Effect of variations in parameters on the crystallization of mordenite zeolite

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Abstract:

Introduction/purpose: This study highlights the importance of synthesis parameters in the crystallization of mordenite zeolite. Precise control of the SiO_2/Al_2O_3 ratio, alkalinity and crystallization time results in mordenite crystals with high crystallinity and optimum purity, while avoiding the formation of secondary or amorphous phases.

Methods: The materials were prepared by the hydrothermal method, using silica gel and sodium aluminate as sources of silicon and aluminum, respectively. Several synthesis parameters were varied, including SiO_2/Al_2O_3 molar ratio, alkalinity (OH^-/Si), as well as crystallization time, in order to assess their effect on mordenite crystal formation. Experiments were carried out at a constant temperature of 170°C and 190°C.

Results: The results show that the SiO_2/Al_2O_3 ratio plays a crucial role in crystal formation. A low ratio, such as 15, combined with high alkalinity, favors the formation of analcime crystals. On the other hand, a high ratio, such as 30, leads to the formation of mordenite crystals with high crystallinity and purity. However, when this ratio reaches 60 and is combined with low alkalinity, crystalline nucleation is impeded, leading to the formation of an amorphous material.

Concerning alkalinity (OH^-/Si), the values of 0.39 and 0.49 result in pure, well-crystallized mordenite crystals, while higher values, such as 0.59, lead to the formation of secondary phases. With regard to crystallization time,

the periods of 48 and 72 hours at 170°C produced pure, well-crystallized mordenite crystals.

Conclusion: This study highlights the importance of synthesis parameters in the crystallization of mordenite zeolite. Precise control of the SiO_2/Al_2O_3 ratio, alkalinity and crystallization time results in mordenite crystals with high crystallinity and optimum purity, while avoiding the formation of secondary or amorphous phases.

Key words: mordenite, alkalinity, Si/Al ratio, crystallization, zeolite.

Introduction

Zeolites are crystalline microporous alumina-silicate materials used in a wide range of practical applications. They belong to the silicate group, the tectosilicate subgroup (M. B. Z. Gili and Conato, 2019; Pérez-Botella et al, 2022), and show remarkable properties and performance in domestic, medical, and industrial fields such as catalysis, separation, adsorption, and ion exchange (Jia et al, 2019; Zhang et al, 2011). These characteristics encourage scientific inquiry into the cost-effective production of zeolites.

The shape and size of crystals strongly influence the properties of zeolites. It is therefore crucial to study the influence of synthesis parameters on zeolitic morphology in order to optimize their use in various applications. Controlling the size and morphology of mordenite crystals is currently the focus of much research (Bolshakov et al, 2019; Gili and Conato, 2018; Khalil and Muraza, 2016; Mohamed et al, 2005). Various sources, such as silica gel, rice ball ash, and smoked silica, are used in some works to prepare mordenite-type zeolites, while metakaolin or faujasite zeolite are used as sources of aluminium (Narayanan et al, 2021). Other studies have focused on the optimization of the mordenite synthesis parameters by microwave heating (Khalil and Muraza, 2016; Li et al, 2011), and the use of mono-quaternary ammonium to synthesize hierarchical mordanite monotiges (Bolshakov et al, 2019).

Zeolites have important characteristics such as high cationic exchange capacity. Mordenite is one of a few zeolitic structures that is useful in industry, especially in petrochemistry where it is used as a catalyst for breaking down hydrocarbons, alkylating benzene, reforming, and deparaffinating (Bajpai, 1986). It is also used as an isomerization catalyst for naphta C5/C6 to increase their bonding and improve the octane index of gasoline. Mordenite also plays a significant role in the adsorption of various pollutants, including heavy metals and radioactive materials (Gili and Conato, 2018), and it serves as a selective adsorbent in the production of oxygen enriched air. It offers better nitrogen adsorption

than oxygen because of a quadripolar interaction between the nitrogen molecule and the surface of the polar zeolite (Golden and Jenkins, 1981). Additionally, mordenite has shown potential in the catalytic conversion of methane to methanol under mild conditions, leveraging its unique acidic and structural properties to facilitate this transformation, which is a critical step toward utilizing methane as a sustainable chemical feedstock (Brezicki et al, 2021; Le et al, 2017).

Mordenite has an orthorhombic structure with a unit cell a = 18.121 Å, b = 20.517 Å, and c = 7.544 Å belonging to the spatial group Cmcm (M. B. Z. Gili and Conato, 2019). The mordenite structure consists of parallel rings with 12 limbs (MR) (6.5 * 7.0 Å) and 8-MR (2.6 * 5.7 Å) along the c axis (Mohamed et al, 2006), interconnected by 8-MR channels (3.4 * 4.8 Å) across the b axis (Nasser et al, 2016; Zhang et al, 2011).

While natural mordenite exists, some applications prefer synthetic mordenite because of its purity and controlled porous structure (Kordala and Wyszkowski, 2024). Natural mordenite crystals typically appear in the form of needles with an elongation in c (Borissenko, n.d.; Hamidi et al, n.d.), and sometimes are thin in the direction [010] (Hamidi et al, n.d.). Synthesis conditions and post-synthesis treatments have a significant impact on mordenite morphology, size, and structural defects (Borissenko, n.d.). Gels with a SiO_2/Al_2O_3 ratio of 8 to 12 and a Na/Al ratio lower than 3 yield to acicular crystals, whereas increasing gel alkalinity and reducing the amount of aluminum yields prism crystals (Borissenko, n.d.). Hamidi et *al*. discovered that dissolving silica gel before mixing it with other reagents can yield short stick-shaped mordenite crystals (60 nm to 240 nm).

