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Results of Hydrothermal Treatment of the Amorphous Phases Obtained by Ball Milling of Zeolites A, X and Synthetic Mordenite

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High-energy ball milling of zeolites A, X and synthetic mordenite for an appropriate time results in the formation of true amorphous aluminosilicate phases having the same chemical composition as the starting (unmilled) crystalline materials (zeolites). Since the solubility of thus prepared amorphous solids in hot alkaline solutions is considerably higher than the solubility of the starting zeolites under the same conditions, it can be expected that hydrothermal treatment of the amorphous solids would result in their transformation to more stable phases by solution-mediated processes. To evaluate this thesis, the X-ray amorphous solid phases obtained by high-energy ball milling of zeolites A, X and synthetic mordenite were hydrothermally treated at 80 °C by 2 M and 4 M NaOH solution, respectively, for 4 h. The products obtained (zeolites A, P and hydroxysodalite) were characterized by X-ray powder diffraction and particle size distribution measurements. It was concluded that the nuclei for zeolite crystallization originate from the residual nano-sized quasicrystalline particles (short-range ordering of Si and Al atoms inside amorphous regions that have not been completely destroyed during milling). Type(s) of the zeolite(s) (zeolite A, zeolite P_2) crystallized by the growth of the nuclei under the given conditions are determined by the chemical composition of the liquid phase (concentrations of Si and Al), and by the chemical composition of the precursor (determined by the type of mechanochemically amorphized zeolite) and the alkalinity of the system (NaOH concentration in the liquid phase), respectively. The results obtained are in agreement with the thermodynamic stabilities of

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the zeolite types that may be crystallized under the given conditions and at relative rates of crystallization.

Key words: zeolite A, zeolite X, synthetic mordenite, amorphization, ball milling, hydrothermal treatment, transformation.

INTRODUCTION

High-energy ball milling of zeolites A, X and synthetic mordenite results in a decrease of crystallinity and formation of X-ray amorphous phases (having the same chemical composition as the starting zeolites),[1–3] followed by a decrease of the mean particle size and a simultaneous increase of the number of particles and their geometric specific surface area.[2] Part of the smaller particles formed by mechanochemical breaking of the original zeolite crystals tend to form aggregates, which cyclically change properties by a series of aggregation and disaggregation processes during the milling. Since there is no reasonable correlation between the change of the amorphous phase fraction and the change of particulate properties during the milling, it was concluded that the process of amorphization and the change in particulate properties are two parallel, but more or less independent processes.[2] Analysis of the change of effective crystallite size during the milling showed that transformation of the crystalline to amorphous phase is caused by the type II amorphization process in accordance with the classification of Weber and Bakker,[4] and that the amorphization is a consequence of the breaking of »external« Si–O–Si and Si–O–Al bonds of the zeolite structure rather than of the lowering of the crystal size below the X-ray detection limit.[1,2] The amorphization process that leads to the formation of »true« amorphous aluminosilicate phases was also confirmed by changes of the I.R. spectra of the samples obtained during the milling of zeolites A, X and synthetic mordenite.[1] The cation exchange capacity,[1] dissolution kinetics[5] and solubility[1,5] of the amorphous aluminosilicates obtained by ball milling of zeolites are comparable with the corresponding properties of the amorphous aluminosilicates precipitated from alkaline sodium aluminate and sodium silicate solutions. These properties of the amorphous aluminosilicates obtained by the ball milling of zeolites, and particularly the increased solubility (factors of 5, 3.7 and 3.5 for zeolites A, X and synthetic mordenite)[1] in alkaline media compared to the solubility of original crystalline phases (zeolites) by solution-mediated processes,[9–12] indicate that the amorphized zeolites may be re-transformed either to their original crystalline form or to other type(s) of zeolite(s) during hydrothermal treatment in alkaline media. Hence, the aim of this work is the characterization of the crystalline phases formed by hydrothermal treatment (heating in 2 and 4 molar NaOH solution at 80 °C) of the amorphous aluminosilicates obtained by the ball milling of zeolites A, X and synthetic mordenite.

