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Enhancement of synthesis of ZSM-11 zeolite by microwave irradiation

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ABSTRACT

A novel and complete description for ZSM-11 zeolite microwave-assisted synthesis is provided. The time required for the synthesis of ZSM-11 zeolite was remarkably reduced down to 3–4 days instead of 14 days under classical conditions, without the use of seed crystals and of any pretreatment step such as aging time. The crystallinity degree of ZSM-11 zeolites synthesized under microwave irradiation is considerably improved (39% higher) with respect to the crystallinity of conventional ZSM-11 zeolites. According to SEM images, the particle sizes of ZSM-11 zeolites synthesized by both methods are identical and a new morphology is observed when microwave irradiation is used. The surface area and microporous volume values for both synthesis methods are similar.

In this work, the synthesis of ZSM-11 zeolite has been successfully intensified by using microwave irradiation. Energy consumption and synthesis time are considerably reduced.

1. Introduction

In the last decades, zeolite materials, both natural and synthetic, have been shown to have excellent catalytic, adsorption and ionic exchange properties [1,2]. Zeolite ZSM-11 (Zeolite Socony Mobil 11), or MEL zeolite (Mobil ELeven), is a high silica zeolite type [3] that was first patented by Chu [4] in 1973 and later reported by Kokotailo et al. [5] in 1978. Its framework structure is very similar to that of ZSM-5 zeolite or MFI zeolite (Mobil FIve). Unlike the MFI structure, ZSM-11 zeolite possesses straight intersecting channels having same elliptical pore size (5.3 Å × 5.4 Å) [5–9]. This property enhances the molecular diffusion, inside the zeolite cavities, by reducing the tortuosity factor. Considerable attention has been devoted to the synthesis of ZSM-11 zeolite because of its catalytic [10–13] and pervaporation [14] properties.

Zeolite ZSM-11 is usually obtained by hydrothermal synthesis using conventional autoclave heating as reported by many authors i.e. Chu [4], Kokotailo et al. [5], Gabelica et al. [15] and Pierella et al. [16–18]. This zeolite synthesis path is a process which considerably consumes energy and which is accomplished in 10–14 days due to weak induction, nucleation and crystallization rates. Thus, there is a clear need to reduce the energy consumption for the synthesis of ZSM-11 zeolite and to enhance its productivity while keeping its physical properties, phase purity, crystallinity and crystal size distribution as the same as zeolites synthesized under the conventional method.

One way presented for reducing the synthesis cost of ZSM-11 zeolite was recently reported by Dey et al. [6]. They used rice husk ash as silica source but they obtained a high ZSM-11 zeolite particle size, between 100 and 150 μm after 12 days of synthesis which is not really an economical process. Another interesting attempt for ZSM-11 low cost synthesis was recently reported by Zhang et al. [19]. They performed an organic template-free synthesis. Thus, the ZSM-11 synthesis cost was successfully diminished but this organic template-free system is only favorable to the aluminum-rich zeolite.

The enhancement of the synthesis of ZSM-11 zeolite is clearly reached when using alternative ways of heating like microwave irradiation. Dielectric heating has become an efficient solution to increase zeolite synthesis rate in the last years. This fact can be attributed to the reduction of three terms in zeolite synthesis: (a) the induction period, (b) the nucleation and (c) crystal growth [20].

The first ZSM-5 zeolite synthesis under microwave irradiation was patented by Chu et al. in 1988 [21]. They affirmed to have provided relatively small crystals at higher rates of production. Nevertheless, the zeolite synthesis was completed in 72 h using seed crystals of the same zeolite and in 6 days without seeding.

The synthesis of several molecular sieves were reported to be enhanced by microwave irradiation as for example: ZSM-5 [20,22–24], AlPO4-5 [25], VPI-5 [26], AlPO4-H [27], Silicalite [28–30], TS-1 [31], TS-2 [32], NaA [33], NaY [34], Beta [35], etc.

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The first patent for the synthesis of ZSM-11 zeolite under microwave irradiation by Liu et al. was published in 2011 [36]. Depending on experimental conditions, the ZSM-11 synthesis was achieved in 5–10 days by using seed crystals of ZSM-11 zeolite and by aging the mixture synthesis during 1–10 days.

Hence, there is still a clear inconvenient to synthesize ZSM-11 zeolites without the use of seed crystals which could contaminate the reaction mixture. Besides, the aging time, used before heating the reaction system, reduces the productivity of ZSM-11 zeolites and increases the energy consumption and synthesis time.

On the other hand, the microwave equipment and synthesis parameters like mixture composition and volume, temperature and pressure during reaction synthesis have rarely been specified in literature. Plus, the zeolite crystal morphology and size distribution are not always presented. The lack of information avoids having a better understanding of molecular sieves synthesis using microwave irradiation.