Mordenites obtained by direct hydrothermal synthesis typically have a SiO_2/Al_2O_3 ratio between 10 and 20. One can use an organic template to achieve a higher ratio, but this method is costly and carries risks of contamination and pollution. Direct hydrothermal synthesis therefore remains the best option for controlling the ratio of mordenite-type zeolites (Gili and Conato, 2018; Li et al, 2023; Lima et al, 2023). We observe a notable correlation between the alkalinity of the synthetic solution and the composition of zeolite, where a higher alkalinity facilitates the incorporation of aluminium (Zhang et al, 2011).

In this study, we synthesized mordenite-type zeolites with various parameters using the hydrothermal method, without the use of organic dimensions. We have evaluated the synthetic products in terms of composition, crystallinity, morphology, and grain size using various characterization techniques.

Experimental

Materials

The reagents used in this study are silica gel, sodium aluminate (Haen Riedel), sodium hydroxide (>98%, Prolabo) and demineralized water. A gel was prepared following a precise sequence: 55 cm^3 demineralized water, sodium hydroxide, 1.034 g sodium aluminate, and silica added progressively. The resulting gel was aged under magnetic stirring at 600 rpm for 120 minutes, then transferred to a stainless steel autoclave for hydrothermal treatment. The temperature was raised to 170°C /190°C, then maintained at this level for varying lengths of time. The solid material was then rapidly cooled, filtered, washed to a pH of 9.00 or less, and dried at 80°C. Several parameters were investigated to improve crystallinity and reduce the mordenite crystal size, including the impact of the Na_2O/SiO_2 ratio, the SiO_2/Al_2O_3 ratios, and the crystallization time.

Effect of the Na_2O/SiO_2 ratio

The variation in relative proportions (x) of Na_2O was explored while keeping the quantities of the other components constant. We modified the mixture's composition using the following formula:

$xNa_2O; Al_2O_3; ySiO_2; 560H_2O$

The molar x/y ratios were adjusted between 0.23 and 0.33, or the OH^-/Si ratios between 0.39 and 0.59, respectively. We carried out the crystallization at 190 °C for a duration of 24 hours.

Effect of the SiO_2/Al_2O_3 ratio

Variations in the SiO_2/Al_2O_3 molar ratio were analyzed by adjusting the ratios according to:

$$7Na_2O; yAl_2O_3; xSiO_2; 560H_2O$$

with $15 \leq x/y \leq 60$.

The crystallization was carried out at 190 °C for a duration of 24 hours.

Effect of the crystallization time

The effect of the crystallization time was evaluated by varying the synthesis time of mordenite between 48 and 72 hours, while maintaining a constant temperature of 170 °C during batch sample preparation.

Characterizations

To find out what kinds of crystals were in the samples, we used X-ray diffraction (XRD) on a Malvern Panalytical diffractometer with Cu Kα (40 kV, 15 mA). Analyses were carried out at a scan speed of 5°/min, over a range from 4 to 50°, at a wavelength of 1.54 Å and a step size of 0.02°. The surface morphology was observed using a scanning electron microscope (SEM) in conjunction with a focused ion beam (Scios 2 Dual Beam). The infrared spectra of zeolites were obtained using Fourier transform infrared spectroscopy (FTIR) and an INVENIO spectrometer.

Results and discussion

Effect of alkalinity

Structure and crystallinity

Alkalinity, defined as the concentration of hydroxide ions (OH^-) in the gel solution, plays a decisive role in zeolite purity, morphology and crystallinity. The results of the X-ray diffraction (XRD) analyses shown in Figure 1 illustrate the effects of variations in the OH^-/Si ratio (between 0.39 and 0.59) on the crystalline phase obtained. An OH^-/Si ratio of 0.39 (S1) favors the formation of pure, well-crystallized mordenite. However, as this ratio increases to 0.49 (S2), a slight decrease in peak intensity is observed. An even higher ratio (0.59) (S3) leads to the formation of another thermodynamically more stable zeolitic phase, identified as analcime.

Alkalinity control is crucial to the crystallization process. At an alkalinity ratio (OH^-/Si =0.39) (S1), pure, well-crystallized mordenite is obtained and crystal growth conditions are optimal.

However, when alkalinity exceeds certain thresholds (OH^-/Si =0.49), an increase in nucleation rate is observed, generating a large number of small nuclei. The latter, competing for Si and Al nutrients, limit individual crystal growth, resulting in the reduction in the crystal size (see Table 1).

High alkalinity (OH^-/Si =0.59) also affects the stability of nuclei over a prolonged period and favours the appearance of secondary phases (Hamidi et al, 2011). In addition, it influences the formation of hydroxyl groups on the mordenite surface, thus modifying its reactive and catalytic properties (Limousy et al, 2013). Crystallization kinetics as well as the final phase obtained are strongly influenced by the key parameters such as the OH^-/Si and H_2O/SiO_2 ratios (Larlus and Valtchev, 2004; Zhang et al, 2018)

Table 1 – Effect of varying parameters on the crystal size, the grain size and the crystal phases.

sample	Synthesis condition					Product characterization					
	Composition (molar ratio)			Crystallization time (h)	Crystallization temperature (°C)	Crystal phase [a]	SiO_2/Al_2O_3 (molar ratio)	Crystallite size [b] (nm)	Grain length a(μm)[c]	Grain width b(μm)[c]	Grain height c(μm)[c]
	OH^-/Si	SiO_2/Al_2O_3	H_2O/SiO_2								
1	0.39	30	18.66	24	190	MOR	10.6	43.11	11	5.57	13.07
2	0.49	30	18.66	24	190	MOR	7.8	38.45	8.86	5.21	13.93
3	0.59	30	18.66	24	190	ANA/ MOR	21.2	37.03	-	-	-
4	0.93	15	37.33	24	190	ANA	4.8	41.49	-	-	-
5	0.19	60	9.33	24	190	Amorph	-	-	-	-	-
6	0.39	30	18.66	48	170	MOR	10.48	28.81	1.4	0.73	0.67
7	0.39	30	18.66	72	170	MOR	9.62	30.48	1.35	0.71	0.48

XRD patterns are used to identify the crystal phases (a); MOR, ANA, and Amorph stand for mordenite, analcime, and amorphism, respectively. (b)Shows the crystal size for each sample, calculated using the Scherrer equation. (c)Average dimensions of thirty grains selected from the SEM image, classified according to length, width and height.

