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Synthesis of Pure Phase NaP2 Zeolite from the Gel of NaY by Conventional and Microwave-Assisted Hydrothermal Methods

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Abstract: The gel of zeolite NaY has potential as a precursor of other zeolites. The particular interest in this work is to convert the gel of NaY to NaP2. We found that the pure phase NaP2 can be produced simply by the conventional hydrothermal (CH) method at 150 °C for 24 h. This NaP2 sample, named CH150, has an average particle size of 10.3 µm and an Si/Al ratio of 1.82. In the case of single crystallization via microwave-assisted hydrothermal (MH) method, various parameters were studied, including the crystallization temperature (90, 150, 175 °C) and time (15, 30, 45, 60 min). The samples were analyzed by X-ray diffraction and scanning electron microscopy. However, mixed phases of P1 and P2 or ANA were obtained from all samples. Another attempt was made by a double crystallization via MH method as followed: at 90 °C for 1 h, quickly cooled down to room temperature in the microwave chamber and aged for 23 h, and finally at 150 °C for 1 h. The sample, named MH90A150, has an average crystal size of 16.45 µm and an Si/Al ratio of 1.85. The high Al content of NaP2 in both samples (CH150 and MH90A150) could lead to interesting applications.

Keywords: NaP2 zeolite; NaY gel; microwave-assisted hydrothermal; conventional hydrothermal

1. Introduction

Zeolite P has a gismondine (GIS) framework type, composed from secondary building units (SBU) containing four- and eight-membered rings of T atoms (T = Si or Al linked together by sharing oxygen atoms). Its pore diameter is 2.8 × 4.8 Å [010] [1] which makes it useful as molecular sieve [2]. Zeolite P is useful for many applications such as the separation of small gases [3–5] or heavy metals [6,7] and application in sensing material [8].

Baerlocher et al. and Mccusker et al. [9,10] have found the differences and flexibility in unit cell structures of zeolite P. The distortion of the SBU planes generates two isotypes, P1 and P2, which have different pore sizes and shapes. Likewise, Oleksiaj et al. [11] have reported that the eight-membered pore of P2 is narrower than that of P1. Consequently, the P2 type has a higher adsorption equilibrium constant for small gases such as H2 [11].

Despite the interesting advantage, the synthesis of P2 requires a long crystallization period and produces impurity phases. Table 1 compares the synthesis of zeolite P with various parameters. The standard procedure from the International Zeolite Association Synthesis Commission (IZA) is the conventional hydrothermal method (CH) which has a long crystallization time, i.e., 60 days (Table 1, entry 1) [12]. Several researchers have improved the synthesis by extending the nucleation time (Table 1, entry 2) [13], employing a structure-directing agent (SDA) (Table 1, entry 3) [14] and seeding (Table 1,
entry 4) [15]. Although the synthesis time is shortened, it still takes several days. An approach for the fast synthesis is the microwave-assisted hydrothermal method (MH (Table 1, entry 5-6) [16,17] due to its rapid and homogeneous heating [18].)

| Table 1. Gel composition and synthesis parameters of zeolite P from the literature. |
|------------------------------------------|--------------|-------------|-----------------|-----------------|
| Gel Composition                        | Main Product | Crystallization Time | Crystallization Temperature (°C) | Method | References |
| 2.2 SiO$_2$: Al$_2$O$_3$: 5.28 NaF: 105.6 H$_2$O | P1           | 60 days      | 85              | CH$^1$ | [12]        |
| Na$_8$Al$_8$Si$_8$O$_32$·15.2 H$_2$O     | P2           | 5 days       | 100             | CH$^1$ | [13]        |
| 3.75 SiO$_2$: 0.2 Al$_2$O$_3$: 4.3 Na$_2$O: 220 H$_2$O | P1           | 42 h         | 100             | CH$^1$ | [14]        |
| 0.0065 NaAlO$_2$: 0.023 Na$_2$Si$_2$O$_7$ (seed) | P2           | 24 h         | 100             | CH$^1$ | [15]        |
| 1 SiO$_2$: 0.25 Al$_2$O$_3$: 3 Na$_2$O: 41H$_2$O | P1           | 3 h          | 110             | MH$^2$ | [16]        |
| 17 Na$_2$O: Al$_2$O$_3$: 17 SiO$_2$: 345 H$_2$O (seed) | P2           | 2 h          | 150             | MH$^2$ | [17]        |