In this work, we provide a way to synthesize ZSM-11 zeolites without the use of seed crystals (high phase purity and crystallinity) and without aging time of the synthesis mixture. Thus, we will present a full efficient method that highly diminishes the energy consumption and the MEL zeolite synthesis time with the use of microwave irradiation. All synthesis parameters are presented as well as a complete description of microwave synthesis system. The microwave irradiation influence in each step of the zeolite synthesis is discussed and the ZSM-11 morphology and crystals size are compared to those of zeolites synthesized under conventional heating. The specific area values of ZSM-11 zeolites synthesized under microwave irradiation are compared to those of ZSM-11 zeolites prepared under conventional heating.

2. Experimental

2.1. Synthesis of ZSM-11 zeolite

The ZSM-11 zeolite having a Si/Al molar ratio of 17 was obtained by hydrothermal synthesis under both conventional heating and microwave irradiation. Silicon dioxide (anhydrous, particle size 0.5–10 μm, Fluka) was used as silicon source and sodium aluminate (anhydrous, Sigma–Aldrich) as aluminum source. The tetrabutylammonium hydroxide (TBA-OH, ~40% in H_2O ~1.5 M, Sigma–Aldrich) was used as template agent. The synthesis mixture was homogenized under magnetic agitation during 10 min. Its molar composition was: SiO_2:Al_2O_3:Na_2O:$(TBA)_2$:H_2O = 33.9:1:1.25:3.2:700 with a basic pH of 10–11. The synthesis gel was placed in a hermetic PTFE reactor for both conventional and dielectric heating.

In order to perform the conventional synthesis of ZSM-11 zeolite, the reactor was placed into an oven at 150 °C during 14 days.

For dielectric heating synthesis, the reactor of 2.8 cm of internal diameter was put into a WR-340 waveguide inside of which a 2.45 GHz electromagnetic wave was confined having a maximal microwave power of 200 W. The volume of the synthesis gel mixture was 20 ml for each run, and filled 80% of the cavity height. The cavity piston was set in order to have the lowest reflected power and the highest absorbed power inside the reactor. The microwave system for the synthesis of ZSM-11 zeolite is presented in Fig. 1.

The reactor for ZSM-11 synthesis under microwave is also shown in Fig. 1. It is provided with three upper openings: (1) it allows the connection of the pressure sensor for recording and monitoring the pressure throughout the synthesis; (2) it allows depressurization of the reactor in case of emergency. The maximum pressure level was set at 10 bars for each experiment and (3) it allows the input of the optical fibre to monitor and record the temperature throughout the synthesis.

The reaction temperature was set at 150 °C ± 0.3 °C using the Labview ® software to which the pressure sensor and optical fibre are connected. The reactor was regularly manually agitated as done under conventional heating in order to respect identical experimental conditions.

Different synthesis times of ZSM-11 zeolites under microwave were studied. In order to study the influence of dielectric heating in the different steps of the zeolite synthesis (induction, nucleation and crystal growth), both kind of heating, dielectric and conventional, were combined. Thus, the reactor was first heated under microwave irradiation at 150 °C and then it was transferred into an oven under conventional heating at the same temperature. During the time under conventional heating, the reactor was manually agitated every 12 h. The experimental conditions are summarized in Table 1. Run #3 was performed in order to ensure the repeatability of the synthesis of ZSM-11 zeolite under microwave irradiation.

After the reaction, the obtained gel was filtered and washed exhaustively with distilled water, and later with acetone in order to remove part of the template agent. Then, it was dried in an oven at 120 °C during 12 h. Finally, the directing agent was eliminated from the zeolite structure by decomposition under calcination in a muffle at 500 °C during 3 h. After all this process, the Na-ZSM-11 zeolite form was obtained.

