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Effect of microwave irradiation on the structural, chemical, and hydrophilicity characteristics of ordered mesoporous silica SBA-15

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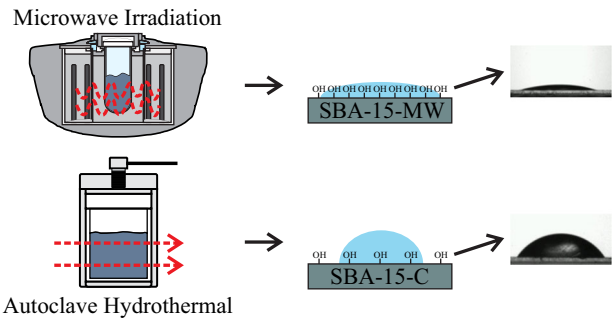
Abstract

Structural, chemical, morphological, and hydrophilic surface characteristics of SBA-15 silica synthesized by hydrothermal method in an autoclave and in a microwave-heated reactor were analyzed in this work. The influence of time and temperature on these characteristics was investigated. Materials were characterized by textural analysis (BET), FTIR, TGA, XRD, goniometer contact angle, SEM, and TEM. It was possible to obtain SBA-15 silicas assisted by microwave heating irradiation with short-time synthesis (0.5–8 h) compared with the hydrothermal classic method (24 h). Materials presented surface area between 650 and 850 $m^2 g^{-1}$. Through the XRD and TEM results, it was observed that mesostructured materials with two-dimensional, long-range, and hexagonal ordered channels are characteristic of SBA-15. This study showed that heating by microwave irradiation during silica synthesis generates materials with a greater amount of silanol groups. It is possible to control the surface properties of SBA-15 during the microwave-assisted synthesis through temperature tuning. Higher temperature provided SBA-15 with higher hydrophilicity.

Graphical Abstract



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Keywords Mesoporous silica · Microwave irradiation · Silanol · Hydrophilic SBA-15

Highlights

- SBA-15 synthesis by MW reduces significant reaction time.
- SBA-15 synthesized in MW at 80 °C showed 805 $m^2 g^{-1}$ of specific surface area.
- Heating by MW in the silica synthesis generates materials with higher hydrophilicity.

1 Introduction

Silica has been used in a variety of applications, including thermal insulation, catalytic supports, optical glasses, and membranes [1–5]. In addition, mesoporous silica-based materials are widely used in adsorption, chromatography, drug delivery, and catalysis [1–7]. In the 1990s, research groups began studies on the production of mesostructured silicas—materials that have medium- and long-range ordered mesopores—as well as a narrow pore size distribution and controlled pore volume such as MCM-41, MCM-48, MSU-H, HMS, SBA-1, SBA-2, and SBA-15 [8–13].

Silica SBA-15 is a mesostructured material that has unidirectional mesoporous structure with ordered pores, high surface average area of 600–1000 $m^2 g^{-1}$ [14], and uniform and hexagonal pore size, besides providing large thermal and hydrothermal stability when compared with other mesostructured materials such the M41S family [15]. Its pore diameter can range from 3 to 30 nm, with narrow pore size distributions. Also, it presents mesopores and micropores interconnected in the silica tubes. Thus, this material has been attracting a great interest to study not only the application of this kind of silica, but also its synthesis steps [16]. Because of these characteristics, SBA-15 has been used in a variety of laboratory applications and industries, such as the removal of organic pollutants from air and water, catalytic reactions, enzyme immobilization, controlled drug release, desulfurization, CO_2 adsorption, removal of metals, and oxidative reactions [1–3, 6, 17–20].

Synthesis of SBA-15 is commonly carried out by the hydrothermal process with conventional heating as reported by Zhao et al. [21]. The authors synthesized the SBA-15 through a treatment at 100 °C by using an autoclave for 24 h. It is verified in this synthesis that high temperature and long reaction time are necessary for the formation of the silica SBA-15. In order to reduce the energy expenditure of this process, there is a constant search for new methods to reduce the reaction times and increase the reaction yield, e.g., intensifying the synthesis process using microwave irradiation [22].

Use of microwave irradiation (MW) in various organic and inorganic synthesis processes has been tested previously [23–26]. The synthesis performed by microwave irradiation offers advantages over conventional synthesis, such as the transfer of energy directly to the sample, there being no physical contact with the heating source, reduction in the reaction time, and a highly uniform, rapid, and volumetric heating rate [27]. This technique also enables an efficient control in the pore size distribution and particle diameter [27–29]. In addition, it is a technique that reduces the use of solvents and energy, since it is a faster process when compared with the synthesis using an autoclave [30, 31].

Tompsett et al. [32] reported that mesoporous silica was first synthesized with MW heating by Wu Bein in 1996. Recently several authors have been synthesizing different silicas with heating using MW irradiation. Chaignon et al. [33] synthesized mesostructured porous silicas of the MCM-41 type using microwave irradiation, and evaluated the effect of temperature (150 and 180 °C) on the synthesis process. The authors verified that the appropriate use of microwave irradiation not only reduces the synthesis time of mesoporous silicas, but when combined with programmed temperatures, it is possible to obtain well-ordered materials with consolidated structures and good thermal stability when compared with materials obtained by the conventional hydrothermal process.

