

# Effect of the Synthesis Parameters on the Physicochemical Properties of NaY Zeolite with Faujasite Structure

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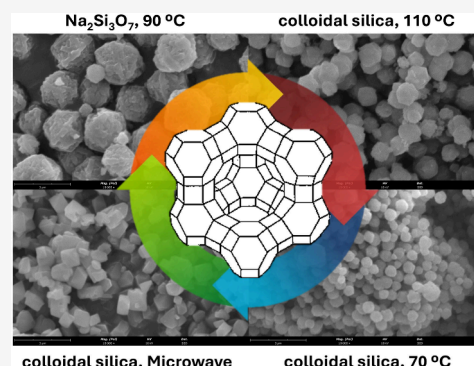
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Supporting Information

**ABSTRACT:** NaY material belongs to the iconic family of zeolites with a Faujasite structure. These microporous aluminosilicate materials possess exceptional properties, making them essential in various applications, e.g., catalysis, gas sorption/separation, and ion exchange. In particular, NaY zeolite is a good model material for teaching fundamental concepts in crystallography, surface chemistry, and adsorption phenomena and a stimulating literature review topic for students. NaY zeolite can be easily synthesized by students at the laboratory scale. Its preparation and characterization offer valuable learning opportunities by linking theoretical knowledge with practical applications in Materials Science and/or Chemical Engineering courses. In this work, we propose a very simple but highly reproducible synthesis method for obtaining a highly crystalline NaY material. This flexible approach enables undergraduate students to investigate the influence of both the crystallization time and temperature on the outcome of NaY synthesis. By combining standard characterization techniques like powder X-ray diffraction, solid-state nuclear magnetic resonance, infrared spectroscopy, and scanning electron microscopy coupled with energy-dispersive spectroscopy, students acquire hands-on experience in Materials characterization, learn about the complementarity and limitations of the different characterization methods, and strengthen their knowledge in Inorganic Chemistry, Materials Science, and Analytical Chemistry.

**KEYWORDS:** *Upper-Division Undergraduate, Inorganic Chemistry, Laboratory Instructions, Hands-On Learning/Manipulatives, Materials Science, Chemical Engineering, Solid State Chemistry, Atomic Spectroscopy, Infrared and NMR Spectroscopies, X-ray Crystallography*



## INTRODUCTION

Zeolites are microporous, crystalline aluminosilicate materials characterized by a three-dimensional porous framework of interconnected channels and cavities, whose dimensions are comparable to those of small molecules such as H<sub>2</sub>O, N<sub>2</sub>, O<sub>2</sub>, and CO<sub>2</sub> and simple hydrocarbons. The aforementioned structural properties, together with the presence of negative charges generated by the isomorphous substitution of Al for Si atoms, make these materials suitable candidates for many industrial applications such as heterogeneous catalysis, ion-exchange, and gas adsorption/separation.<sup>1</sup> Besides, the large amount of available zeolite structures with different Si/Al ratios (ranging from 1 to ∞) provides the possibility to select an appropriate material for a very specific target. Y zeolite, with a Si/Al ratio of about 1.5–3.0, belongs to the Faujasite family, together with X zeolite (Si/Al = 1–1.5). Y zeolite serves as a precursor for the preparation of ultrastable Y (USY) zeolites through dealumination, resulting in materials with a higher Si/Al ratio. These USY zeolites are widely employed in the petrochemical industry, particularly in processes such as Fluid Catalytic Cracking (FCC) and hydrocracking (metal/Y zeolite), for the production of gasoline and kerosene/diesel,

respectively.<sup>2</sup> These solid catalysts are a good example of the tremendous impact of heterogeneous catalysis in the outstanding advancements observed in the oil refinery sector over the last 50 years.