Vibrational groups

Figure 2 presents the Fourier Transform Infrared Spectroscopy (FTIR) results of the synthetic products at different alkalinity ratios. For OH^-/Si (0.39), the significant bands are as follows. Large bands at 3339.52 and 3240.43 cm^{-1} are due to the asymmetrical stretching vibration of isolated silanol groups (Si-OH) and the stretching of the absorbed water molecules onto the solid surface (Hincapie et al, 2004). A narrow band of low intensity is centered at 1645.03 cm^{-1} which is attributed to the flexion vibration of residual water molecules (H-O-H) in the zeolite voids (Aloulou et al, 2017; Gili and Conato, 2018). The absorption band at 1185.45 cm^{-1} represents the external asymmetrical stretch vibration between the zeolitic SiO_4 and AlO_4 tetrahedra (De Macedo et al, 2004; Gili and Conato, 2018). The band at 1001.50 cm^{-1} corresponds to the asymmetrical stretch vibration of the tetrahedric bonds (TO), while the low intensity bands at 807.65 cm^{-1} and 767.07 cm^{-1} refer to the symmetric stretch of the internal tetrahedral. The thin bands at 700.62 and 629.60 cm^{-1} represent the symmetrical stretching vibration of the external bonds (Aloulou et al, 2017). A centered band at 552.40 cm^{-1} corresponds to the vibration of the five-chain rings of the tetrahedral (V. Rahbari et al, 2017). The results confirm the presence of zeolitic and/or aluminosilicate materials.

On the other hand, the FTIR spectra associated with the alkalinity OH^-/Si (0.49) appear to show a strong analogy with those for OH^-/Si (0.39), except that the absorption bands for the hydroxyl group (-OH) and the bending vibrations of water molecules (H-O-H) show lower intensities, suggesting a reduction in moisture content (M. Gili and Conato, 2019). The band at 1000.17 cm^{-1} corresponds to the asymmetric stretching vibration of tetrahedral bonds (TO) and becomes more pronounced with increasing alkalinity. This feature is specific to amorphous aluminosilicates, where the tetrahedra have the freedom to vibrate without a specific constraint, unlike crystalline zeolites, which feature a regular structure, thus limiting certain tetrahedra vibrations due to their confinement within a fixed lattice or framework (Gili and Conato, 2018).

Seen from another angle, the FTIR spectra of OH^-/Si (0.59) appear closely similar to those of OH^-/Si (0.39), with the exception of the intensification of all absorption bands, suggesting the presence of a different type of zeolite to that observed for OH^-/Si (0.39) and OH^-/Si (0.49). However, the band at 1027.61 cm^{-1}, corresponding to the asymmetric stretching vibration of the tetrahedral (TO) bonds, remains unchanged, indicating the persistence of a regular structure.

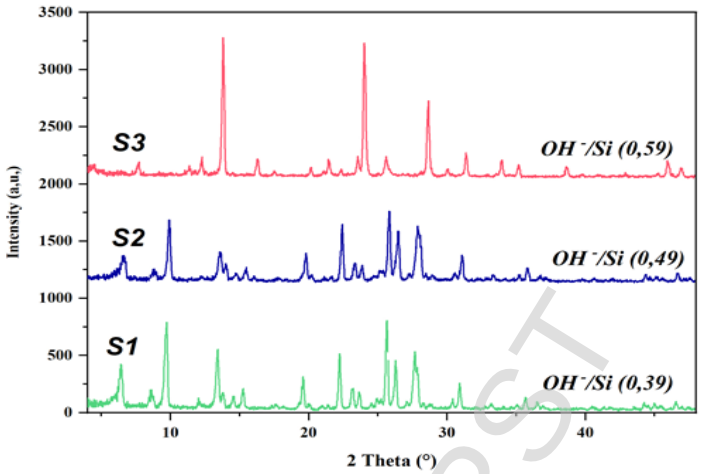


Figure 1 – XRD models of the synthesized MOR samples with various alkalinity ratios

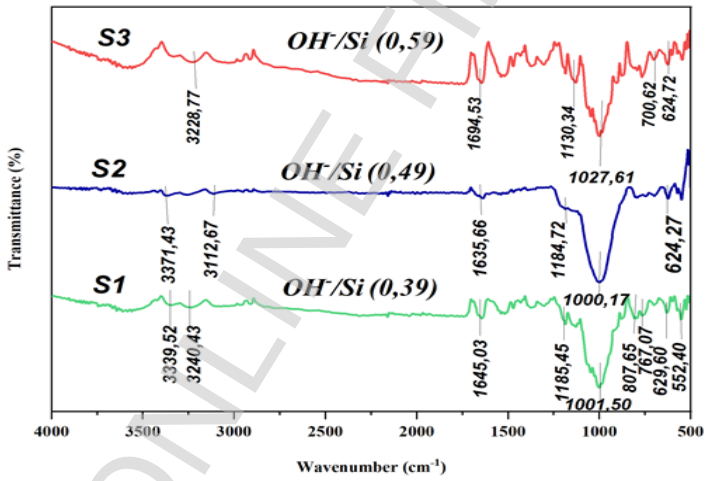


Figure 2 – FTIR spectra of the MOR samples synthesized with different alkalinity ratios

Morphology

Figures 3a to 3c show the SEM images of the synthetic powder products with different alkalinity ratios.