De Conto et al. [34] performed the synthesis of silica xerogel modified with an ionic liquid by the sol–gel method with heating by microwave irradiation at a power of 300 W in three different temperatures (40, 60, and 80 °C). An increase in surface area and pore volume was observed as the reaction temperature increased. In the work of Peres et al. [25], the synthesis of silica nanoparticles was carried out through the rice husk using microwave heating at a temperature of 80 °C and time of 30 min. The authors concluded that microwave synthesis generated a material with higher surface area, pore volume, and porosity, as well as a material with higher purity in a shorter time. This happens because, in comparison with conventional methods, microwave heating is rapid volumetric heating without the conductive heat process, which can achieve even heating in a short period of time [31]. Thus, it can be said that heating by microwave irradiation is an efficient tool to be used in synthesis of organic–inorganic materials, and that besides reducing the reaction time, it is also possible to provide a greater uniformity in the pores of the same.

Pirez et al. [35] studied the influence of HCl concentration on the synthesis of SBA-15 silica by conventional heating and

reported that the higher the amount of acid present in the synthesis step, more hydrophilic would be the silica surface. Lopez et al. [36] evaluated the influence of the calcination temperature on the removal of the surfactant P123 from the silica SBA-15 and found out that the temperature had a great effect on improving the texture properties, and in the total amount of silanol groups. Recalling that it is of great importance to evaluate and control these textural and morphological characteristics, these properties are primordial for knowledge of the material and its later applications.

However, there are no reports in the literature on the influence of heating by microwave irradiation on the hydrophilic or hydrophobic characteristics of ordered mesoporous silicas. In this context, the objective of this study was to compare the structural, chemical, and morphological properties of the SBA-15 synthesized by the conventional method (autoclave in an oven) and using a single-mode microwave reactor (CEM, Discover model). Moreover, the reaction time, temperature, and the effect of microwave irradiation on the structural characteristics, such as surface area and pore volume, chemical with respect to chemical surface groups indicating the hydrophilicity of the materials, were evaluated, besides checking the morphological characteristics of the mesoporous ordered SBA-15 silicas.

2 Materials and methods

2.1 SBA-15 hydrothermal synthesis by autoclave (AC)

Synthesis of SBA-15 material was performed as an adaptation of Zhao et al. [10] procedure and it is summarized in Fig. 1 — route A. A mass of 4.0 g of triblock copolymer Pluronic® P123 (Sigma Aldrich) was dissolved in a round-bottom flask with 150 mL of hydrochloric acid (Merck) solution at 1.6 mol L^{-1}. The mixture was heated up to 40 °C under stirring, and then 9.1 mL of tetraethyl orthosilicate (Sigma Aldrich) was added dropwise. The system was kept under stirring in ambient atmosphere at 40 °C for 24 h, and soon after, transferred to a Teflon coated stainless-steel autoclave, heated up to 100 °C, and maintained at these conditions for 24 h. The obtained white solid was filtered, washed with distilled water, and dried at 80 °C for 8 h. To remove the surfactant and clear the pores, the material—hereafter called SBA-15-C—was calcined for 6 h at 550 °C (heating rate of 10 °C min^{-1}).

2.2 SBA-15 hydrothermal synthesis by microwave irradiation (MW)

Figure 1 — route B presents the synthesis of SBA-15 assisted by MW irradiation, where the silica synthesis procedure followed the steps of topic 2.1. However, in the correspondent hydrothermal step the mixture was transferred to a quartz tube and introduced in a microwave CEM reactor, model Discovery Synthesis, with temperature and power control. Two temperatures were evaluated (60 and 80 °C), changing reaction times between 0.5 and 8 h, with a maximum power of 300 W. The experimental parameters are presented in Table 1. The white solid obtained was filtered, washed with distilled water, dried, and calcined at the same conditions of the SBA-15-C silica. These samples will be called SBA-15 M*x*-*y*, where *x* indicates the temperature and *y* is the reaction time under microwave irradiation.

2.3 Characterization

All samples were pretreated in oven for 20 h at 110 °C for analysis achievement. Specific surface area (S_{BET}) was determined by a multipoint BET (Brunauer-Emmett-Teller) method using adsorption data. The pore volume and pore size distribution were calculated using desorption branches

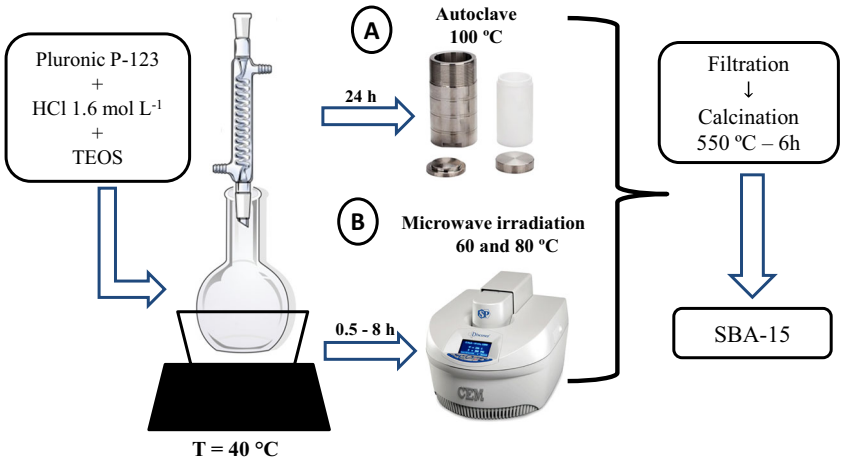

Fig. 1 Scheme with synthesis routes (hydrothermal treatment in an autoclave and microwave reactor) used to obtain SBA-15 materials

