to Al-O and Si-O tetrahedral bonds and Si-O-Si bonds, respectively, are seen at 300°C. These absorption bands are shifted to lower wave numbers than those for the sample without heat treatment. As the temperature increases, it may be observed that in spectra of 600, 900, 1000, and 1200°C there are a shift and a change in shape of the bands that correspond to Al-O and Si-O-Si bonds, due to a rearrangement in the material structure by thermal treatment effect. At 900°C and 1000°C it

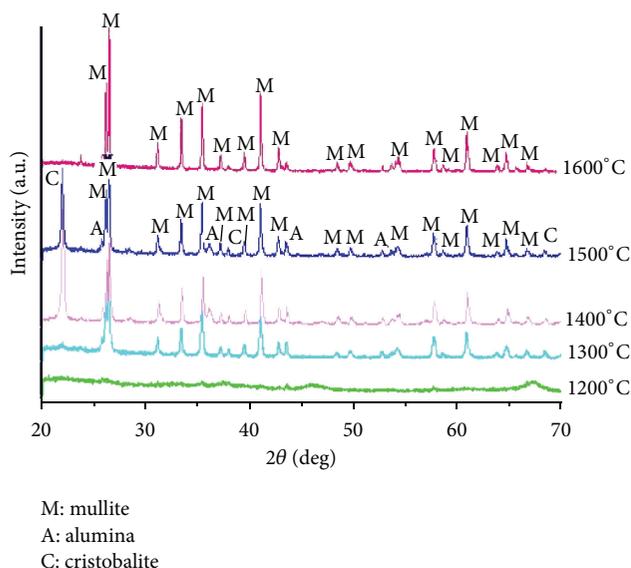


FIGURE 7: XRD spectra of synthesized samples with  $\text{AlCl}_3$  and thermal treated in the range of 1200°C to 1600°C.

may be observed that the maximum of the band at  $1014\text{ cm}^{-1}$  in the spectra of 300°C is shifted to  $1080\text{ cm}^{-1}$ . Spectra 1200, 1300, 1400 and 1500°C are similar. In these spectra five absorption bands localized at 1164, 1104, 845, 750 and  $635\text{ cm}^{-1}$  are found. The absorption band at  $1064\text{ cm}^{-1}$  is assigned to vibration of the Al-O bond, the Si-O bond is present at  $1104\text{ cm}^{-1}$ , two bands characteristic of the mullite formation are observed at 845 and  $750\text{ cm}^{-1}$ , the last band that appears at  $635\text{ cm}^{-1}$  is assigned to vibration of the Al-O octahedral bond. As may be seen from these results, the presence of absorption bands corresponding to the formation of mullite are present from 1200°C, in agreement with XRD results. Finally, at 1400 and 1500°C an absorption band at  $788\text{ cm}^{-1}$  assigned to the presence of Al-O or Si-O tetrahedral  $[\text{AlO}_4]$  and  $[\text{SiO}_4]$  is present. The spectrum of 1600°C is similar to the spectrum of 1500°C with the difference that the band at  $788\text{ cm}^{-1}$  assigned to Si-O bond that corresponds to the cristobalite is no longer seen. This agrees with the results of XRD where, at 1600°C, the formation of mullite with little alumina presence is observed. In this spectrum (1600°C) the absorption bands corresponding to mullite, located at 1164, 1104, 845, 750 and  $635\text{ cm}^{-1}$ , discussed above may be observed. According to the above, it may be said that the M1 sample synthesized with TSBAI shows the formation of mullite at a temperature of 1200°C. This is transformed completely at 1500°C/8 h or 1600°C/4 h. This agrees with the analysis of X-ray diffraction.

FT-IR spectra of the M2 sample synthesized with  $\text{AlCl}_3$  in Figure 9 show the evolution of the mullite formation from the sample without heat treatment to the sample heat-treated at 1600°C. Figure 9 shows an absorption band with a peak at  $1080\text{ cm}^{-1}$  assigned to Si-O-Si bond characteristic of the tetrahedron of  $[\text{SiO}_4]$  at 300°C. The spectra at 600, 900, 1000, and 1200°C are similar. In these spectra, there are two

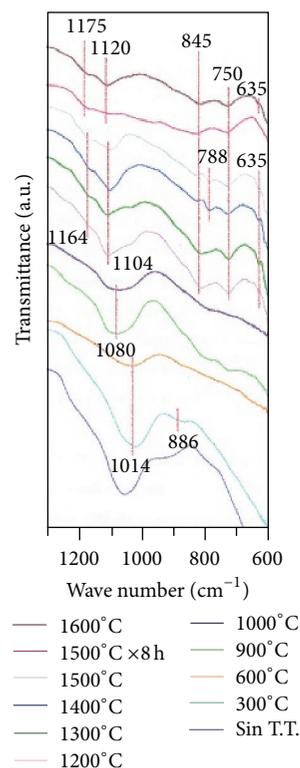


FIGURE 8: FT-IR spectra of the samples synthesized with TSBAI and heat-treated at 300 to 1600°C.