EXPERIMENTAL

Zeolites 4A, 13X and synthetic mordenite (all products of Union Carbide Corp.) were amorphized by high-energy ball milling. For this purpose, a certain amount of each zeolite was placed into a sintered bowl containing 10 balls (φ = 10 mm) (both the bowl and the balls are made of tungsten carbide + 6% Co material). The bowl was rotated (rotation speed 3000 rpm) for a predetermined time, t_m (2 h for zeolites 4A and 13X and 3 h for synthetic mordenite), using a planetary ball mill (Fritsch Pulverisette type 7).

Hydrothermal transformation of the amorphized samples was carried out as follows: 3 g of precursor (amorphized zeolite A, X and/or synthetic mordenite) was placed into a stainless-steel reaction vessel that contained 150 ml of 2 or 4 M NaOH solution and preheated to the transformation temperature (80 °C). The reaction vessel was provided with a thermostated jacket and fitted with a water-cooled reflux condenser and a thermometer. The reaction mixture was heated for 4 h at 80 °C. During the heating, the reaction mixture was stirred with a Teflon-coated magnetic bar driven by a magnetic stirrer. The suspension obtained after the treatment (heating at 80 °C for 4 h) was centrifuged to stop the transformation process and to separate the solid from the liquid phase. After removal of the clear liquid phase, the solid phase was redispersed in distilled water and centrifuged repeatedly. The procedure was repeated until the pH value of the liquid phase above the sediment was about 10. The wet washed solids were dried at 105 °C for 24 h, then cooled in a desiccator with silicagel, and subsequently pulverized in an agate mortar. The powdered solids were used for X-ray powder diffraction analysis, IR spectroscopy and particle size analysis.

The X-ray powder diffractograms of all samples (starting zeolite powders, amorphous aluminosilicates obtained by ball milling and the solid phases obtained by hydrothermal treatment of amorphized zeolites) were taken with a Philips PW 1820 Vertical Goniometer mounted on Philips PW 1300 X-ray generator (Cu-Kα radiation) in the corresponding region of Bragg's angles. Data were collected in the 2θ range from 5 to 60°, in steps of 0.02°, with 1 s per step. The mass fractions of different crystalline and/or amorphous phases in powdered samples were calculated by the Hermans-Weidinger method,[6] using the summation of normalized intensities of the most intensive reflections of the relevant crystalline phases.

Infrared transmission spectra of solid samples were obtained by the KBr wafer technique. The spectra were recorded on a FT-IR spectrometer SYSTEM 2000 FT-IR (Perkin-Elmer) in the region of 400 to 4000 cm^{-1} (20 scans).

Particle size distribution curves of the samples were determined using a Disc Centrifuge with photosedimentometer Mark-III (Joyce-Loebl). The mean hydrodynamic particle diameter, D, geometric specific surface area, A_s, and specific number of particles (no. of particles/g), N_s, were calculated from the relevant particle size distribution curves as:

$$D = \sum \phi_i D_i / \sum \phi_i \tag{1}$$

$$A_s = 6 \sum \phi_i (D_i)^2 / \rho \sum \phi_i (D_i)^3 \tag{2}$$

$$N_s = 6 \sum \phi_i / \Pi \rho \sum \phi_i (D_i)^3 \tag{3}$$

where ϕ_i is the number frequency of the particles having the hydrodynamic diameter between D and ΔD, $D_i = D + \Delta D/2$ and ρ is the solid phase density. The corresponding values of $\sum\phi_i$, $\sum\phi_i D_i$, $\sum\phi_i (D_i)^2$ and $\sum\phi_i (D_i)^3$ were calculated from the particle size distribution curves by the procedures described earlier.[7,8]

RESULTS AND DISCUSSION

Analysis of the X-ray powder diffractograms presented in Figure 1 shows that the X-ray amorphous solid obtained by the ball milling of zeolite A (diffractogram a in Figure 1) spontaneously transforms to a mixture of zeolite A (mass fraction, w, 79%) and hydroxysodalite (*ca.* 21%) (X-ray powder diffractogram b in Figure 1) during its heating in 2 M NaOH solution. The same amorphous solid transforms to a mixture having 62% of zeolite A and 38% of hydroxysodalite (X-ray powder diffractogram c in Figure 1) during its heating in 4 M NaOH solution. The higher percentage of zeolite A in the products may be explained in terms of Ostwald's law of stages,[13] namely the