Table 1 Experimental parameters used in the microwave-irradiated synthesis of SBA-15 materials in the temperatures of 60 and 80 °C with power of 300 W

Experimental conditions		
Sample	Temperature (°C)	Time (h)
SBA-15 M60-0.5	60	0.5
SBA-15 M60-1		1
SBA-15 M60-2		2
SBA-15 M60-4		4
SBA-15 M60-8		8
SBA-15 M80-0.5	80	0.5
SBA-15 M80-1		1
SBA-15 M80-2		2
SBA-15 M80-4		4
SBA-15 M80-8		8

of nitrogen isotherms by BJH (Barrett-Joyner-Halenda) and HK (Horvath and Kawazoe) models performed in a Micromeritics 3020 Krypton equipment. The samples were previously heated at 120 °C under vacuum and maintained for 10 h. Fourier transform infrared spectroscopy (FTIR) was performed using an attenuated total reflectance spectrometer (ATR, Perkin Elmer). Spectra of silica materials were obtained in the wavenumber range of 4000–650 cm^{-1}, with 32 cumulative scans and maximum resolution of 2 cm^{-1}. X-ray diffraction (XRD) analyses were obtained using a diffractometer (Siemens model D500) with Cu-Kα as X-ray source ($\lambda = 0.15418$ nm), with 2-theta angle from 0.2° to 10° and step of 0.02°. Thermogravimetric analyses (TGA) were performed in the Shimadzu instrument model TGA-50, under argon flow (50 mL min^{-1}), heating rate of 10 °C min^{-1}, from 25 °C up to 800 °C. Materials morphology were obtained by scanning electron microscopy (SEM) using a FEI microscope model Quanta 250. The samples were added to a double-sided carbon conductive tape and coated with a thin film of gold. Transmission electron microscopy (TEM) images were acquired in a JEOL JEM-1220 microscope, operated at 80 kV. Samples were dispersed by sonication in isopropyl alcohol for 5 min, and three drops of the supernatant were placed onto a carbon-coated Cu grid.

To verify the materials hydrophilicity the contact angle between the silicas and ultrapure water was determined. For these analyses, the glass substrates (18 × 18 mm, WWR) on which the silicas were deposited were firstly cleaned. Thus, the glass substrates were placed in Falcon tubes submerged in tetrahydrofuran (THF-Synth, 99%), after which they were placed in tubes with water and soap and finally in tubes with distilled water. Substrates were sonicated for 5 min and dried with N_2. In the last step the substrates were submerged in acetone (Synth, 99%), sonicated for 15 min, and dried with N_2. The silica samples were dispersed in isopropanol. Then three drops of the suspension were dripped in the cleaned substrate. This procedure was repeated four times with 10 min intervals. The contact angle (CA) analyses were performed in a pendant drop tensiometer (Teclis Tracker, IT Concept). Water drop (5 µL) was dripped onto the substrate and the contact angle between the water and the surface was determined.

3 Results and discussion

SBA-15 samples were obtained by microwave irradiation varying the temperature and irradiation time, and were compared with SBA-15-C obtained by the hydrothermal classic method. Nitrogen adsorption/desorption isotherms were obtained for the synthesized materials and are presented in Fig. 2. Type IV (A) isotherms are observed for all materials, with parallel curves typical of H1 hysteresis that indicates the uniform distribution of mesopores [37]. Usually, the H1 hysteresis loop represents a steep adsorption step, indicating capillary condensation in the pores of mesoporous silica, where the isotherm showed a sharp inflection at a relative pressure P/P_0 of 0.5–0.8. Figure 3 presents the BJH pore size distribution of the materials, in which the microwave-irradiated samples (SBA-15-MW) showed a unimodal but broader curve when compared with autoclave hydrothermally treated material (SBA-15-C). Furthermore, Fig. 3 shows the pore size distribution by the HK model, thus showing that in addition to mesopores the silica SBA-15 presents micropores in the region of 0.6–2 nm.

Among the microwave-irradiated materials, the BJH pore size distribution for the temperature of 80 °C was narrower than 60 °C, thus indicating a more uniform pore distribution. Regarding the time of irradiation, there are only slight influences on the pore diameter for the SBA-15-M80 series, while the SBA-15-M60 series with irradiation time lower than 2 h presented smaller pores and broader pore distribution [34]. BET specific surface area and BJH pore volume of the materials are summarized in Table 2. SBA-15 silica obtained by the autoclave conventional hydrothermal method (SBA-15-C) presented higher surface area and pore volume when compared with the microwave-irradiated materials, however the amount of time involved in autoclave synthesis is 12 times higher, when comparing with materials that have been synthesized for 2 h in microwave, such SBA-15-M80-2 material. The smallest surface area of silicas synthesized by microwave irradiation is related to radiation action on the sol–gel reaction kinetics, leading to a more condensed network [38]. Among the materials synthesized by microwave irradiation, the specific surface area for most of them was very similar, but it is worth