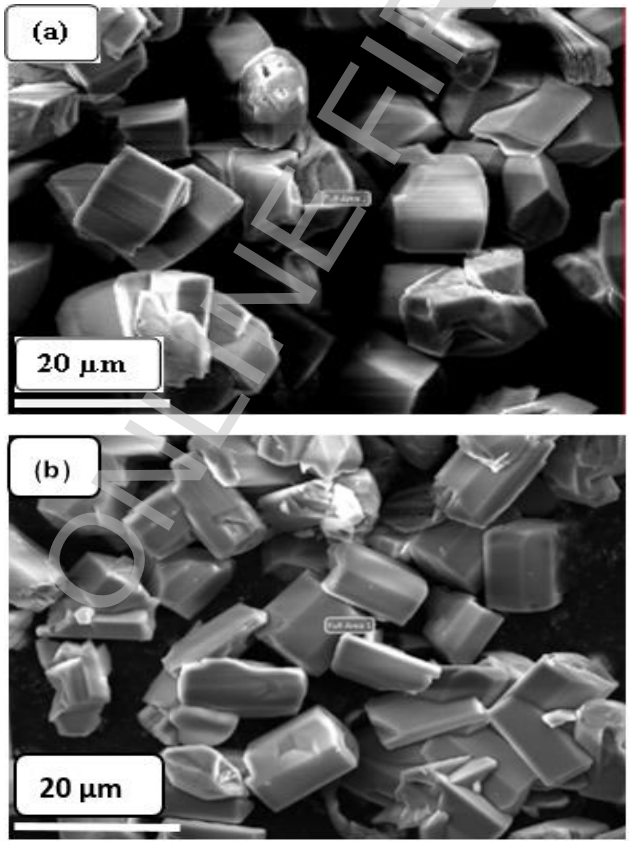
The synthesized sample with an OH^-/Si ratio of 0.39 (Figure 3a) consists of grains with clearly defined linear edges. A significant proportion of these grains have a hexagonal shape, although most are irregular in size and morphology. Grain crystals measure on average:

Length: 11 μm,

Width: approximately 5.57 µm, and
Height: about 13.07 µm.

The synthesized sample with an OH^-/Si ratio of 0.49 (Figure 3b) is characterized by grains of irregular shape. The bulk grains show a hexagonal configuration with average dimensions:
Length: 8.857 µm,
Width: about 5.21 µm, and
Height: about 13.93 µm.

The synthetic powder with an OH^-/Si ratio of 0.59 (Figure 3c) is composed of spherical-shaped grains, corresponding to the analcime phase. Some grains appear fragmented or damaged due to very high alkalinity, producing amorphous mordenite.



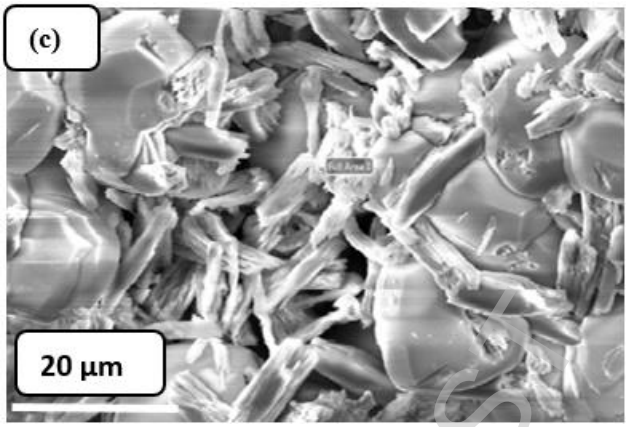


Figure 3 – SEM images of the synthesized MOR samples synthesized with different alkalinity ratios (a) OH^-/Si (0.39), (b) OH^-/Si (0.49), (c) OH^-/Si (0.59).

Effect of the SiO_2/Al_2O_3 ratio

Structure and crystallinity

Figure **4** shows diffractograms of the zeolites produced by hydrothermal synthesis at different SiO_2/Al_2O_3 ratios. At a SiO_2/Al_2O_3 ratio of 30 (S1), the resulting product exhibits a high degree of crystallinity. Clearly defined diffraction peaks appear at angles 2θ = 6.43, 9.73, 13.42, 19.59, 22.23, 25.63, 26.26, 27.66, corresponding respectively to the (110), (200), (111), (330), (150), (202), (350), (511) crystal planes of mordenite. These results confirm the formation of the mordenite phase, with a crystallite size measured at 43 nm, testifying to efficient crystallization.

At a lower SiO_2/Al_2O_3 ratio of around 15 (S4), sharp, well-defined peaks were detected at angles 2θ = 15.82, 18.30, 26.01, 30.62, 33.40, 35.93, 37.13, corresponding to the (211), (220), (400), (332), (431), (521), (440) crystal planes characteristic of analcime zeolite (Gili and Conato, 2018; Güngör and Özen, 2021). In addition, further peaks at 2θ = 14.30, 23.45, 27.62 were identified, corresponding to the (021), (002), (511) planes of mordenite, respectively.