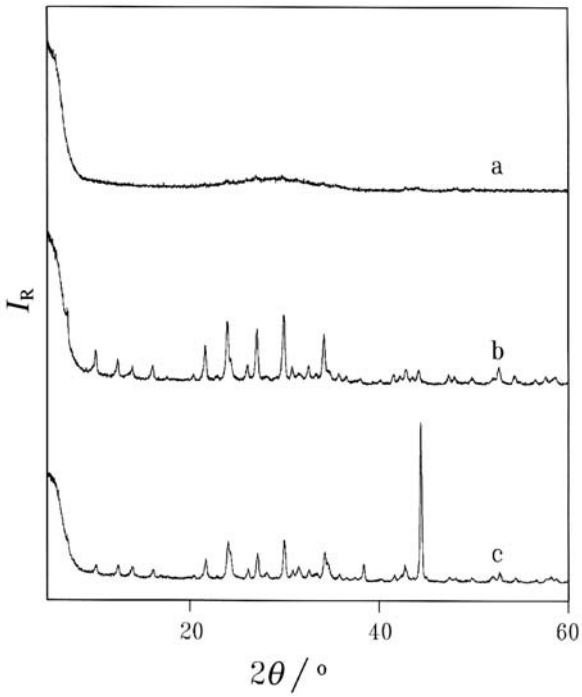

Figure 1. X-ray powder diffractograms (relative intensity I_R *vs.* Bragg's angles 2θ) of the amorphous solid prepared by the ball milling of zeolite A (a) and of the crystalline end products obtained by hydrothermal treatment (heating at 80 °C for 4 h) of the amorphous solid in 2 M (b) and 4 M (c) NaOH solutions, respectively.

rate of crystallization of the thermodynamically less stable zeolite A is higher than the rate of crystallization of hydroxysodalite. Since the crystallization of hydroxysodalite from gel, as well as transformation of zeolite A to hydroxysodalite, is favoured at higher alkalinities of the reaction mixture,[12,14–16] an increase in the system alkalinity increases the crystallization rate of hydroxysodalite more than the crystallization rate of zeolite A. Therefore, the higher content of hydroxysodalite in the product obtained by heating the amorphous solid in 4 M NaOH solution, compared to that obtained by the use of 2 M NaOH solution, was expected. Almost identical particle size distribution curves (Figure 2) and almost the same specific number, N_s, of particles in both products (Table I) indicate that hydroxysodalite

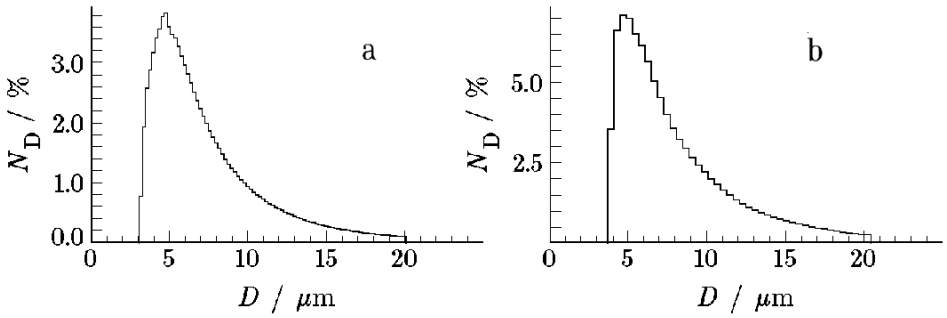

Figure 2. Particle size distribution by the number of the crystalline end products obtained by hydrothermal treatment (heating at 80 °C for 4 h) of amorphized zeolite A in 2 M (a) and 4 M (b) NaOH solutions, respectively. N_D is the number percentage of particles having the size between D and $D + \Delta D$.

TABLE I

Average size, D; geometric specific surface area, A_s; and specific number of crystals, N_s; of the crystalline end products obtained by hydrothermal treatment[a] of the X-ray amorphous solids, prepared by the high-energy ball milling of zeolites A, X and synthetic mordenite (SM)

Type of amorphized zeolite	D / μm		A_s / m^2 g^{-1}		N_s[b]	
	2 M NaOH	4 M NaOH	2 M NaOH	4 M NaOH	2 M NaOH	4 M NaOH
4A	8.03	7.78	0.156	0.108	4.2×10^8	4.2×10^8
13 X	5.87	5.38	0.170	0.234	1.3×10^9	2.3×10^9
SM	6.06	4.29	0.207	0.262	1.6×10^9	3.9×10^9