$^1$ CH = conventional hydrothermal method, $^2$ MH = microwave-assisted hydrothermal method, $^3$ use of seeding for nuclei preparation, use of structure-directing agent: $^4$ D-methionine, $^5$ triethanolamine, $^6$ triisopropanolamin.

The impurity phases from the conventional synthesis method can be minimized by recrystallization, also called double crystallization. For example, Sousa et al. [19] recrystallized ZSM-22 (structure type TON) and ZSM-35 (structure type FER) by basic treatment to produce a pure phase of zeolite NaP1. Nevertheless, there are no reports about the synthesis of zeolite NaP2 from recrystallization. Although zeolite NaP2 has been synthesized from NaY gel, the mixed zeolite phases were obtained [20]. Consequently, this study aimed to synthesize the high purity of zeolite NaP2 from the gel of zeolite NaY through double crystallization without the separation of solid product after the first heating. Seeding and microwave radiation will be used to decrease hydrothermal time and avoid uneven temperature.

2. Materials and Methods

2.1. Materials

Chemicals in this study were sodium hydroxide anhydrous pellets (NaOH, 97%, Carlo Erba), silicon dioxide (SiO$_2$, 99%, Carlo Erba) and sodium aluminate (NaAlO$_2$, 95%, Sigma Aldrich). Sodium silicate (Na$_2$SiO$_3$), which was a silicon source precursor, was prepared according to the following chemical composition: 28.7 wt% SiO$_2$, 8.9 wt% Na$_2$O. Moreover, nitric acid (HNO$_3$, 69%, ANaPURE), hydrochloric acid (HCl, 37%, ACI Labscan), hydrofluoric acid (HF, 49% QRëC) and boric acid (H$_3$BO$_3$, 99.5%, Merck) were used in the digestion step.

2.2. Preparation of Zeolite Synthesis Gel

The overall gel with the molar ratio of 10SiO$_2$:Al$_2$O$_3$:4.62Na$_2$O:180H$_2$O was prepared by the method from that of zeolite NaY [21] with the scale on-fourth, from a seed gel (10SiO$_2$:Al$_2$O$_3$:10.67Na$_2$O:180H$_2$O) and feedstock gel (10SiO$_2$:Al$_2$O$_3$:4.30Na$_2$O:180H$_2$O). It was aged for 24 h before crystallization. All the gel ratios were calculated from the weight of each component.

2.3. Single Crystallization

The overall gel was crystallized by conventional and microwave-assisted hydrothermal methods (CH and MH, respectively). In the CH method, the overall gel was poured into a Teflon-lined autoclave (250 mL) and heated with a rate of 5 °C/min to 150 °C and held for 24 h in a muffle furnace (Carbolite, CWF 12/23) and cooled down outside the furnace to room temperature. This sample was named CH150. In the MH method, the overall gel was transfused into two microwave tubes (100 mL) with equal amount and heated by microwave (Anton Paar, Multiwave 3000) with XF-100 rotor and the power of 900 W to 150 °C within 4 min, kept for the desired period of 1 h and cooled down quickly in the microwave chamber. This sample was named MH150.
To understand the temperature effect, the samples MH90 and MH175 were prepared by microwave-assisted method and crystallized for 1 h at 90 °C and 175 °C, respectively. The result of the time was investigated by crystallization at 150 °C and 175 °C, held for 15, 30, 45 and 60 min.