The appearance of the analcime phase is generally associated with a low SiO_2/Al_2O_3 ratio or a prolonged crystallization time during mordenite synthesis (Gili and Conato, 2018; Mohamed et al, 2005). This phase is thermodynamically favored under such conditions, as it can incorporate a greater amount of aluminum into its structure. Consequently, analcime appears as a competing product to mordenite when the SiO_2/Al_2O_3 ratio is low and under conditions of high alkalinity.

Conversely, a high SiO_2/Al_2O_3 ratio, such as 60 (S5), alters crystallinity due to alkalinity deficiency. Undissolved silica, which remains unreactive, prevents adequate dissolution of raw materials and hampers the formation of crystalline structures. To achieve even higher SiO_2/Al_2O_3 ratios, up to 100, the use of organic templates is necessary, in line with previous work (Mohamed et al, 2005).

Vibrational group

Figure 5 shows the FTIR spectra of the samples synthesized at different SiO_2/Al_2O_3 ratios. The functional groups detected are characteristic of zeolites and/or aluminosilicates. The FTIR spectrum of the sample synthesized with a SiO_2/Al_2O_3 ratio of 15 (S1) appears closely similar to that obtained with a ratio of 30 (previously interpreted in Section1), with the exception of the intensification of all absorption bands. This indicates the existence of another variety of zeolite.

For the sample synthesized with a very high SiO_2/Al_2O_3 ratio, around 60, we observe almost the same vibrational bands as those present in the samples with ratios of 15 and 30. However, the band at 1026.46 cm^{-1}, corresponding to the asymmetric stretching vibration of the tetrahedral (TO) bonds, presents a higher intensity, suggesting a significant amount of amorphous phases, which is in line with the results obtained by X-ray diffraction.

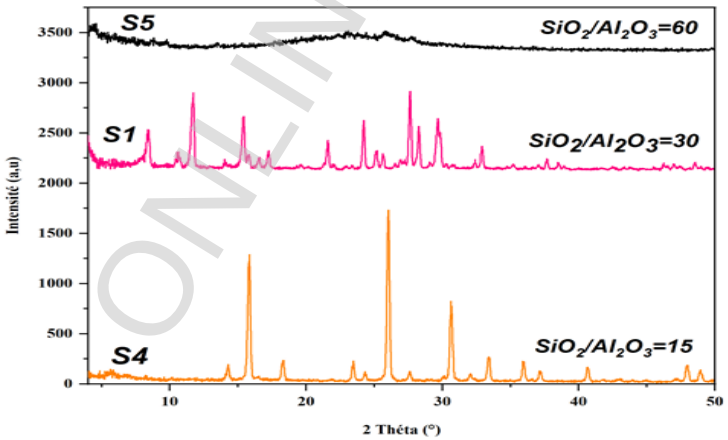


Figure 4 – XRD patterns of the MOR samples synthesized with different SiO_2/Al_2O_3 ratios

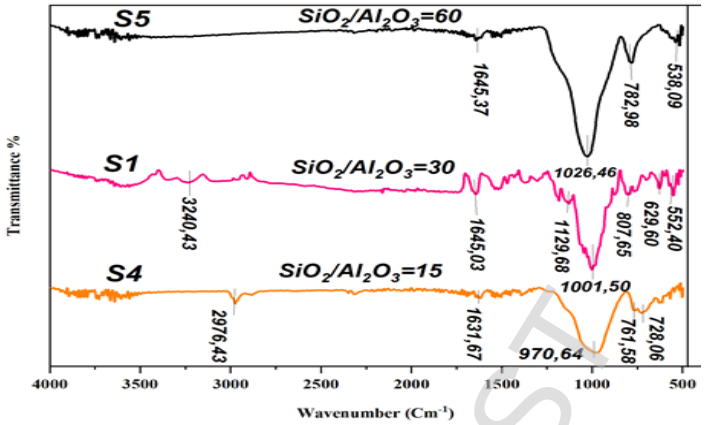
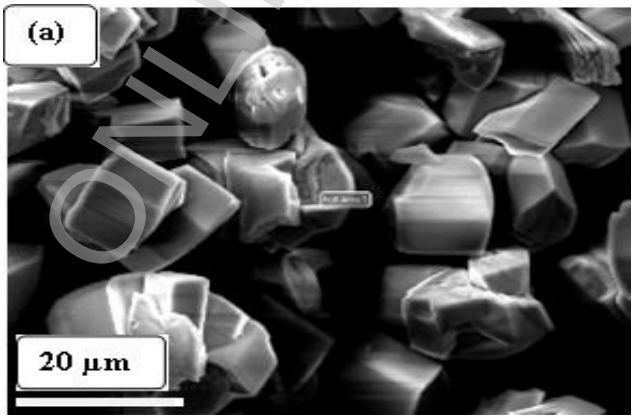


Figure 5 – FTIR spectra of the MOR samples synthesized with different SiO_2/Al_2O_3 ratios

Morphology

Scanning electron microscopy (SEM) images of the synthesized products are shown in Figure 6. The sample prepared with an SiO_2/Al_2O_3 ratio of 30 is shown in Figure 6a. It consists of particles with well-defined contours although most of them display an irregular shape. A significant proportion of these particles adopt a hexagonal shape. For the sample synthesized with a SiO_2/Al_2O_3 ratio of 15, spherical analcime single crystals with an irregular surface were obtained, as shown in Figure 6b. Spheres with jagged contours can be observed, while others have a dodecahedron-like appearance, which is characteristic of analcime as it is commonly encountered (Chen et al, 2017).