[a] Heating at 80 °C in 2 M and/or 4 M NaOH solutions, respectively, for 4 h.
[b] No. of particles / g.

is not formed by the transformation of the already crystallized zeolite A, but that crystallizations of both zeolites are parallel processes having different rates.[17,18] If it is taken into consideration that nuclei originate from the residual nano-sized quasicrystalline particles (short-range ordering of Si and Al atoms inside amorphous regions that have not been completely destroyed during milling, indicated by the weak broad peak with a minimum at 579 cm^{-1} in the FT-IR spectrum a of the amorphous precursor in Figure 3),[1] the number of particles is constant regardless of the NaOH concentration in the liquid phase. Since the particles of quasicrystalline phase can act as nuclei for all types of zeolites that may crystallize under the given conditions,[19–21] the same specific number of particles (crystals) is available for recrystallization from the same precursor when resuspended in 2 M NaOH solution and 4 M NaOH solution, respectively.[22]

Heating of the amorphous solid obtained by ball milling of zeolite X (X-ray powder diffractogram a in Figure 4 and FT-IR spectrum b in Figure 3) at 80 °C in 2 M NaOH solution for 4 h resulted in formation of a mixture having 60% (w) of zeolite A and 40% of zeolite P_2 (tetragonal form of zeolite P)[23] (X-ray powder diffractogram b in Figure 4). The appearance of the peaks of tungsten carbide at 2θ = 21.35°, 35.64° and 48.28° in the X-ray powder diffractograms indicates contamination of the milled zeolite powders

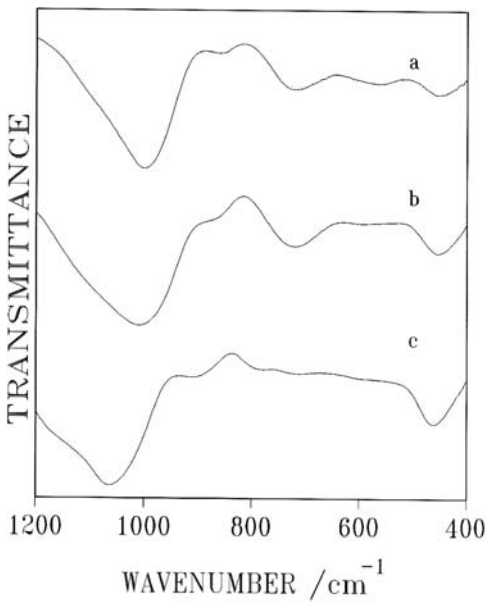

Figure 3. Infrared transmission spectra of the amorphous solids, prepared by the ball milling of zeolite A (a), zeolite X (b), and synthetic mordenite (c).

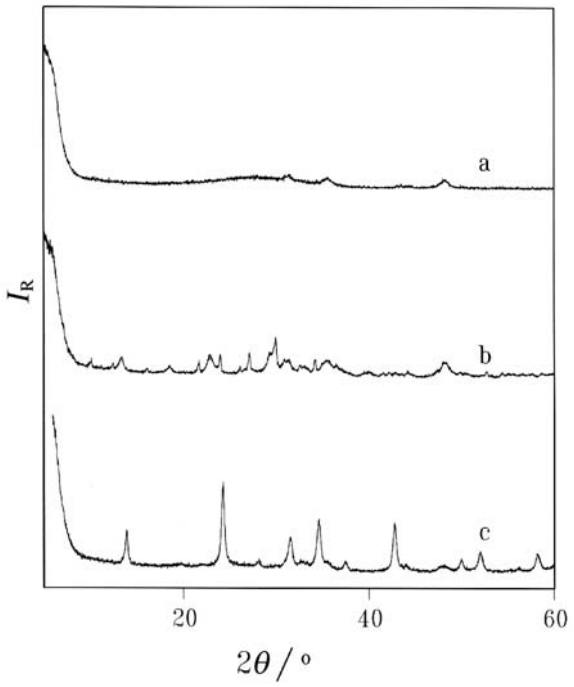

Figure 4. X-ray powder diffractograms (relative intensity I_R *vs*. Bragg's angles 2θ) of the amorphous solid prepared by the ball milling of zeolite X (a) and of the crystalline end products obtained by hydrothermal treatment (heating at 80 °C for 4 h) of the amorphous solid in 2 M (b) and 4 M (c) NaOH solutions, respectively.