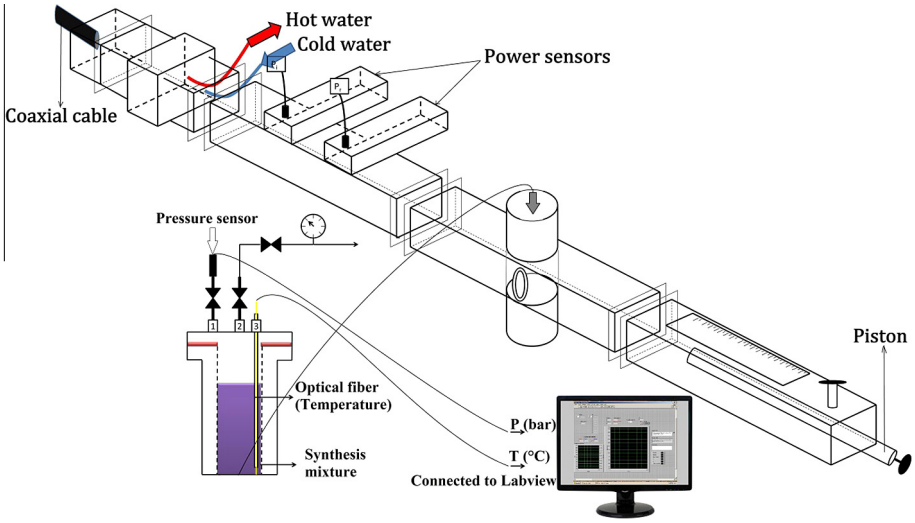

Fig. 1. Microwave system for ZSM-11 synthesis.

Table 1
Experimental conditions for ZSM-11 synthesis under microwave irradiation.

Run #	Time under microwave irradiation (h)	Time under conventional heating (days)
0	0	14
1	4	0
2	4	2
3	4	2
4	4	3
5	2	3
6	1	3

2.2. Zeolite ZSM-11 characterization

2.2.1. Verification of the crystalline structure of ZSM-11 zeolites

The verification of the crystalline structure of microwave-assisted ZSM-11 zeolites was performed by X-ray diffraction technique (XRD) of the ZSM-11 zeolite powders in a Siemens D5000 diffractometer. In all measurements, CoKα radiation having a wave length of 0.1789 nm was used. The diffraction data of the ZSM-11 zeolites were collected between $2\Theta = 5–60^\circ$ in intervals of 0.1° and speed of 2° per min. The samples' volume and mass were kept constant for each X-ray diffraction run.

2.2.2. Determination of ZSM-11 zeolites surface area and micropore volume

The zeolites surface area values of ZSM-11 zeolites were measured from the N_2 adsorption–desorption isotherms using the Brunauer–Emmett–Teller method in a Micromeritics Asap 2400 equipment. Before surface area measurements, zeolites were heated at 500 °C during 6 h under inert atmosphere (70% N_2, 30% He) at a pressure lower than 10^{-3} Pa. The microporous volume determinations were performed by using the Dubinin–Radushkevich equation and the "*t*-plot" method. The mesoporous volume was estimated by subtracting the microporous volume to the one at $p/p_0 = 0.99–0.995$.

2.2.3. Scanning electron microscopy of ZSM-11 zeolites

Zeolites particles observations were performed by a scanning electron microscope Leo 1530-Gemini (Zeiss) with an accelerating voltage of 5 kV, and using the secondary electron Everhart–Thornley detector. Beforehand, samples are covered with 20 nm of gold layer using the sputter-coater 208HR (Cressington).

3. Results and discussion

3.1. Synthesis of ZSM-11 zeolites under conventional and dielectric heating

The synthesis of ZSM-11 zeolite was carried out by combining both conventional and dielectric heating, as proposed by other authors [37,38], in order to exhibit the microwave energy effect in the synthesis process. The XRD patterns of each ZSM-11 zeolite, synthesized under the experimental conditions presented in Table 1, are shown in Fig. 2. The typical XRD patterns of crystalline ZSM-11 zeolites present two peaks between 9 and 11° and also two peaks between 26 and 28° of 2Θ, depending of radiation type. All experimental runs presented a crystalline ZSM-11 zeolite XRD pattern except the Run #1. The XRD pattern of this experimental run corresponds to a typical amorphous phase.

The crystallinity degrees of ZSM-11 zeolite were calculated with background subtraction, for all the experimental runs, by comparing the relative intensities of the main peak that is to say, the one at $2\Theta = 27^\circ$, from X-ray diffraction patterns [19]. The ZSM-11 zeolite fully synthesized under conventional heating was designated as standard and a relative crystallinity of 100% was attributed to this zeolite because it was our reference in our previous works [39,40].

In all experiments the piston position has been tuned in order to maximize the absorbed power (75–80 W) and to minimize the reflected one (19–21 W). Identical microwave power profiles were observed for all runs.

By comparing the Run #0 to all the others, the synthesis time is much shorter when microwave irradiation is used (3 days in total against 14 days for a full synthesis under conventional heating). Besides, the Runs #4 and #5 demonstrate that a high crystallinity degree can be reached when microwave energy is used. The crystallinity is even 39% higher than the one obtained after 14 days under conventional heating (Run #0).