Although X and Y zeolites possess the same topology, they differ mainly in their Si/Al ratios. Generally, X zeolite can be obtained from a synthesis gel with a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio varying from 3 to 5. On the other hand, the Y zeolite crystallizes from a synthesis gel with a much higher SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub>, typically 10 or higher.<sup>3</sup> The two Faujasite zeolites are usually obtained under hydrothermal conditions, i.e., mild temperatures (50–150 °C) and alkaline media (pH above 9). In particular, the Si/Al ratio of the as-synthesized Y zeolite is a paramount parameter that will dictate the physicochemical properties of the final material: ion exchange capacity, acidity, etc. Its determination by either

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direct or indirect method is a relevant step for further postmodification treatments (e.g., dealumination) that intend to tune the final zeolite properties. This knowledge enables students to clearly relate their synthesis conditions to structural properties and catalytic performance, providing a rigorous understanding of how subsequent modifications can tailor Y zeolite for industrial applications. At least nine papers, published in the *Journal of Chemical Education*, have reported the preparation and/or application of zeolite materials, eight of which focused on the FAU structure.<sup>4–12</sup> Although these studies present interesting applications in catalysis, adsorption, and ion exchange, none provided a comprehensive characterization of the FAU samples. In particular, detailed determination of both the global and framework Si/Al ratios using various (direct and indirect) analytical methods has often been lacking. This work addresses this gap and introduces a laboratory activity in which students synthesize and characterize a NaY zeolite by employing several analytical techniques to determine its Si/Al ratio. The main objective of this laboratory activity is to help students understand the relationship between synthesis conditions, crystal structure, and physicochemical properties of zeolitic materials. Through hands-on laboratory sessions, students perform the hydrothermal synthesis of the NaY zeolite using different recipes, recover and characterize the products, and finally compare their results with those of a reference NaY sample. They learn to define the key synthesis parameters that influence crystallization and material quality and develop skills in the interpretation of experimental characterization data. Through integrated data analysis and discussion sessions, they use their findings to establish structure–property relationships and evaluate the key synthesis variables that control the properties of the final zeolite. The present results derive from synthesis protocols implemented in a Master's in Chemical Engineering laboratory course but can be easily adapted to other courses involving Inorganic Chemistry and/or Heterogeneous Catalysis. Ideally, students should also have a good background in Inorganic/Materials Chemistry (solid-state chemistry, sol–gel synthesis, precipitation) and basic knowledge of X-ray diffraction/crystallography and vibrational spectroscopy.

## ■ EXPERIMENTAL PROCEDURE

The following section summarizes the three different synthesis experiments used by the students for the preparation of the NaY materials. In all cases, hydrothermal synthesis was employed, while different recipes and crystallization conditions (microwave vs electrical heating) are attempted. The NaY materials obtained were then characterized, and the experimental results were analyzed and discussed by students on a collaborative basis, fostering a deeper understanding of how both chemical and physical parameters may influence the final zeolite properties.

The first synthesis recipe (**recipe 1**) employs the “seeding” method<sup>5</sup>, which is based on the separation of nucleation and growth processes<sup>4</sup>. It consists of the preparation of a seed gel that is further combined with a feedstock gel to form the final overall synthesis gel. This method promotes the formation of the desired zeolite phase and accelerates the crystallization of NaY crystals. **Recipe 1** is used here as a reference synthesis route, as it is commonly used in industrial zeolite manufacturing.<sup>3</sup> However, despite its scientific interest, the seed gel-based strategy involves multiple synthesis steps, making its implementation less suitable for laboratory classes. As an alternative, two simple synthesis methods (**recipes 2** and **3**) are proposed to undergraduate students.<sup>13,14</sup> In these cases, a single synthesis gel is prepared and then subjected to crystallization using either conventional electrical or microwave (MW) heating. For both alternative

recipes, the students explored different crystallization times and temperatures. The experimental results, collected over the past four years, enables the construction of the crystallization curves for **recipes 2** (70, 90, and 110 °C) and **3** (120 °C). **Recipe 4** is also presented as an example, allowing the preparation of the NaP1 phase which often competes with NaY since both crystallize under very similar conditions.