due to the abrasion of the tungsten carbide bowl and balls. Taking into consideration the congruent dissolution of amorphous aluminosilicates in hot alkaline solutions,[5] the relatively low molar ratio Si : Al (= 1.25) in the liquid phase of the crystallizing system at a relatively high alkalinity of the crystallizing system (2 M NaOH in the liquid phase) favours the crystallization of zeolite A. However, an »excess« of Si over Al in the liquid phase promotes the crystallization of P type zeolites,[14,19–21] or even spontaneous transformation of zeolite A to zeolites of P_1 or P_2 type.[24] The type of the reaction (parallel crystallization of zeolites A and P or solution-mediated transformation of already formed zeolite A to zeolite P) cannot be specified at present. Future analyses of the results of kinetic experiments are planned. Heating of amorphized zeolite X in 4 M NaOH solution at 80 °C for 4 h results in complete transformation of the amorphous solid to hydroxysodalite (X-ray powder diffractogram c in Figure 4). Since the heating of amorphized zeolite A under similar conditions results in the formation of a mixture of zeolite A (62%) and hydroxysodalite (38%), it can be concluded

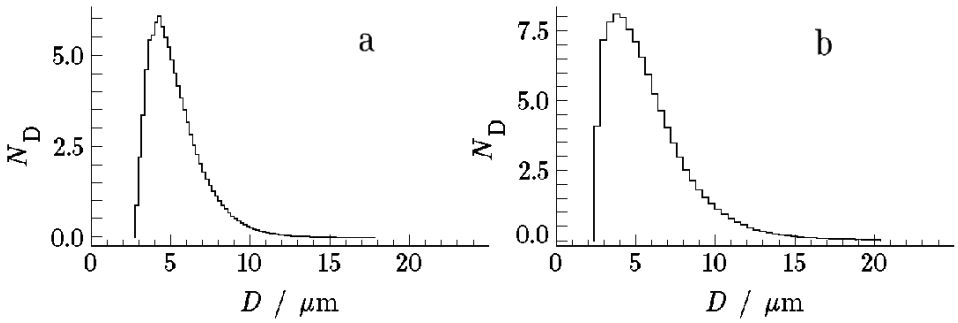

Figure 5. Particle size distribution by the number of the crystalline end products obtained by hydrothermal treatment (heating at 80 °C for 4 h) of amorphized zeolite X in 2 M (a) and 4 M (b) NaOH solutions, respectively. N_D is the number percentage of particles having the size between D and $D + \Delta D$.

that an increase of the molar Si : Al ratio in the liquid phase increases the rate of crystallization of hydroxysodalite over the rate of crystallization of other competitive types of zeolites. However, the difference in the particle size distribution curves (Figure 5) and particulate properties of the crystalline end products obtained by the heating of amorphized zeolite X in 2 M and 4 M NaOH solutions, respectively, (Table I), indicates that the hydroxysodalite is formed *via* the solution-mediated transformation of already formed zeolites A and/or P, respectively, rather than by direct crystallization from amorphized zeolite X.

Heating of the amorphous solid obtained by ball milling of synthetic mordenite (X-ray powder diffractogram a in Figure 6 and FT-IR spectrum c in Figure 3) at 80 °C in 2 M NaOH solution for 4 h resulted in the formation of a mixture having 30% of zeolite A and 70% of zeolite P_2 (X-ray powder diffractogram b in Figure 6), and the heating of the same amorphous solid in 4 M NaOH solution resulted in the formation of hydroxysodalite, contaminated with traces of zeolites P_1 (cubic form of zeolite P), P_2 and trydimite (X-ray powder diffractogram c in Figure 6). It was also found that a considerably higher amount of zeolite P_2, and consequently a lower amount of zeolite A, in the crystalline end product was obtained by heating amorphized synthetic mordenite (Si : Al ≈ 5 in the liquid phase) compared to the crystalline end product obtained by the heating of amorphized zeolite X (Si : Al = 1.25 in the liquid phase). These results confirm the conclusions based on the analysis of the results obtained during the hydrothermal treatment of amorphized zeolites A and X, respectively, namely that an increase of the molar ratio Si : Al in the liquid phase favours crystallization of P type zeolites at lower alkalinity (*e.g.*, 2 M NaOH in the liquid phase), and that

the increase of the molar ratio Si : Al in the liquid phase favours crystallization of hydroxysodalite at higher alkalinity (*e.g.*, 4 M NaOH in the liquid