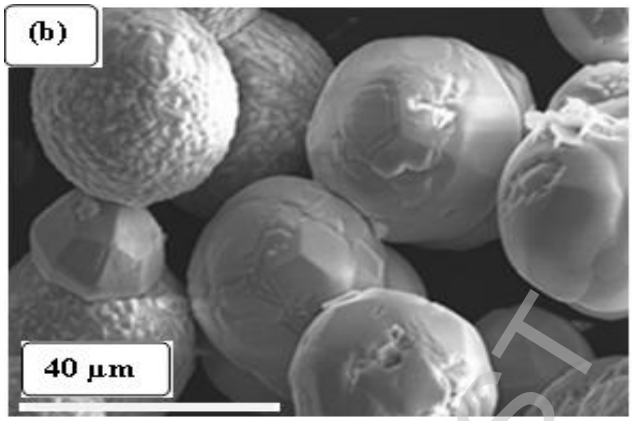


Figure 6 – SEM images of the MOR samples synthesized with different SiO_2/Al_2O_3 ratios (a) SiO_2/Al_2O_3=30, (b) SiO_2/Al_2O_3=15

Effect of the crystallization time

Structure and crystallinity

Figure 7 shows the XRD patterns for mordenite (MOR) synthesized at a constant temperature of 170°C with variable crystallization times. These patterns reveal a high degree of crystallinity in the two samples analyzed (S6) (S7). The XRD spectrum of the sample crystallized for 48 hours is comparable to that obtained after 72 hours, indicating that the structural stability of mordenite is maintained even after prolonged crystallization, without the appearance of undesirable secondary phases.

However, very short crystallization times lead to the formation of amorphous phases, as reported in Gili's study (M. Gili and Conato, 2019). Conversely, excessively long crystallization times can lead to the coexistence of several crystalline phases. The analysis also shows that longer crystallization times favour an increase in crystallite size. This is explained by the longer time available for crystallite growth before the system reaches the thermodynamic equilibrium.

The work of Zhu et *al* (Nazir et al, 2020; Zhu et al, 2014), confirms the significant influence of crystallization time on crystallite growth and dehydration performance of mordenite membranes. This research indicates that longer crystallization times enable increased crystal growth, which improves material performance. In addition, further studies (Zhu et al, 2016) show that longer crystallization times promote better organization of crystalline structures, leading to larger, more homogeneous crystallites.

Vibrational group

Figure 8 shows the FTIR spectra of the synthesized samples at different crystallization times. Both samples show the same profile in the IR spectra. Thus, the samples reveal vibrational bands typical of mordenite (Klunk et al, 2020). The FTIR spectra of mordenite show two vibrational bands of 3336.02 cm^{-1} and 3232.15 cm^{-1} for the sample synthesized at 170 °C for 48 hours and 3334.21 cm^{-1} for the sample synthesized at 170 °C for 72 hours associated with silanol end groups (Si-O-H and Si-OH-Al). In addition, the H-OH bending bands were observed at 1694.14 cm^{-1} and 1645.46 cm^{-1}, while the absorption bands at 1130.23 cm^{-1} and 1130.04 cm^{-1} represent the external asymmetric stretching vibration between the SiO_4 and AlO_4 tetrahedra of zeolite. Vibrational stretching was found at 1027.51 cm^{-1} and 1001.11 cm^{-1}, linked to the TO group. The wavelengths of (808.20 cm^{-1} - 794.38 cm^{-1}), (626.53 cm^{-1} - 605.34 cm^{-1}) and (574.41 cm^{-1} - 549.62 cm^{-1}) for both spectra are related to Al-O-Al, Si-O-Si vibrations, and internal asymmetric stretching of Al-O and Si-O (Klunk et al, 2020).

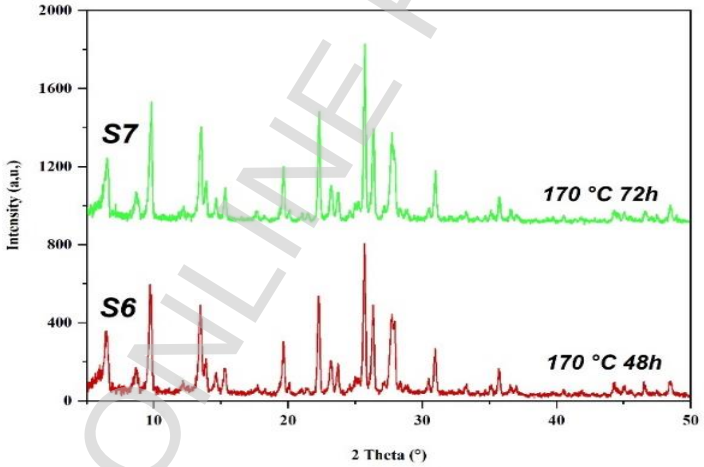


Figure 7– XRD patterns of the MOR samples synthesized with different crystallization times (48 hours and 72 hours)

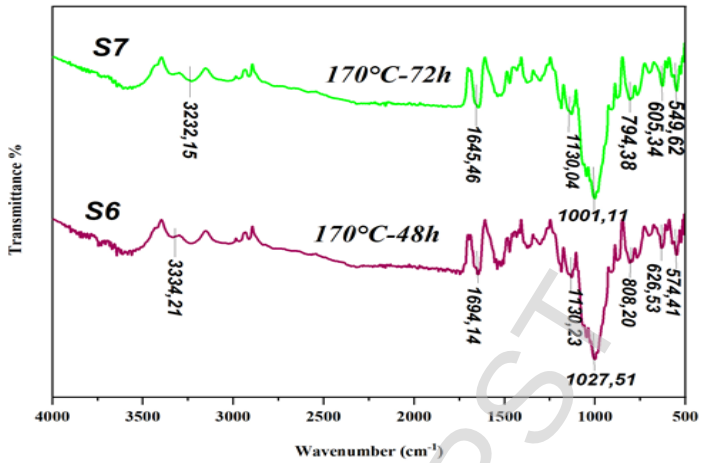


Figure 8 – FTIR spectra of the MOR samples synthesized with different crystallization times (48 and 72 hours)