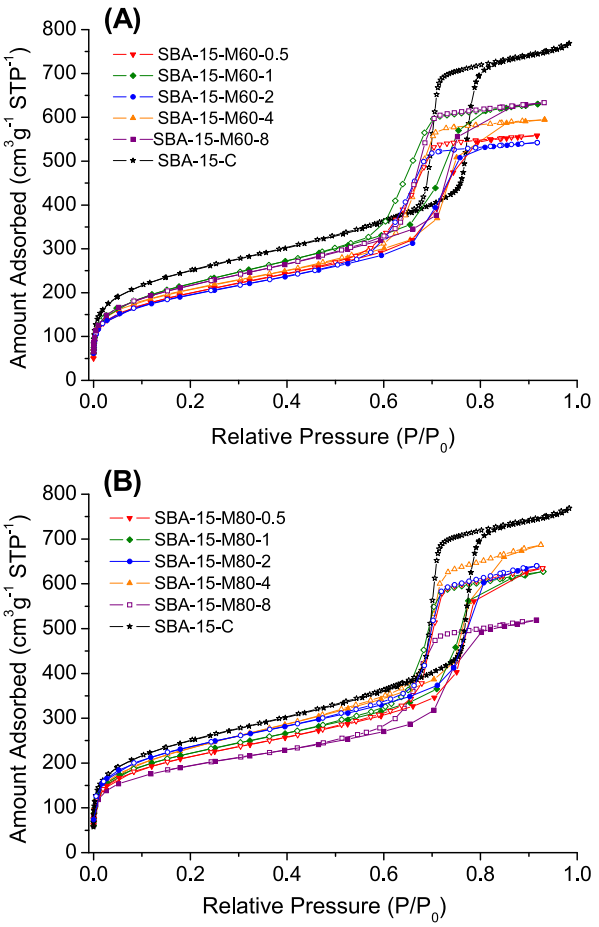

Fig. 2 Nitrogen adsorption/desorption isotherms obtained for the materials synthesized by microwave irradiation in the temperatures 60 °C (**a**) and 80 °C (**b**), and by the autoclave conventional hydrothermal method

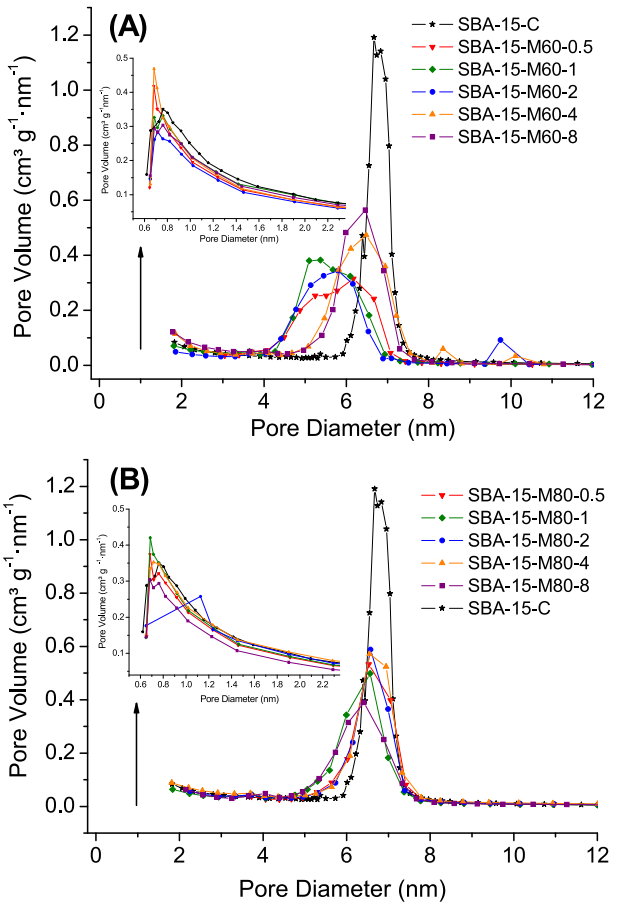

Fig. 3 BJH and HK pore size distribution obtained for the materials synthesized by microwave irradiation in the temperatures 60 °C (**a**) and 80 °C (**b**), and by the autoclave conventional hydrothermal method

mentioning that the materials prepared at 80 °C for 2 and 4 h presented the highest surface area values.

At a temperature of 80 °C, it was possible to obtain materials with a larger surface area and pore volume. The difference in the area of these materials was around 80 $m^2 g^{-1}$, showing that with only 30 min it was possible to obtain a material with high surface area. Finally, it should be noted that the SBA-15-M80-2 sample presented better textural properties, since with only 2 h of reaction it presented results similar to SBA-15-M80-4, being this difference of 7 $m^2 g^{-1}$ and 0.10 $cm^3 g^{-1}$ in the surface area and pore volume, respectively. The advantages when using microwave irradiation to prepare silica materials were also perceived by Dudarko et al. [39], where the authors succeeded in obtaining mesoporous silica and pointed out the fast and homogeneous heating, quick crystallization, and low time required for synthesis when compared with the conventional method using autoclave [40]. Lai et al. investigated the efficiency of microwaves in removing P123 surfactant and showed that the method generated materials with a larger surface area, lower structural shrinkage, and a higher amount of silanol groups on the surface than the sample treated by calcination [41].