The crystalline structure of ZSM-11 zeolite was obtained for all experiments, except for the run 1 which was stopped after 4 h under microwave heating without conventional heating treatment. It can be therefore deduced that this protocol, combining a minimum time of 2–4 h under microwave at 150 °C followed by a conventional heating treatment at the same temperature during 2–3 days allows the production of the ZSM-11 zeolite in its crystalline

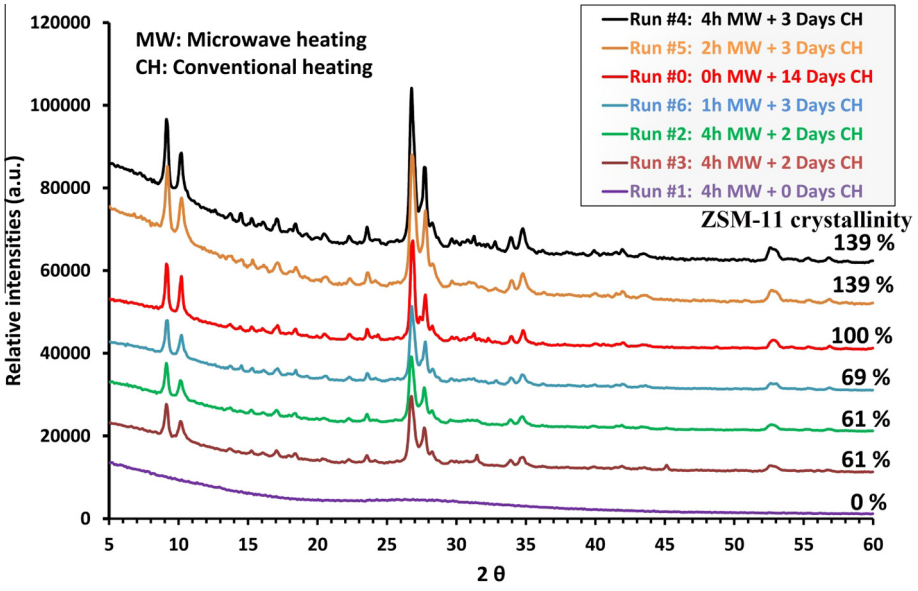

Fig. 2. XRD patterns of ZSM-11 zeolites.

form. This original process reduces from 14 to 2–3 days the synthesis time of ZSM-11 zeolite without using seed crystals and any aging time before synthesis. Thus, this way of ZSM-11 synthesis presents a clear advantage, both in productivity and in energy saving. For both conventional and microwave-assisted synthesis, the ZSM-11 crystalline zeolite yield is 5 grams for each experimental run. The reproducibility of the runs has been checked. For the sake of simplicity only the Runs #2 and #3 are presented in this work.

These results show the importance of both the time under microwave irradiation and the time under conventional heating as well. The highest crystallinity is obtained with a minimum of 2 h at 150 °C under microwave irradiation followed by 3 days under conventional heating in an oven (Run #5). One hour under microwave heating followed by 3 days in a conventional oven (Run #6) allows to get the ZSM-11 zeolite but with a low rate of crystallization (69%). So, this comparison proves that microwave heating has a significant effect on the induction and/or nucleation period, which take place from the beginning of the process.

By considering Run #1, 4 h only under microwave irradiation do not allow obtaining a ZSM-11 crystalline zeolite. The crystallization process does not seem to be initiated since the Run #1 XRD pattern is typical of an amorphous phase. This fact shows that a minimum of time is required for the crystal growth to happen, which remains a quite slow phenomenon even if the induction and nucleation rates have been accelerated.

As a conclusion, five to ten grams of ZSM-11 zeolite can be synthesized in a significantly shorter time, compared to conventional heating, by microwave irradiation between 1 and 4 h, followed by conventional heating between 1 and 3 days at 150 °C.

Phenomena involved during the microwave heating period can be of several types and converge toward the intensification of the process. First, the synthesis temperature (150 °C) is reached much more quickly inside the waveguide than in a conventional oven, as shown on the temperature and pressure profiles recorded during the experimental runs [Fig. 3]. The full hours indicated in Table 1, for the microwave irradiation period, were strictly chronometrically controlled. Nevertheless, in Fig. 3, the hours indicated on the abscissae does not necessary coincide to the full hours of Table 1 since the software has also recorded some periods of time which are not considered as experimental time. Theses death periods of time are composed of the time that the reaction mixture needs to reach the synthesis temperature (150 °C) and