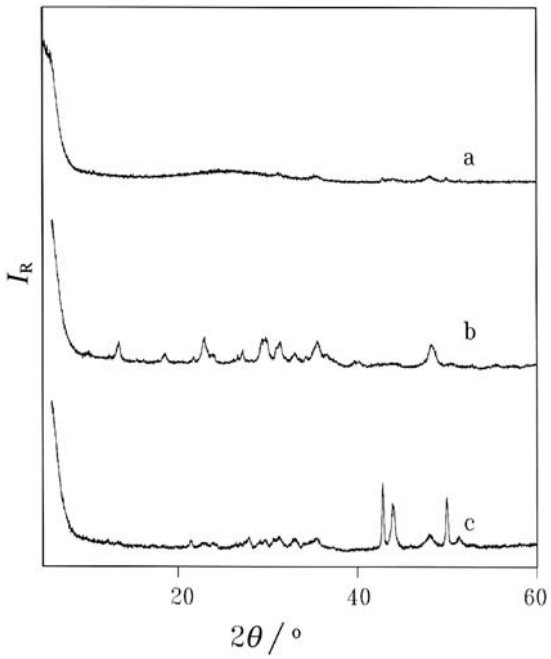

Figure 6. X-ray powder diffractograms (relative intensity I_R *vs*. Bragg's angles 2θ) of the amorphous solid prepared by the ball milling of synthetic mordenite (a) and of the crystalline end products obtained by hydrothermal treatment (heating at 80 °C for 4 h) of the amorphous solid in 2 M (b) and 4 M (c) NaOH solutions, respectively.

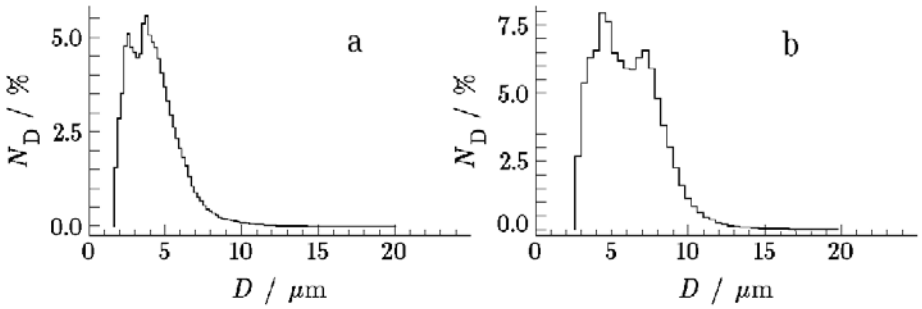

Figure 7. Particle size distribution by the number of the crystalline end products obtained by hydrothermal treatment (heating at 80 °C for 4 h) of amorphized synthetic mordenite in 2 M (a) and 4 M (b) NaOH solutions, respectively. N_D is the number percentage of particles having the size between D and $D + \Delta D$.

phase). The presence of traces of zeolite P_2 in the crystalline end product obtained in 4 M NaOH solution is probably caused by the fact that the rate of transformation of zeolite P_2 into hydroxysodalite is slower than the rate of transformation of zeolite A into hydroxysodalite, thus adding weight to the assumption that hydroxysodalite is formed by the solution-mediated transformation of already formed zeolites A and/or P, rather than by direct crystallization from the amorphous precursor. The difference in the particulate properties of the crystalline end products obtained by heating amorphized synthetic mordenite in 2 M and 4 M NaOH solutions, respectively, (Figure 7 and Table I), also supports this assumption.