Morphology

Figures 9a and 9b show the SEM images of the powder products synthesized at varying crystallization times. The sample crystallized for 48 hours is composed of hexagonal-shaped particles with surprisingly linear, well-defined edges, while the other grains are irregular in size and shape. The grain crystals have an average size of a = 1.4 µm, b = 0.73 µm, and c = 0.67 µm. In parallel, the sample crystallized over 72 hours is composed of particles of various sizes and irregular shapes, while the bulk particles have rounded edges. The grain crystals have a length of a = 1.35 µm, a width of b = 0.71 µm, and a height of c = 0.48 µm. The results demonstrate that crystallization time has an impact on particle size and morphology.

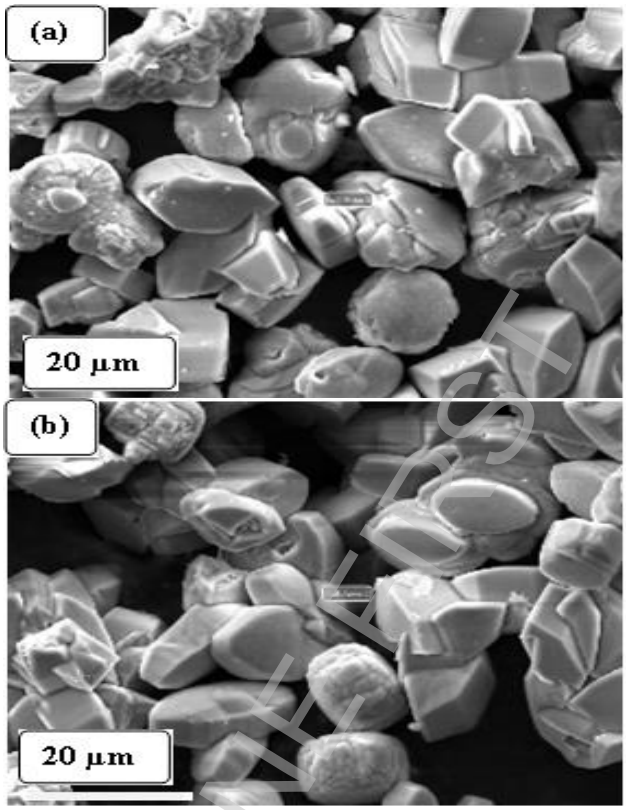


Figure 9 – SEM micrographs of the MOR samples synthesized with different crystallization times (a) 48 hours and (b) 72 hours

Conclusions

This study underscores the critical importance of controlling synthesis parameters to achieve the desired zeolite phase, particularly mordenite. Among these parameters, the OH^-/Si and SiO_2/Al_2O_3 ratios are pivotal in optimizing the crystallinity and purity of the resulting phase. A well-calibrated OH^-/Si ratio promotes the formation of pure, crystalline mordenite, whereas deviations from the optimal value can lead to the emergence of impure or undesirable phases. Similarly, a balanced SiO_2/Al_2O_3 ratio of 30 has been shown to favor the formation of crystalline mordenite. Deviations from this ratio can result in alternative phases, such as analcime or amorphous materials, highlighting the SiO_2/Al_2O_3 ratio as a key determinant of the crystalline phases formed and their degree of crystallinity.

In addition to chemical composition, crystallization time plays a decisive role. A duration of 48 hours is sufficient to produce well-crystallized mordenite, while longer crystallization periods enhance structural stability but may lead to morphological irregularities. Crystallization time is therefore a critical parameter in mordenite synthesis, influencing not only the crystal structure but also the material's functional properties. Striking the right balance is essential to optimize crystal growth while minimizing the formation of amorphous or secondary phases.

These findings provide valuable insights for the design and optimization of zeolite materials. By carefully controlling synthesis parameters, it is possible to develop zeolites with tailored properties for a wide range of applications, paving the way for advancements in material science and industrial applications.

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Efecto de las variaciones de parámetros sobre la cristalización de la zeolita mordenita

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CAMPO: materiales, tecnología química
TIPO DE ARTÍCULO: artículo científico original

Resumen:

Introducción/objetivo: Este estudio destaca la importancia de los parámetros de síntesis en la cristalización de la zeolita mordenita. El control preciso de la relación SiO_2/Al_2O_3, la alcalinidad y el tiempo de cristalización da como resultado cristales de mordenita con alta cristalinidad y pureza óptima, evitando a la vez la formación de fases secundarias o amorfas.

Métodos: Los materiales se prepararon mediante el método hidrotérmico, utilizando gel de sílice y aluminato de sodio como fuentes de silicio y aluminio, respectivamente. Se modificaron diversos parámetros de

síntesis, como la relación molar SiO_2/Al_2O_3, la alcalinidad (OH^-/Si) y el tiempo de cristalización, para evaluar su efecto en la formación de cristales de mordenita. Los experimentos se realizaron a temperatura constante de 170 °C y 190 °C.

Resultados: Los resultados muestran que la relación SiO_2/Al_2O_3 desempeña un papel crucial en la formación de cristales. Una relación baja, como 15, combinada con una alcalinidad alta, favorece la formación de cristales de analcima. Por otro lado, una relación alta, como 30, conduce a la formación de cristales de mordenita con alta cristalinidad y pureza. Sin embargo, cuando esta relación alcanza 60 y se combina con una alcalinidad baja, se impide la nucleación cristalina, dando lugar a la formación de un material amorfo.