Table 2 Textural properties of the materials synthesized by the microwave irradiation and autoclave conventional hydrothermal method

Sample	BET surface area ($m^2 g^{-1}$)	BJH pore volume ($cm^3 g^{-1}$)
SBA-15-C	850	1.06
SBA-15-M60-0.5	682	0.80
SBA-15-M60-1	763	0.87
SBA-15-M60-2	664	0.74
SBA-15-M60-4	702	0.86
SBA-15-M60-8	742	1.02
SBA-15-M80-0.5	726	0.85
SBA-15-M80-1	751	0.86
SBA-15-M80-2	798	0.83
SBA-15-M80-4	805	0.93
SBA-15-M80-8	650	0.75

Table 3 Textural parameters of SBA-15-C, SBA-15-M60-2, and SBA-15-M80-2

Sample	d_p (nm)	2θ	d_{100} (nm)	a_o (nm)	t_w
SBA-15-M60-2	5.72	0.89	9.947	11.49	–
SBA-15-M80-2	6.59	0.86	10.418	12.03	5.44
SBA-15-C	6.75	0.84	10.488	12.11	5.36

d_p = BJH pore diameter, d_{100} = interplanar spacing for (100) reflection, a_o = distance between the centers of two adjacent cylinders, t_w = wall thickness estimated from ($a_o - d_p$)

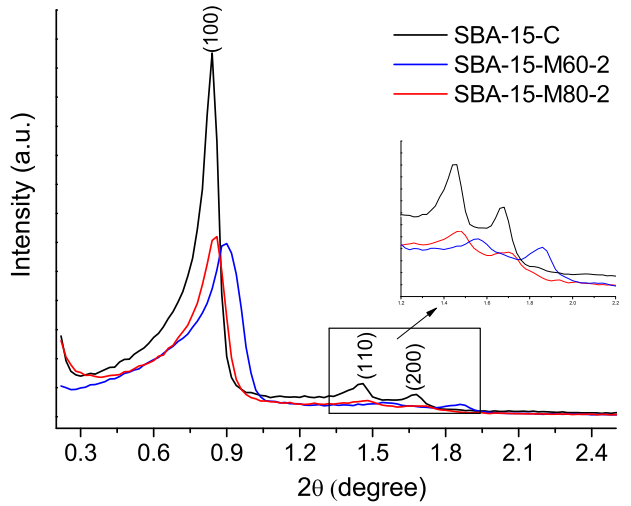

Fig. 4 X-ray diffraction profile obtained for the autoclave conventional hydrothermal method and microwave-irradiated (60 and 80 °C—2 h) SBA-15

Due to small variation in the surface area values, it was decided to choose the samples synthesized in 2 h at 60 and 80 °C to perform the other characterizations.

The hexagonal structure of the synthesized materials was evaluated by XRD. Figure 4 presents the obtained diffraction profile for SBA-15 synthesized by the autoclave conventional hydrothermal method and microwave irradiation. For all materials, the main peak located at $2\theta = 0.84 - 0.89°$ is notably present and is attributed to the (100) plane, while the two secondary peaks are assigned to (110) and (220) planes. This set of peaks is characteristic of the hexagonal and well-ordered arrangement of the SBA-15 channels [10]. It is noted that the autoclave-synthesized silica presents more defined peaks; however, it is noteworthy that the synthesis time is 12 times higher (AC—24 h, MW—2 h). Furthermore, the appearance of the three reflections peaks and the angular values (2θ) relative to the plane (100)—allowable range $2\theta =$ 0.5–3—confirms the formation of a two-dimensional hexagonal mesoporous structure with p6mm symmetry for all materials [10].

In Fig. 4, it is clearly observed that with increasing the temperature applied during synthesis by microwave irradiation, the shift of the diffraction plane (100) occurs to lower 2θ, which indicates an increase in the interplanar spacing and in the distance between the centers of two adjacent pores. Analyzing the results of silica synthesized by the autoclave conventional hydrothermal method, it is clear that the displacement and the intensity of the silicas synthesized by microwave irradiation reduce the interplanar spacing, however, this displacement does not hinder the formation of the ordered silica network [16, 29, 35]. According to Lakhi et al. [28], this shift is related to the temperature applied in the synthesis, and as higher is the temperature, greater is the interplanar spacing. The interplanar spacing credited to the (100) plane (d_{100}) can be related to the distance between the centers of two adjacent pores (a_o), and a shift of these values indicates changes in the thickness of the pore wall and/or in the pore size. This feature is related either to a thickening of the pore wall or to bigger pore diameters for SBA-15-M80-2 material. From the XRD data, it was possible to estimate the a_o parameter, and by combining with the pore size diameter obtained by textural analysis, it was possible to estimate the thickness of the pore wall. The obtained data are presented in Table 3. However, it was not possible to calculate the wall thickness of the SBA-15-M60-2 because it had a wide pore size distribution as can be seen in Fig. 3, which causes the error of this distribution to be high, and consequently, the thickness calculation cannot be accurately measured.

The morphology of the ordered mesoporous materials was observed by SEM analysis and is presented in Fig. 5. Through the microstructural analysis, it was possible to observe the long-rod morphology with relatively uniform sizes (1st column) and spherical morphology (2nd column) [16]. Also, it can be observed that even by using the microwave irradiation in the synthesis, the samples SBA-15-M60-2 and SBA-15-M80-2 presented the same morphology of the SBA-15-C prepared in the autoclave conventional hydrothermal method.

TEM images of SBA-15 are shown in Fig. 6, where the hexagonal channels (1st column) with long-range ordering for all materials can be seen. It can be observed that through the microwave irradiation process it is possible to obtain ordered mesostructured materials similar to the classical hydrothermal method (24 h—100 °C), but with lower temperatures (60 and 80 °C) and much shorter (2 h) time. In addition, images acquired perpendicular to the axis of the pores (2nd column) showed that the set of channels are equidistant and well ordered, where the clear regions are the channels and the dark region, the pore walls. Dudarko et al. [39] also confirmed the structural ordering of the SBA-15 material by acquiring images in perpendicular direction to the mesopores, also along with the channels. It was also verified that even using the microwave irradiation in the synthesis of silica, the materials presented an ordered characteristic of the SBA-15.