2.4. Double Crystallization via MH Method

There were three steps to investigate the phase change from each step. Firstly, the overall gel was transferred into the microwave tubes with an equal amount for evenness. The sample was heated by microwave from room temperature up to 90 °C and crystallized for 1 h before cooling down. This sample was named MH90. Secondly, the sample was kept unopened (aged) for 23 h at room temperature in the same microwave tubes. This sample was named MH90A. Finally, the sample after aging was crystallized again at 150 °C for 1 h. This sample was named MH90A150.

After crystallization, all solid products were separated by centrifugation, washed several times with deionized (DI) water until the pH of the filtrates was 7–8, and dried at 90 °C for 18 h to obtain white powder samples.

2.5. Characterization of Solid Products

Phases and structures of crystalline products were analyzed by powder X-ray diffraction (XRD) on a Bruker (AXS D8, Billerica, MA, USA) diffractometer using Cu Kα radiation with a current of 40 mA and a potential of 40 kV. All the samples were analyzed on the same day for a confident comparison of crystallinity. The functional groups of the samples were determined by Fourier-transform infrared spectroscopy (FTIR) on a Bruker (Tensor 27, Billerica, MA, USA), using attenuated total reflectance (ATR) mode with a resolution of 4−1. Morphology was studied by a field emission scanning electron microscope (FESEM), JEOL (JSM-7800F, Tokyo, Japan). The silicon–aluminum (Si/Al) mole ratios in the solid products were analyzed by inductively coupled plasma optical emission spectrometry (ICP–OES) using an Perkin Elmer (Optima 8000, Waltham, MA, USA) with argon carrier gas. The samples were digested [22] as follows. Fifty milligrams of each sample was mixed in 0.5 mL aqua regia, 1HNO₃:3HCl volumetric ratio, and 3.0 mL of HF. The mixture in a 250 mL polypropylene (PP) bottle was heated at 110 °C for 1 h and cooled down to room temperature. Then, 2.8 g of H₃BO₃ and 10 mL of DI water were added into the mixture and the volume was adjusted with DI water in a 100 mL PP volumetric flask. The emission wavelength of silicon and aluminum in solid samples were 251.611 and 396.153 nm, respectively.

3. Results and Discussion

3.1. Comparison of Conventional and Microwave-Assisted Hydrothermal Methods of Single Crystallization

Figure 1 compares the XRD patterns of zeolite products from single crystallization by the conventional hydrothermal method at 150 °C for 24 h (CH150) and microwave-assisted hydrothermal method at 150 °C for 1 h (MH150). The pattern of CH150 corresponds to the characteristic phase of zeolite NaP2. The splitting of peaks refers to two rotating types of secondary building units (SBUs) planes or four-membered ring (4MR) in GIS structure [11]. Other zeolite phases are not observed. In comparison with Khabuanchalad et al. [20], the crystallization of the gel of NaY at 100 °C for 5 days produced zeolite NaP2 with a trace of NaY. This work successfully synthesizes the pure phase NaP2 from the gel of zeolite NaY by the conventional hydrothermal method through crystallization at 150 °C for 24 h.