CONCLUSION

The crystalline end products, resulting from the hydrothermal treatment (heating in 2 M and 4 M NaOH solutions, respectively, at 80 °C for 4 h) of amorphous aluminosilicates, prepared by high-energy ball milling of zeolites A, X and synthetic mordenite, were characterized by X-ray powder diffraction and particle size analysis. Characterization of the crystalline end products obtained by the hydrothermal treatment of amorphous solids shows that:

– Nuclei for crystallization of zeolite(s) originate from the residual nano-sized quasicrystalline particles (short-range ordering of Si and Al atoms inside amorphous regions) that have not been completely destroyed during milling.
– Type(s) of the zeolite(s) to be crystallized by the growth of the nuclei at a given temperature for a given time are determined by the chemical composition of the liquid phase (Si and Al concentrations), and thus by the chemical composition of the precursor (determined by the type of mechanochemically amorphized zeolite) and by the alkalinity of the system (NaOH concentration in the liquid phase), respectively.
– The mixture of zeolite A (79%) and hydroxysodalite (21%; mass fractions, *w*), obtained by heating amorphized zeolite A in 2 M NaOH solution (at 80 °C for 4 h), indicates that the crystal growth of both zeolite A and hydroxysodalite, takes place simultaneously, and that the rate of crystal growth of zeolite A is considerably higher than the rate of crystal growth of hydroxysodalite under the same conditions. On the other hand, a higher content of hydroxysodalite in the product obtained by heating the amorphous solid in 4 M NaOH solution rather than in 2 M NaOH solution indicates that an increase in the alkalinity of the system increases the rate of crystallization of hydroxysodalite more than the rate of zeolite A crystallization.

– An increase of the molar ratio of Si/Al in the liquid phase favours simultaneous growth of zeolite A and zeolite P_2, so that the heating (at 80 °C for 4 h) of amorphized zeolite X (molar ratio Si/Al = 1.25, in the liquid phase) results in the formation of a mixture of 60% of zeolite A and 40% of zeolite P_2. The heating of amorphized synthetic mordenite (molar ratio Si/Al = 5, in the liquid phase) under the same conditions results in the formation of a mixture of 30% of zeolite A and 70% of zeolite P_2. An increase of the system alkalinity (transformation in 4 M NaOH solution) results in complete transformation of the already formed zeolites (A and P_2) into hydroxysodalite. The rate of transformation of zeolite A into hydroxysodalite is higher than the rate of transformation of zeolite P_2 into the same phase.
– The results obtained are in agreement with the thermodynamic stabilities of the types of zeolites that may be crystallized under the given conditions and the relative rates of their crystallization. Hence, hydrothermal treatment of different types of amorphized zeolites under controlled conditions (alkalinity of the liquid phase, temperature of heating, time of heating) offers the possibility of preparing different types of zeolites (mixtures and/or pure forms) with the desired particulate properties.

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SAŽETAK

Rezultati hidrotermalne obrade amorfnih faza dobivenih mljevenjem zeolita A, X i sintetskog mordenita u kugličnom mlinu

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Visokoenergijskim mljevenjem zeolita A, X i sintetskog mordenita u kugličnom mlinu nastaju amorfni alumosilikati istog kemijskog sastava kao i početne kristalne faze (zeoliti). Budući da je topljivost tako pripravljenih amorfnih krutina u vrućim lužnatim otopinama znatno veća od topljivosti zeolita iz kojih su dobivene, može se očekivati njihova transformacija (posredstvom tekuće faze) u stabilnije faze tijekom hidrotermalne obrade. Radi provjere navedene pretpostavke, rentgenski amorfne krutine dobivene visokoenergijskim mljevenjem zeolita A, X i sintetskog mordenita zagrijavane su na 80 °C u 2 M i 4 M otopini NaOH tijekom 4 h. Dobiveni produkti (zeoliti A, P i hidroksisodalit) karakterizirani su rentgenskom difrakcijom i mjerenjem raspodjele veličine čestica. Utvrđeno je da je izvor nukleusa za kristalizaciju zeolita ostatak kvazi-kristalnih čestica nano-veličine (uređenje kratkog dosega atoma Si i Al unutar amorfne faze koje nije potpuno uništeno tijekom mljevenja). Tipovi zeolita (zeolit A, zeolit P_2), koji su kristalizirani rastom nukleusa pri danim uvjetima, definirani su kemijskim sastavom tekuće faze (koncentracijama Si i Al), kemijskim sastavom prekursora (koji je određen tipom mehanokemijski amorfiziranog zeolita) i lužnatosti sustava (koncentracija NaOH u tekućoj fazi). Dobiveni rezultati u skladu su s termodinamičkim stabilnostima tipova zeolita koji mogu kristalizirati pri danim uvjetima i brzinama kristalizacije.