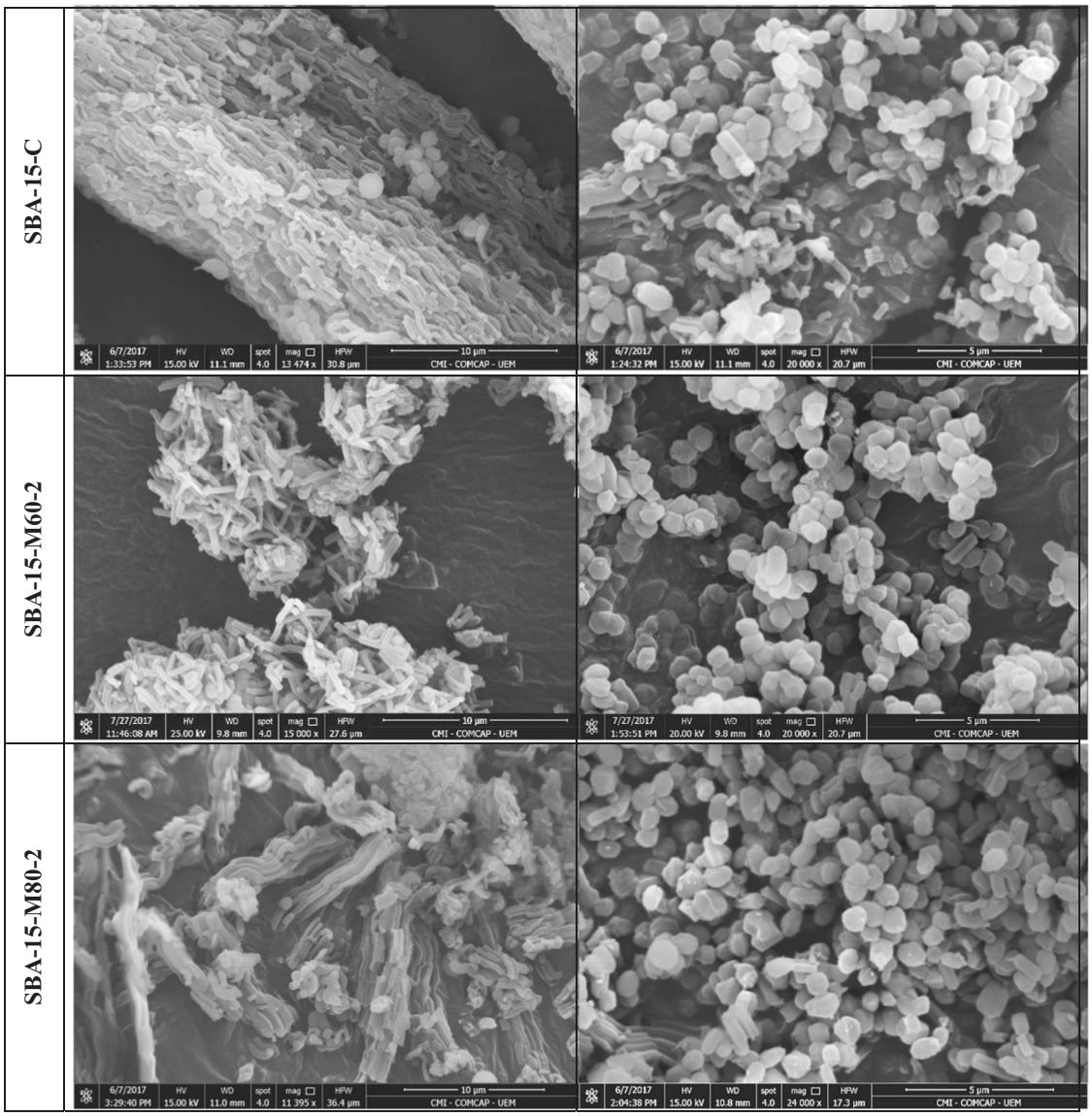

Fig. 5 SEM images acquired for the autoclave conventional hydrothermal method and microwave-irradiated (60 and 80 °C—2 h) SBA-15

Figure 7 presents the FTIR spectra for the autoclave conventional hydrothermal method and microwave-irradiated SBA-15 for heating temperatures of 60 and 80 °C and irradiation time of 2 h. In all silicas it is possible to verify the band at 1640 cm^{-1} characteristic of hydroxyl groups of water bending. Three bands located close to 800, 900, and 1100 cm^{-1} were observed for all materials and were ascribed to the vibrations of Si–O–Si and Si–OH coming from free silanol groups, typical of silica materials [42]. These bands are well defined indicating that all materials present uniform network vibrations, probably as a consequence of the well-ordered and condensed structure. It is also noted in Fig. 7a that the materials exhibited vibrational modes over the range 3000–3700 cm^{-1} assigned to isolated, germinal, and vicinal silanol groups, and adsorbed water molecules [35]. As all samples were pretreated (110 °C in oven—20 h) before the analyses, it is believed that the higher intensity of the bands is due to the effect of microwave irradiation in the synthesis step. According to Pirez et al. [35], the increase of gelling time in acid media (2–24 h) generates the condensation of vicinal silanols, causing more hydrophobic silanol species to be redistributed. Furthermore, it is noticed in Fig. 7b that the materials synthesized by microwave reactor have more intense bands in the region of the silanol groups (960 cm^{-1}) than the materials synthesized by the autoclave conventional hydrothermal method, generating an increase in the extension of polymerization reactions, indicating that this band belongs to a more condensed silica network [33, 38]. This fact can be related to the higher hydrophilicity of these silicas.