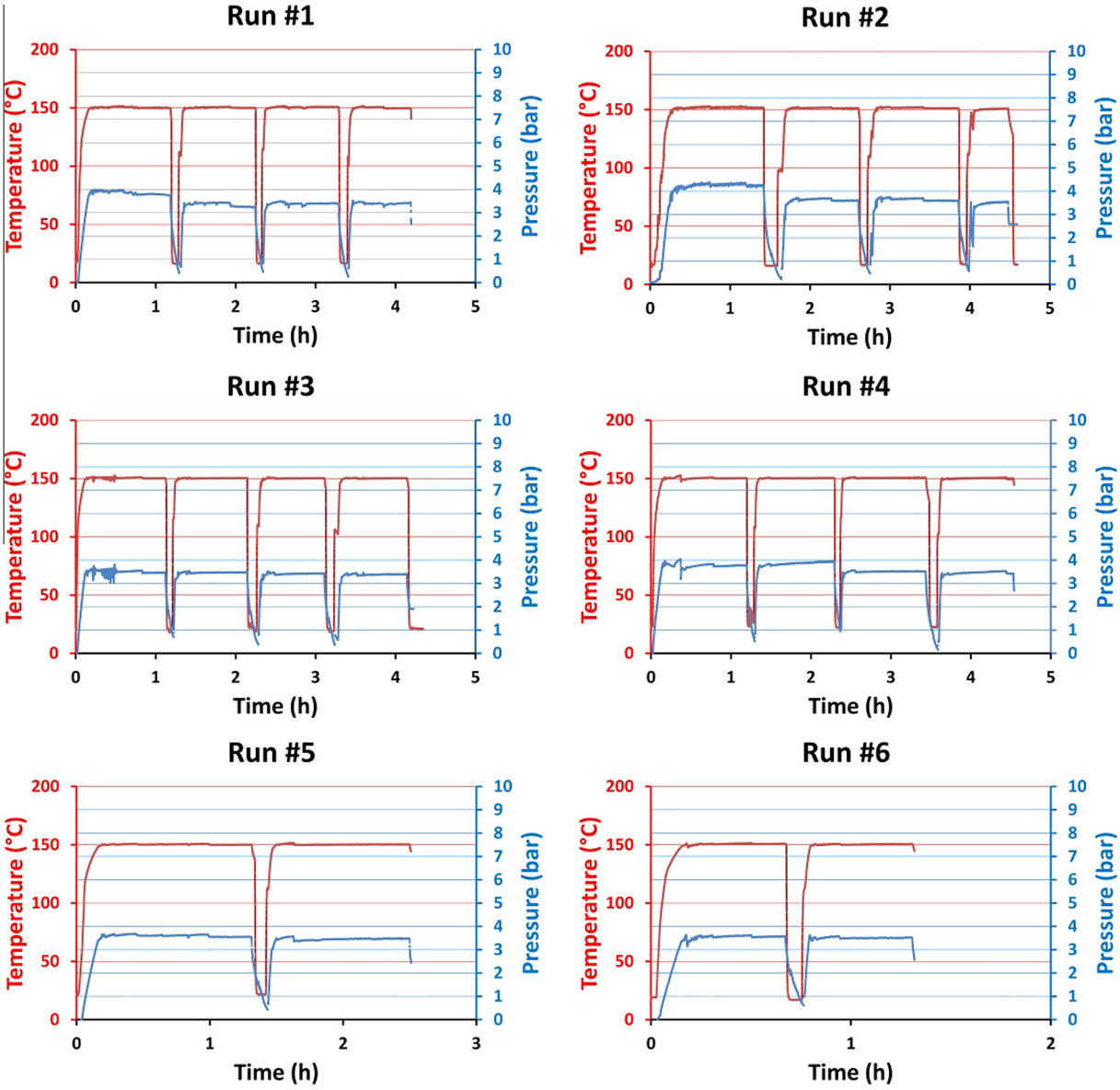

Fig. 3. Pressure and temperature profiles during the synthesis of ZSM-11 zeolite.

the pauses that were performed in order to stir the synthesis solution.

The self-generated pressure inside the hermetic reactor was of 4 bars for all the experimental runs. Only 4–5 min are needed to reach the synthesis temperature under microwaves while several hours are required in conventional heating. Thus, the thermal lag time is essentially eliminated under microwave irradiation. In Fig. 3, the curves discontinuity corresponds to microwave power cut off for reactor stirring purpose. The initial synthesis gel mixture of ZSM-11 zeolites possesses high dielectric properties due to water presence in solution and to the strong ionic character of the mixture that leads to a high conductivity. Thus, when synthesis is started under microwave irradiation, the synthesis gel heats very efficiently and quickly, while classical heating is governed by the slow convection phenomena. Therefore, the microwave energy increases the rate of heat of the synthesis mixture, increasing the synthesis rate, as reported by authors in the last decades [41].

Second, induction and nucleation start. It is thought that the rapid microwave mode of heating significantly reduces the induction time and increases nutrient dissolution and nucleation rate, which are the limiting steps in the synthesis of zeolites as claimed by several authors [42–44].