En cuanto a la alcalinidad (OH^-/Si), los valores de 0,39 y 0,49 dan lugar a cristales de mordenita puros y bien cristalizados, mientras que valores superiores, como 0,59, conducen a la formación de fases secundarias. En cuanto al tiempo de cristalización, los periodos de 48 y 72 horas a 170 °C produjeron cristales de mordenita puros y bien cristalizados.

Conclusión: Este estudio destaca la importancia de los parámetros de síntesis en la cristalización de la zeolita mordenita. El control preciso de la relación SiO_2/Al_2O_3, la alcalinidad y el tiempo de cristalización da como resultado cristales de mordenita con alta cristalinidad y pureza óptima, evitando a la vez la formación de fases secundarias o amorfas.

Palabras claves: mordenita, alcalinidad, relación Si/Al, cristalización, zeolita.

Влияние изменений параметров на кристаллизацию цеолита морденита

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РУБРИКА ГРНТИ: 61.13.21 Химические процессы,
ВИД СТАТЬИ: оригинальная научная статья

Резюме:

Введение/цель: Данное исследование подчеркивает важность параметров синтеза при кристаллизации цеолита морденита. Точный контроль соотношения SiO_2/Al_2O_3, щелочности и времени

кристаллизации позволяет получить кристаллы морденита с высокой кристалличностью и оптимальной чистотой, избегая при этом образования вторичных или аморфных фаз.
Методы: Материалы были получены гидротермальным методом с использованием силикагеля и алюмината натрия в качестве источников кремния и алюминия. Были изменены некоторые параметры синтеза, включая молярное соотношение SiO_2/Al_2O_3, щелочность (OH^-/Si), а также время кристаллизации для того, чтобы оценить их влияние на формирование кристаллов морденита. Эксперименты проводились при постоянной температуре: 170°C и 190°C.
Результаты: Результаты показывают, что соотношение SiO_2/Al_2O_3 играет решающую роль в образовании кристаллов. Низкое соотношение, например, 15 в сочетании с высокой щелочностью способствует образованию кристаллов анальцима. С другой стороны, высокое соотношение, например, 30 приводит к образованию кристаллов морденита с высокой степенью кристалличности и чистоты. Однако когда это соотношение достигает 60 и сочетается с низкой щелочностью, усложняется зарождение кристаллов, что приводит к образованию аморфного материала.
Что касается щелочности (OH^-/Si), то значения 0, 39 и 0, 49 приводят к образованию чистых, хорошо кристаллизованных кристаллов морденита, а более высокие значения, такие как 0, 59, приводят к образованию вторичных фаз. По времени процесс кристаллизации в течение 48 и 72 часа при 170C привел к образованию чистых, хорошо кристаллизованных кристаллов морденита.
Выводы: Данное исследование подчеркивает важность параметров синтеза при кристаллизации цеолита морденита. Точный контроль соотношения SiO_2/Al_2O_3, щелочности и времени кристаллизации способствует образованию кристаллов морденита с высокой кристалличностью и оптимальной чистотой, избегая при этом образования вторичных или аморфных фаз.
Ключевые слова: Морденит, щелочность, соотношение Si/Al, кристаллизация, цеолит.

Утицај варијација параметара на кристализацију зеолита морденита

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ОБЛАСТ: материјали, хемијске технологије
КАТЕГОРИЈА (ТИП) ЧЛАНКА: оригинални научни рад

Сажетак:

Увод/циљ: У овом истраживању наглашава се значај параметара синтезе у кристализацији зеолита морденита. Прецизна контрола односа SiO_2 /Al_2 O_3 , алкалности и времена кристализације резултира кристалима морденита високе кристалности и оптималне чистоће, уз избегавање формирања секундарних или аморфних фаза.

Методе: Материјали су припремљени хидротермалном методом, коришћењем силика гела и натријум алумината као извора силицијума, односно алуминијума. Неколико параметара синтезе је варирано, укључујући моларни однос SiO_2 /Al_2 O_3 , алкалност (OH^- /Si), као и време кристализације, како би се проценио њихов утицај на формирање кристала морденита. Експерименти су изведени на константној температури од 170°C и 190°C.

Резултати: Резултати показују да однос SiO_2 /Al_2 O_3 има кључну улогу у формирању кристала. Низак однос, као што је 15, у комбинацији са високом алкалношћу, фаворизује формирање кристала аналкима. С друге стране, висок однос, као што је 30, доводи до формирања кристала морденита високе кристалности и чистоће. Међутим, када овај однос достигне 60 и комбинује се са ниским алкалитетом, кристална нуклеација је ометана, што доводи до формирања аморфног материјала.

Што се тиче алкалности (OH^-/Si), вредности од 0,39 и 0,49 резултирају чистим, добро кристализованим кристалима морденита, док веће вредности, као што је 0,59, доводе до формирања секундарних фаза. Када се посматра време кристализације, периоди од 48 и 72 сата на 170°C дали су чисте, добро кристализоване кристале морденита.

Закључак: Ово истраживање наглашава значај параметара синтезе у кристализацији морденита као врсте зеолита. Прецизна контрола односа SiO_2 /Al_2 O_3 , алкалности и времена кристализације резултира кристалима морденита високе кристалности и оптималне чистоће, уз избегавање формирања секундарних или аморфних фаза.

Кључне речи: морденит, алкалитет, Si/Al однос, кристализација, зеолит.

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