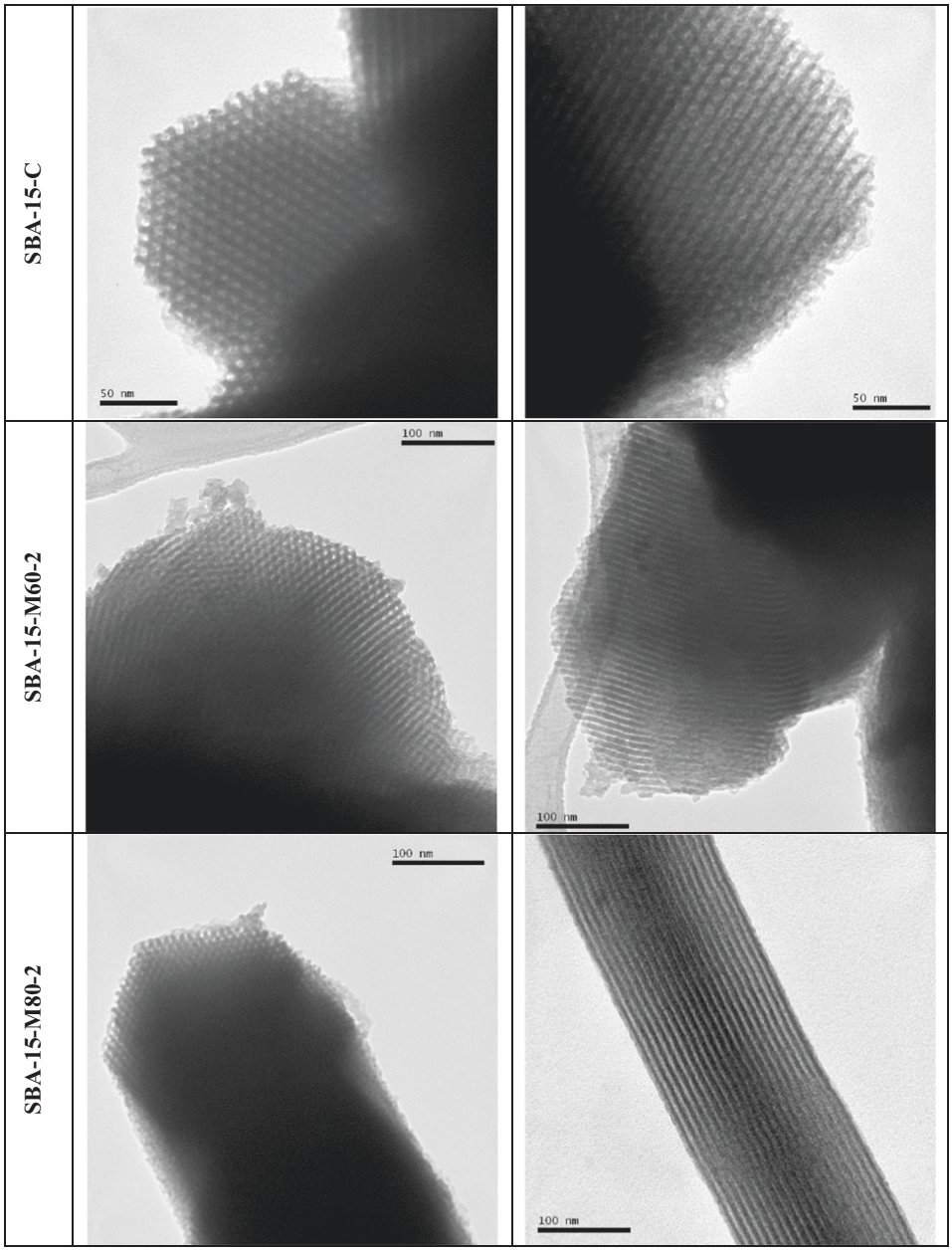

Fig. 6 TEM images acquired at 80 kV for the autoclave conventional hydrothermal method and microwave-irradiated (60 and 80 °C—2 h) SBA-15

TGA data are presented in Fig. 8. The main weight losses were observed in the temperature range between 25 and 150 °C and they were attributed to water desorption. In the range from 150 to 550 °C, the weight decrease could be ascribed to the decomposition and desorption of surfactant that remained of the calcination process. According to the literature the degradation temperature of Pluronic® P123 is 350 °C, therefore above 550 °C the mass loss being observed is due to dehydroxylation reactions [43, 44]. In addition, there is a greater mass loss for the silicas synthesized by microwave irradiation (SBA-15-M60-2 and SBA-15-M80-2), where a greater amount of silanol groups on the surface of the materials was observed according to FTIR.

To better understand the effect of the microwaves on the hydrophilicity of the silicas, contact angle analyses were performed. The videos presented in the supplementary material show the interactions of the water droplets with the substrate. Table 4 presents the values of contact angles in the SBA-15 synthesized, where it was verified that it was not possible to measure the angle for the samples synthesized in the microwave reactor at the temperature of 80 °C, as the water droplets dispersed quickly in the substrate (Fig. 9), proving the high amount of silanol and consequently greater hydrophilicity of this material.