Another important effect which is present, when microwave energy is used, is that microwave energy enhances the dissolution of the precursor gel [45,46] also increasing the nucleation rate. The solubility of precursor synthesis gel is highly favored diminishing the induction period. Plus, the nucleation step is also favored since the high temperatures provoke a diminution of the local values of viscosity and surface tension.

In the next step, when the crystals grow in pseudo-steady state conditions, no significant difference between conventional heating and dielectric heating is supposed to be observed according to the literature [47]. The induction and nucleation periods are the slowest steps of the zeolites synthesis process. So, crystallization could not start unless the slow reagent digestion step and nuclei formation were completed [20]. The main microwave effect in zeolite synthesis derives from the acceleration of precursor digestion of the reaction mixture. Crystal growth process is not essentially different under both conventional and dielectric heating.

3.2. Morphology of ZSM-11 zeolites synthesized under conventional and dielectric heating

According to the SEM images [Fig. 4], the morphology of all ZSM-11 zeolites (except run #1 having amorphous phase) presents prismatic-like crystals which is characteristic of ZSM-11 zeolites. The particle size is similar for all zeolites. It is between 200 and 400 nm.

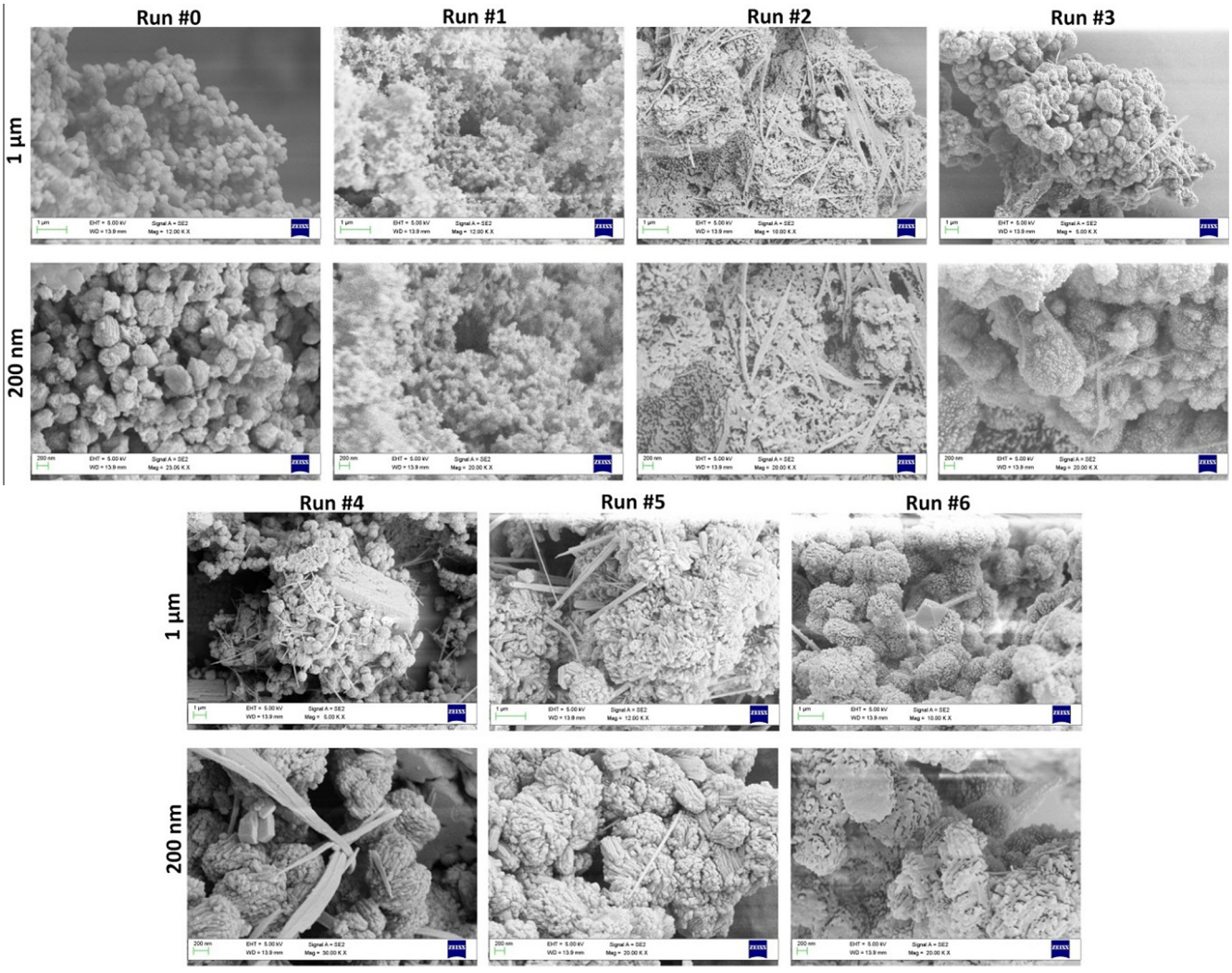

Fig. 4. SEM images of ZSM-11 zeolites synthesized under classical and dielectric heating.