The XRD pattern of MH150 shows the mixed zeolite phases. The main phase is NaP1 and the minor phase is NaP2. At the same crystallization temperature, Le et al. [17] obtained NaP2 as the main phase with the trace of NaY. However, they used a different gel composition, microwave power (1600 W) and longer crystallization time (2 h). The crystallization at 100 °C and 120 °C only produced zeolite NaY [17]. When they used the gel with Si/Al = 5 and a crystallization time of 0.5 h, the product was pure phase NaP2.
The faster heating rate by MH method provides the faster crystal growth rate, producing the larger
µ2020 Crystals the homogeneous growth of nuclei that a NaY by CH and MH methods [25]. Moreover, the homogeneous heating from microwave results in
on the microsphere surface and the size of each particle is below one micrometer. The observation of
image (see Figure 2b) shows larger particles with a di
Consequently, one can conclude that NaP2 has various morphology. In the case of MH150, the SEM
sample is 1.82. The morphology of CH150 is similar to zeolite P2 in the literature [11,17]. Although the
an average of 10.3 temperature MH150 results in
zeolite NaY particle size faster heating rate by MH method faster heating rate by MH method
Figure 2a shows the SEM image of CH150. This sample has polycrystals with polygons, some have
a diamond-like shape and some have trisecting cracks. The particle size is between 4 and 14 µm with
an average of 10.3 µm (calculated from 50 particles). From the ICP analysis, the Si/Al ratio of this sample is 1.82. The morphology of CH150 is similar to zeolite P2 in the literature [11,17]. Although the sample has various shapes and morphology, the XRD results only indicate the pure phase of NaP2. Consequently, one can conclude that NaP2 has various morphology. In the case of MH150, the SEM image (see Figure 2b) shows larger particles with a different morphology from CH150. The average particle size of MH150 is 17.4 µm and the Si/Al ratio was 1.77. The major phase has a cheese ball-like morphology of NaP1 similar to those in the literature [23,24]. Those zeolite microcrystals are well grown on the microsphere surface and the size of each particle is below one micrometer. The observation of mixed phases from SEM is consistent with the XRD results.

From the XRD and SEM results, the phase of products depends on the heating methods. The faster heating rate by MH method provides the faster crystal growth rate, producing the larger particle size compared with the CH method. This behavior was also observed in the synthesis of zeolite NaY by CH and MH methods [25]. Moreover, the homogeneous heating from microwave results in the homogeneous growth of nuclei that affect the narrow particle size distribution of MH150. Therefore,
the crystallization of MH is about 24 times faster than CH at the same temperature. Microwave radiation significantly reduced the time of crystallization due to fast spreading heat to all positions [26]. However, the fast heating rate results in mixed phases.

3.2. Effect of Temperature and Time on Single Crystallization by Microwave-Assisted Hydrothermal Methods

Figure 3a shows the XRD patterns of MH90, MH150 and MH175 which are the products from crystallization by microwave heating at 90, 150 and 175 °C, respectively. The MH90 sample has pure phase NaY zeolite [27]. This result is consistent with the work by Le et al. [17] that NaY was obtained at 100 and 120 °C. The sample MH150 shows mixed phases of NaP1 and NaP2 zeolites. Finally, the MH175 sample displays the mixed phases of NaP2 as the major phase and Anacime (ANA) zeolite as the minor phase [28]. The densities of faujasite (FAU) [29], GIS [1] and ANA [30] zeolites are 13.3, 16.4 and 19.2 T/1000 Å³, respectively. The results confirm that the higher crystallization temperature produces zeolite with a higher framework density [17]. Although the exact densities of the two GIS zeolites are not clear, our results imply that the density of NaP2 is higher than NaP1 due to the smaller pore size [11]. From these results, the suitable crystallization temperature for NaP2 is 175 °C. However, the product still contains ANA zeolite as an impurity. Consequently, we investigated the shorter crystallization time and lower temperature to improve purity.

Figure 3. Powder XRD patterns of zeolites from single crystallization via MH (a) for 1 h at 90 °C, 150 °C and 175 °C, (b) at 175 °C, and (c) 150 °C for various times: 15, 30, 45 and 60 min.

Figure 3b displays the XRD patterns of zeolites from single crystallization by MH at 175 °C for 15, 30, 45 and 60 min. All samples still contain an ANA phase and the sample from 60 min has more ANA phase than the others. The observation agrees with the literature that the longer crystallization time
generates a product with high density [31] with a competitive phase. Consequently, we investigated the crystallization at 150 °C to reduce the ANA phase. Figure 3c shows the XRD patterns of the samples crystallized for 15, 30, 45 and 60 min. The main phase of all samples was NaP1 [11] along with NaP2.

Figure 4 presents the SEM images of zeolites from single crystallization via MH at 150 °C for 15, 30, 45 and 60 min. The sample from 15 min has an amorphous phase which is from a short crystallization time. Moreover, other samples have a mixed phase of NaP1 (cheese ball like) [23,24] and NaP2 (polygonal) [11,17] consistent with the XRD patterns. The presence of the mixed-phase suggests that a longer aging time is necessary.