localized absorption bands at 1080 and  $781\text{ cm}^{-1}$ . The first band shows small shifts to the right and left in the same location represented in the Si-O-Si bond present at 300°C. The band formed at  $781\text{ cm}^{-1}$  is assigned to Al-O or Si-O tetrahedral bond  $[\text{AlO}_4]$  and  $[\text{SiO}_4]$ . In the spectrum at 1200°C a third band at  $717\text{ cm}^{-1}$  is observed that represents the Al-O octahedral bond. The spectra at 1300, 1400, and 1500°C have six absorption bands localized at 1241, 1175, 1120, 845, 750 and  $634\text{ cm}^{-1}$ . The observed band at  $1241\text{ cm}^{-1}$  is assigned to the vibration of the Si-O bond. Four bands, characteristic of mullite formation, are observed at 1175, 1120, 845 and  $750\text{ cm}^{-1}$ . The last band at  $634\text{ cm}^{-1}$  is assigned to the vibration mode of the Al-O octahedral bond. The spectrum at 1600°C shows five absorption bands: four are characteristic mullite bands present at 1175, 1120, 845, and  $750\text{ cm}^{-1}$ , and the last band at  $634\text{ cm}^{-1}$  represents the Al-O octahedral bond. The spectrum of 1500°C/8 h shows similarity with the spectrum of 1500°C/4 h. In the analysis of the M2 sample synthesized with  $\text{AlCl}_3$  the formation of mullite from 1300°C is observed. In addition, it was observed that the increase in the residence time of the sample in the heat treatment (1500°C/8 h) does not favor the total formation of mullite, which agrees with the results of X-ray diffraction, previously analyzed.

**3.3. Microstructure.** Figure 10 shows a micrograph taken with a SEM of the M1 sample synthesized with TSBAI and heat-treated at 1500°C/8 h. This micrograph shows the massive

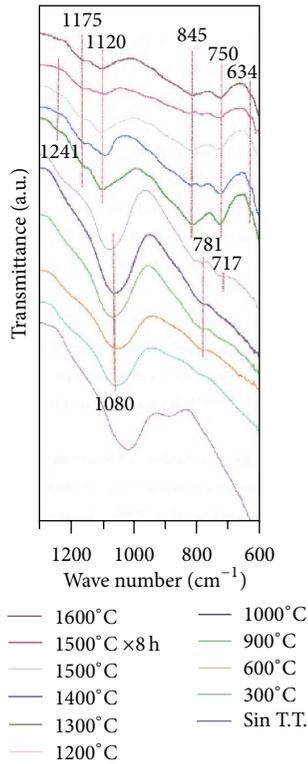


FIGURE 9: FT-IR spectra of the samples synthesized with  $\text{AlCl}_3$  and heat-treated at 300 to 1600°C.

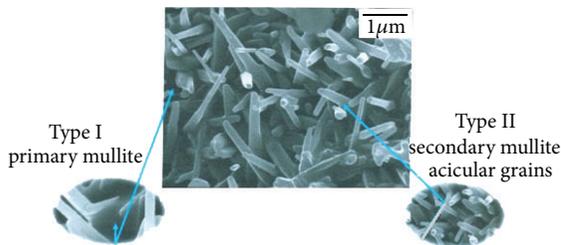


FIGURE 10: Micrograph taken with a SEM of the M1 sample synthesized with TSBAI and heat-treated at 1500°C/8 h. (a) Inner sample; (b) magnified inner sample.

formation of type II secondary mullite acicular grains with a random orientation, obtaining an average grain size of  $\sim 1.5 \mu\text{m}$  long and  $0.25 \mu\text{m}$  wide. These mullite grains are embedded in a network of primary mullites. The random orientation with respect to length size of grain and towards the left from the mullite matrix type I, generates porosity between the grains as shown in the cavity in Figure 10(b). The density of this M1 sample is high due to narrow accommodation of the acicular mullite crystalline grains. These results agree with those obtained by XRD with the presence of total mullite and in FT-IR with the unique formation of the characteristic bands of mullite.

Figure 11 shows the morphology of the M2 sample synthesized with  $\text{AlCl}_3$  and heat-treated at 1500°C/8 h. This micrograph shows the formation of secondary mullite acicular grains with a random orientation, obtaining an average grain

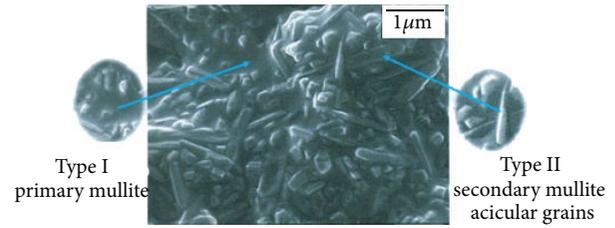


FIGURE 11: Micrograph taken with a SEM of the M2 sample synthesized with  $\text{AlCl}_3$  and heat-treated at 1500°C/8 h. (a) Inner sample; (b) magnified inner sample.

size of  $\sim 1.25 \mu\text{m}$  long and  $0.25 \mu\text{m}$  wide. These grains are embedded in a network of primary mullite, in which the grain size is heterogeneous thus decreasing the density and increasing porosity of the material, due to the same grain size difference [12].

#### 4. Conclusions

- (i) According to FT-IR results, the sample synthesized with TSBAI without heat treatment presents the formation of Si-O-Al bonds due to the presence of alumina and silica, whereas the sample synthesized with  $\text{AlCl}_3$  without heat treatment presents the formation of Si-O-Si and Al-O bonds corresponding to cristobalite and corundum, respectively. This corroborates that mullite is present from the synthesis stage (hydrolysis-condensation).
- (ii) Based on the results of XRD and FT-IR, the onset of crystallization of mullite was obtained at 1200°C in the sample synthesized with aluminum alkoxide (TSBAI), taking advantage of 100°C compared to the sample synthesized with aluminum chloride ( $\text{AlCl}_3$ ).
- (iii) Based on the XRD, FT-IR and SEM results and comparing the two samples obtained at 1600°C/4 h, the sample synthesized with TSBAI presents mullite, corundum, and minimal cristobalite formation, while the sample synthesized with  $\text{AlCl}_3$  only presents the formation of mullite and corundum.

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