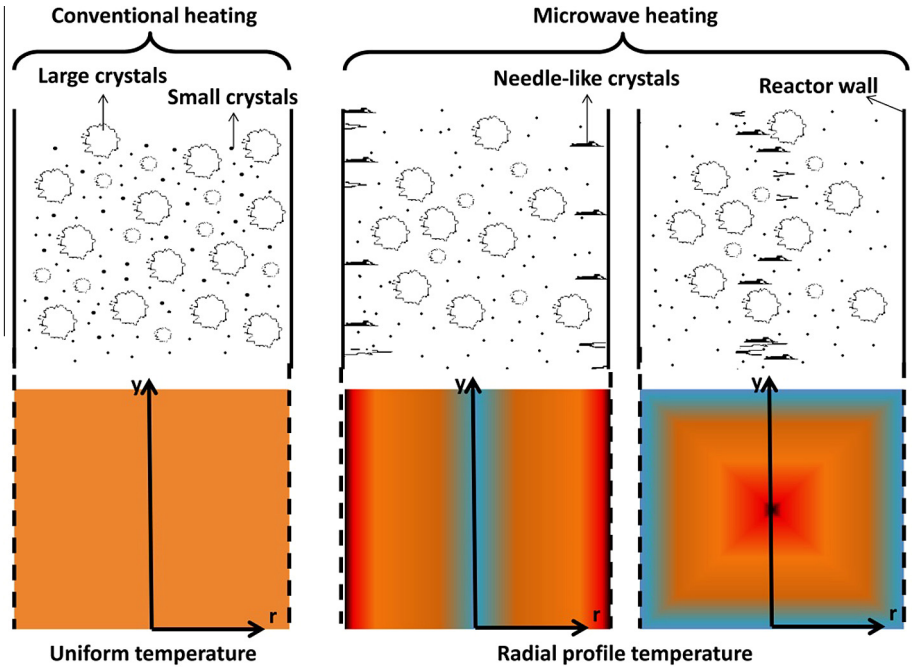

Fig. 5. Potential temperature profile inside the synthesis mixture through the transversal cross section.

Table 2
Surface area and porosity characterization of ZSM-11 zeolites.

	Run 0	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6
BET surface area [$m^2 g^{-1}$]	385	14	307	240	330	371	305
Volume at p/p_0 max. [$cm^3 g^{-1}$]	0.253	0.067	0.204	0.176	0.207	0.178	0.163
p/p_0 max.	0.991	0.990	0.992	0.992	0.995	0.998	0.993
Microporous surface area by *t*-plot [$m^2 g^{-1}$]	346	10	283	221	276	355	294
External surface area [$m^2 g^{-1}$]	39	4	23	19	54	17	11
Microporous volume by *t*-plot [$cm^3 g^{-1}$]	0.134	0.004	0.108	0.094	0.116	0.135	0.110
Diameter at BJH adsorption volume/2 [nm]	47.8	108.8	62.6	43.9	23.4	43.0	57.3
Diameter at BJH desorption volume/2 [nm]	31.7	68.4	43.2	31.8	9.3	65.2	46.2
Diameter at d*V*/d*D* max. BJH adsorption [nm]	3.7	4.0	3.9	1.8	1.8	2.1	1.8
Diameter at d*V*/d*D* max. BJH desorption [nm]	3.7	3.3	3.5	3.7	3.6	3.6	3.8
Dubinin surface BET [$m^2 g^{-1}$]	361.5	14.4	282.9	246.8	278.4	345.1	278.4
Dubinin volume [$cm^3 g^{-1}$]	0.166	0	0.127	0.103	0.116	0.144	0.116
Liquid volume ($p/p_0 = 0.10$) [$cm^3 g^{-1}$]	–	0	–	0.101	0.114	–	0.114
Liquid volume ($p/p_0 = 0.95$) [$cm^3 g^{-1}$]	0.201	0.020	0.153	0.139	0.135	0.260	0.135

Microwave irradiation also gave rise to a new kind of morphology in ZSM-11 zeolites which was not observed when the synthesis was carried out under conventional heating (Run #0). In microwave-assisted synthesis, a needle-like or ribbon-like morphology was found as shown in Fig. 4 (Run #2 to #6).