### Figure 3.
Powder XRD patterns of zeolites from single crystallization via MH (a) for 1 h at 90 °C, 150 °C and 175 °C, (b) at 175 °C, and (c) 150 °C for various times: 15, 30, 45 and 60 min.

### Figure 4.
FESEM images of zeolite products from microwave-assisted hydrothermal methods with single crystallization at 210 ˚C for various times: (a) 15, (b) 30, (c) 45 and (d) 60 min.

#### 3.3. Effect of Double Crystallization by Microwave-Assisted Hydrothermal Methods

Figure 5 compares the XRD patterns of MH90 with the further aged sample (MH90A) and crystallized at 150 °C (MH90A150). The patterns of MH90 and MH90A correspond to NaY [27]. The peaks from MH90A are stronger than those of MH90, indicating that the longer aging time improves crystallinity. After crystallization at 150 °C, the pattern of MH90A150 exhibits the pure phase of NaP2 zeolite. These results suggest that the synthesis condition of MH90A150 is suitable to transform the gel of zeolite NaY to NaP2. Concurrently, the second crystallization under microwave heating causes NaY to depolymerize the small building units [19] of NaP2 which has the denser structure.

Figure 6 compares the FTIR spectra of MH90 and MH90A150 and the interpretations are summarized in Table 2. The spectra of MH90 and MH90A150 correspond to the functional groups of zeolite NaY [32–34] and NaP [34–37], consistent with the XRD results.

Figure 7 exhibits the SEM images of MH90 and MH90A150. The morphology of MH90 (Figure 7a) is a polygon shape similar to the NaY zeolite in the literature [38]. The average particle size of MH90 is 230 nm and the Si/Al ratio is 1.88. The MH90A150 particles (Figure 7b) have the polygonal morphology, similar to NaP2 zeolite in the literature [11,17]. To ensure the reproducibility of pure phase NaP2 zeolite from this method, we repeated the synthesis with the same manners. The similar XRD patterns (Figure 8) indicate that pure NaP2 could be produced by our synthesis method. The average particle size of MH90A150 is 16.45 µm and the Si/Al ratio is 1.85. The results are consistent with the XRD and FTIR data. Comparing between NaP2 from CH150 and MH90A150, the particle size distribution of MH90A150 is narrower than that of CH150 (standard deviation of 7.5% vs. 27.6%).
Figure 5. XRD patterns of zeolites from microwave-assisted hydrothermal method at 90 °C for 1 h (MH90), aged at room temperature for 23 h (MH 90A) and crystallized at 150 °C for 1 h (MH90A150).

Figure 6. Fourier-transform infrared spectroscopy (FTIR)–attenuated total reflectance (ATR) spectra of zeolite products: MH90 from the microwave-assisted hydrothermal method at 90 °C for 1 h and MH90A150 from the microwave-assisted hydrothermal method at 90 °C for 1 h followed by aging at room temperature for 23 h and recrystallization at 150 °C for 1 h.

Figure 7. FESEM images of zeolites from the microwave-assisted hydrothermal method (a) at 90 °C for 1 h (MH90), followed by aging at room temperature for 23 h and recrystallized (b) at 150 °C for 1 h (MH90A150).
Table 2. Band assignment of FTIR spectra of MH90 (FAU) and MH90A150 (GIS) samples.