In general, it was possible to verify that all the SBA-15 synthesized are hydrophilic (angle < 90°) [45], and that the

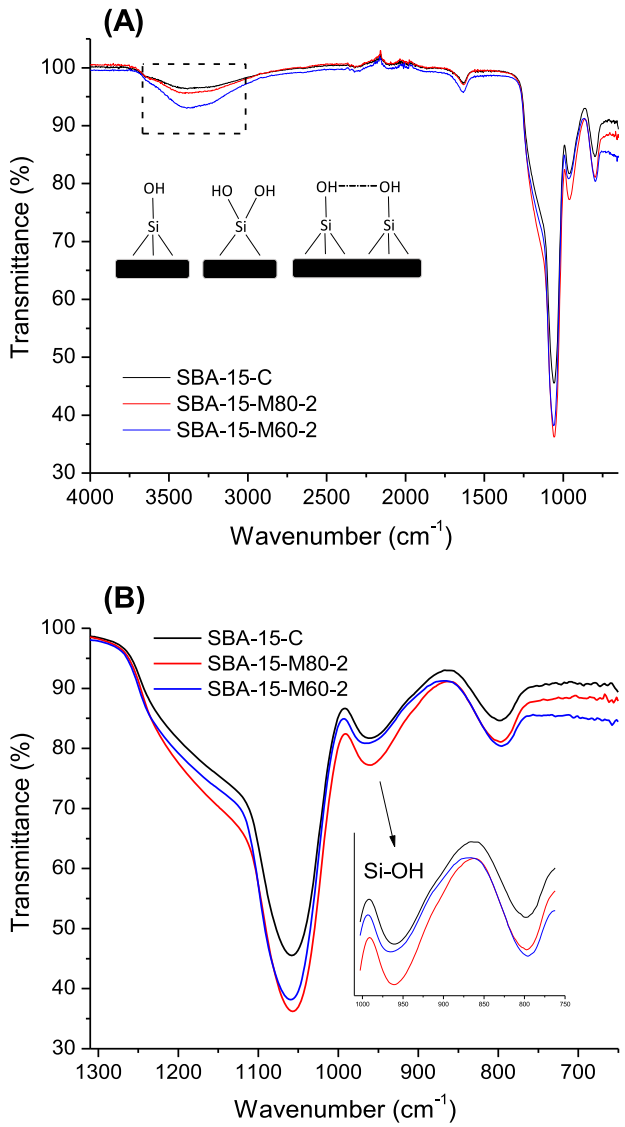

Fig. 7 **a** FTIR spectra of SBA-15 materials synthesized by the autoclave conventional hydrothermal method and microwave-irradiated with emphasis on the region of isolated, germinal, and vicinal silanol groups. **b** Zoom of the FTIR of silanol bands

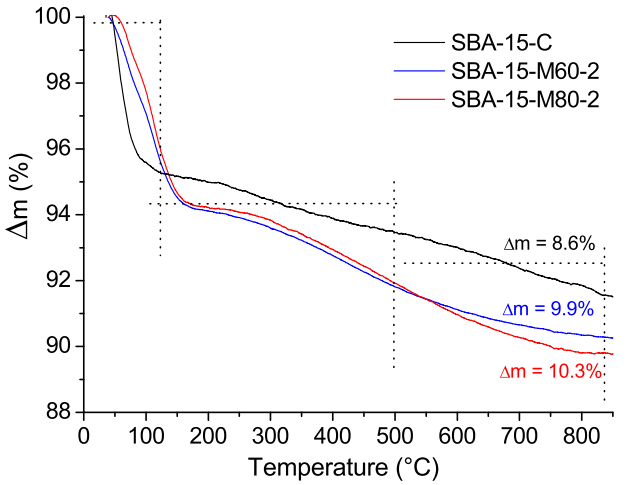

Fig. 8 Thermogravimetric analysis of the autoclave conventional hydrothermal method and microwave-irradiated SBA-15 obtained in the region of 10 °C min^{-1}

Table 4 Contact angle measurements of SBA-15 silicas

Sample	Contact angle (°)
SBA-15-C	53.7 ± 2.3
SBA-15-M60-0.5	13.9 ± 2.8
SBA-15-M60-2	16.2 ± 1.9
SBA-15-M60-8	17.4 ± 2.5
SBA-15-M80-0.5	0
SBA-15-M80-2	0
SBA-15-M80-8	0

SBA-15 synthesized in a microwave reactor has a number of hydroxyl groups on the surface higher than those synthesized in an autoclave, since they are present at a lower contact angle; consequently, these materials are more hydrophilic [46]. These results corroborate with previous analyses (FTIR and TGA) and show a higher amount of silanol on the silicas synthesized by microwave reactor.

4 Conclusions

It was verified that the synthesis assisted by microwave irradiation showed a significant reduction in the time when compared with the conventional autoclave synthesis, without modifying the structural and morphological characteristics of the materials, proving that it is an efficient synthesis for ordered mesoporous materials. The adsorption/desorption isotherms of N_2 and X-ray diffractograms confirmed the characteristic of the SBA-15 silicas, and the transmission electron micrographs showed that the materials synthesized by both autoclave and microwave irradiation hydrothermal methods have well-defined pores and hexagonal channels. In addition, through the analysis of FTIR, TGA, and CA it was found that the materials synthesized by microwave irradiation have a greater amount of hydroxyl groups on the surface than the materials synthesized in autoclave, thus proving a greater hydrophilicity of these silicas. Thus, this study showed that it is possible to control the properties of the material when it is desired to obtain SBA-15 with greater hydrophilicity.

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Fig. 9 Contact angle images of SBA-15 silicas synthesized by the microwave irradiation (60 and 80 °C—2 h) and by the autoclave conventional hydrothermal method

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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