The needle-like morphology was also observed in the synthesis of several molecular sieves under microwave irradiation like VPI-5 [26], $AlPO_4$-5 [25]. This kind of morphology is also typical of the ZSM-23 zeolites. The ZSM-23 zeolite architecture results in one-dimensional channels running parallel to the growth direction of the needles [48]. It has been claimed that the presence of needle-like morphology in the SSZ-51 reinforces the purity of the crystalline phase [49].

In order to explain the presence of the new morphology in ZSM-11 zeolites synthetized under microwave irradiation, the presence of thermal gradients can be quoted. When microwave energy is used, the temperature profile in the synthesis mixture is likely non-uniform since that several hot spots can exist. This fact is directly linked to the electric field shape inside the reactor as also mentioned by Cundy et al. [20]. Depending on the microwave tuning and the dielectric properties of the heterogeneous mixture, the electric field is concentrated in the mixture hot spots which create a radial temperature profile from them. The electric field could have a maximum next to the reactor walls or in the center of the reactor as illustrated in Fig. 5. The radial temperature profile having its center in the hot zones allows the crystal growing in a radial way. The solubility of precursor synthesis gel is highly favored in these zones diminishing the induction period. Plus, the nucleation step is also favored since the high temperatures provoke a diminution of the local values of viscosity and surface tension. Thus, the zeolite crystallization and nuclei growth take place in a radial way by forming a needle-like morphology in ZSM-11 zeolites from the hot spots zones. Natural convection probably occurs, leading to a partial homogenization of the mixture but it is likely that "dead zones" are still remaining at the bottom of the reactor or in the corners.

As already mentioned in the experimental protocol (part 2.1), the reactor was manually agitated during the time under conventional heating every each 12 h. This regular agitation provokes the re-homogenization of the medium, favoring the crystal growth

in a prismatic manner and partially breaking the formed needles. Finally, the resulting ZSM-11 zeolites present both kind of morphology as shown on the SEM images.

It is likely that the difference of morphology between ZSM-11 zeolites synthesized under classical and dielectric heating will have an important impact on adsorption, ion exchange and catalytic properties. It is then necessary to compare the surface area values of ZSM-11 zeolites synthesized using the two methods.

3.3. Surface area and microporous volume of ZSM-11 zeolites synthesized under conventional and dielectric heating

The zeolites surface area values together with microporous volumes of ZSM-11 zeolites are presented in Table 2.

Surface area values of ZSM-11 zeolites synthesized under microwave irradiation are similar to those of the ZSM-11 zeolite fully prepared under conventional heating, except for the run 1 (amorphous zeolite). The surface area of ZSM-11 zeolite for the run #5, having the higher crystallinity, is only 3% lower than the one obtained under full conventional heating. The microporous volume values and the pore diameters distribution estimated by the BJH method are also similar. This indicates that the new microwave-assisted synthetized zeolites are likely to possess similar properties as catalysts, adsorbents and ion exchangers.

4. Conclusions

In this work, we provide a complete description for ZSM-11 zeolite microwave-assisted synthesis. The time required for the synthesis of ZSM-11 zeolite is remarkably reduced down to 3–4 days instead of 14 days under classical conditions, without the use of seed crystals and of any pretreatment step such as aging time. This original process starts the reaction under dielectric heating in order to shorten the induction and nucleation times and goes on under classical heating during 3–4 days for completing the crystal growth process. Plus, the crystallinity degree of ZSM-11 zeolites synthesized under microwave irradiation was considerably enhanced (39% higher) with respect to the crystallinity of conventional ZSM-11 zeolites. The particle sizes of ZSM-11 zeolites synthesized by both methods are identical. Pore volume is also similar for all the cases. A high crystalline and pure ZSM-11 zeolite can be successfully synthesized by heating under microwaves the gel mixture during 2 h followed by 3 days under conventional heating.

Microwave energy increases the induction and nucleation rates because it enhances the dissolution of the gel precursors principally in some zones of the reaction mixture where hot zones and thermal gradients are generated. It is likely that the electric field shape inside the synthesis gel establishes a radial temperature profile favoring the presence of a new kind of ZSM-11 zeolite morphology. The needle-like morphology was only observed when microwave energy was used. The new morphology does not affect ZSM-11 zeolites main properties since the surface area, microporous volume and pore diameter values are similar to those of ZSM-11 zeolites synthesized in a classical way.

The synthesis of ZSM-11 zeolite has then been intensified by using microwave irradiation. Energy consumption and synthesis time has been remarkable reduced.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.micromeso.2014.07.006.

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