| Wavenumber (cm$^{-1}$) | Assignment                                      | References     |
|------------------------|-------------------------------------------------|----------------|
| FAU zeolite            |                                                 |                |
| 1151                   | Asymmetric stretching vibration of external linkage TO$_4$ (T = Si, Al) | [32–34]        |
| 985                    | Asymmetric stretching vibration of internal tetrahedral TO$_4$          |                |
| 780                    | Symmetric stretching vibration of external linkage TO$_4$              |                |
| 696                    | Symmetric stretching vibration of internal linkage TO$_4$              |                |
| 571                    | Vibration of double-6-membered ring (D6R)                      |                |
| 453                    | Bending vibration of T-O bond in internal tetrahedron               |                |
| GIS zeolite            |                                                 | [34–37]        |
| 1118                   | Asymmetric stretching vibration of external linkage TO$_4$              |                |
| 963                    | Asymmetric stretching vibration of internal tetrahedral TO$_4$          |                |
| 796                    | Symmetric stretching vibrations of external linkage TO$_4$             |                |
| 752                    | Vibration of 4-membered ring (4MR)                              |                |
| 691                    | Symmetric stretching vibration of internal linkage TO$_4$              |                |
| 610                    | Double ring vibration in GIS zeolite framework                      |                |
| 431, 403               | Bending vibration of T–O                                       |                |

Figure 8. Powder XRD patterns of NaP2 zeolites from microwave-assisted double crystallization for repetition at the first time (MH90A150–1) and the second time (MH90A150–2).

All the conditions and results from this work are summarized in Table 3. The pure phase NaP2 can be synthesized by two methods: CH method at 150 °C for 24 h and double crystallization via MH method at 90 °C and 150 °C. Single crystallization via MH method at 90 °C produced pure phase NaY but at 150 and 175 °C produced mixed phases of NaP2 and NaP1 or ANA.

Table 3. Gel composition, synthesis parameters and phase of the products in this study.

| Sample Name | Crystallization Conditions | Phase of Product $^4$ |
|-------------|---------------------------|-----------------------|
|             | Method | Synthesis Process | Temperature (°C) | Time (min) | Major | Minor |
| CH150       | CH $^1$ | Single           | 150              | 1440       | NaP2 | -     |
| MH90        | MH $^2$ | Single           | 90               | 60         | NaY  | -     |
| MH150       | MH $^2$ | Single           | 150              | 15–60      | NaP1 | NaP2  |
| MH175       | MH $^2$ | Single           | 90               | 15–60      | NaP2 | ANA   |
| MH90A       | MH $^2$ | Single           | 90 and aged $^3$ | 60         | NaY  | -     |
| MH90A150    | MH $^2$ | Double           | 90, 150          | 60, 60     | NaP2 | -     |

$^1$ CH = conventional hydrothermal method, $^2$ MH = microwave-assisted hydrothermal method, $^3$ crystallization at 90 °C followed by aging at room temperature for 23 h, $^4$ measurement by XRD technique.
4. Conclusions

The synthesis gel of zeolite NaY with the molar ratio 10SiO$_2$:Al$_2$O$_3$:4.62Na$_2$O:180H$_2$O was prepared and used as a precursor of zeolite NaP. The pure phase zeolite NaP2 was obtained after aging the synthesis gel of zeolite NaY for 24 h followed by the crystallization by conventional hydrothermal (CH) method at 150 °C for 24 h. The sample (CH150) has polycrystals with polygons, some have a diamond-like shape, and some have trisecting cracks. The average size was 10.3 μm and the Si/Al ratio was 1.82.

From the single crystallization via microwave-assisted hydrothermal (MH) method, the pure phase NaY, the mixed phase of NaP1 (major) and NaP2 (minor), and the mixed phase of NaP2 (major) and ANA (minor) were produced after crystallization at 90 °C, 150 °C and 175 °C for 1 h, respectively. The crystallization for various times (15–60 min) at 150 °C and 175 °C also produced the mixed phases.

From double crystallization via microwave-assisted hydrothermal (MH) method, the pure phase zeolite NaP2 was obtained by three steps: (1) crystallization at 90 °C for 1 h and quickly cooling down to room temperature in the microwave chamber, (2) aging at room temperature for 23 h, and (3) crystallization at 150 °C for 1 h. This NaP2 sample has a similar morphology to the CH150 sample with an average crystal size of 16.45 μm and Si/Al ratio 1.85.

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