The **Supporting Information** file details all the different recipes used in this work (**section 1.4**). It also provides an example of an implementation plan for our laboratory course (**section 1.5**). Students are divided into groups of 3 people. They start with a 3 h introductory session about zeolites, covering their physicochemical properties, synthesis methods, and main applications. This is followed by one or more practical sessions (each lasting 3 h) depending on the synthesis recipe used. The instructor assigns the specific recipes, along with the corresponding crystallization times and temperatures, in order to prevent experiment duplication and to ensure sufficient experimental data for crystallization curve building. Note that the time required for each recipe changes a lot (**Figure S1**). **Recipes 1** and **3** are preferred when the number of students or groups is relatively small. In this case, each student's group performs all the synthesis steps (gel preparation, gel crystallization, and product recovery/drying) sequentially, over consecutive days if necessary. These two recipes require more than one 3 h session. Conversely, when the number of students and/or groups is larger, **Recipe 2** is recommended as it can be completed within a single 3 h session. There, the different groups can collaborate with each synthesis step for a given NaY sample carried out by a different group. This approach ensures that all the groups perform each synthesis step at least once (gel preparation, crystallization, recovery, and drying).

The NaY samples synthesized can be characterized by using a combination of different experimental techniques, including chemical analysis, powder X-ray diffraction (PXRD), Fourier-transform infrared (FTIR) and nuclear magnetic resonance (NMR) spectroscopies, and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS) analysis (**Supporting Information, section 2**). For PXRD and FTIR analysis, students prepare samples, collect the corresponding PXRD patterns and IR spectra, and interpret the results during a single 3 h session. For the other characterization methods, data are obtained by technical staff and provided to the students for analysis. An additional 3 h session might be scheduled to discuss these results. On the other hand, since some groups may obtain amorphous or semicrystalline NaY materials, a pure and highly crystalline NaY reference sample (synthesized using **Recipe 1**) is distributed to all groups for comparative characterization.

## ■ CHEMICAL REAGENTS

Sodium aluminate solid, NaAlO<sub>2</sub> (Carlo Erba, 54 ± 1 wt % Al<sub>2</sub>O<sub>3</sub>)

Sodium silicate solution, (NaOH)<sub>x</sub>(Na<sub>2</sub>SiO<sub>3</sub>)<sub>y</sub>·zH<sub>2</sub>O, Sigma-Aldrich, reagent grade (10.6 wt % Na<sub>2</sub>O, 26.5 wt % SiO<sub>2</sub>)

Colloidal silica, Ludox AS-40, Aldrich, 40.0 wt % in water.

Sodium hydroxide pellets, NaOH Sigma-Aldrich, 98.0%.

## ■ HAZARDS

The usual laboratory safety rules should be respected. In the case of zeolite synthesis, hot sodium hydroxide-containing solutions are generally handled. On the other hand, polypropylene-based bottles are used for the crystallization (hydrothermal) step. For these reasons, additional safety considerations are needed. Students and instructors are advised to read the chapter from H. Robson (safety considerations in zeolites synthesis, in *Verified Syntheses of zeolites*, second edition<sup>15</sup>).

Table 1. Details of the Learning Objectives and Expected Learning Outcomes

Learning objectives	Learning outcomes
The activity will give the students tools to understand and perform an hydrothermal synthesis and a basic characterization of a zeolite. Specifically, the student is expected to gain knowledge in:	The student should be able to identify and understand the effect of key parameters in the zeolite synthesis:
- Hydrothermal synthesis; supersaturation; Ostwald ripening; growth and nucleation processes; crystallization curves.	<b>Physical parameters</b> – time, temperature, heating source.
- Structure of zeolites; zeolites topology; pores shape and pores size; ion-exchange, adsorption.	<b>Chemical parameters</b> – nature of reagents, initial Si/Al ratio, pH.
- Infrared spectroscopy of zeolites; main building units' frequencies; structure-sensitive and structure-non sensitive bands.	The students should be able to assess materials properties through the collection of PXRD patterns, evaluation of zeolite purity, degree of crystallinity and framework Si/Al ratio.
- Basics in solid-state NMR spectroscopy applied to zeolites.	The student should be able to use and analyze the results of routine techniques used for zeolites characterization. In particular, the student should be able to (a) acquire an IR spectrum (KBr transmission mode) and analyze a NaY IR spectrum; (b) acquire and analyze a powder X-ray diffraction pattern; (c) analyze a solid-state NMR spectrum of a NaY zeolite.
- Basics in Electronic Microscopy applied to zeolites.	The students should be able to present a critical review related to zeolites synthesis.

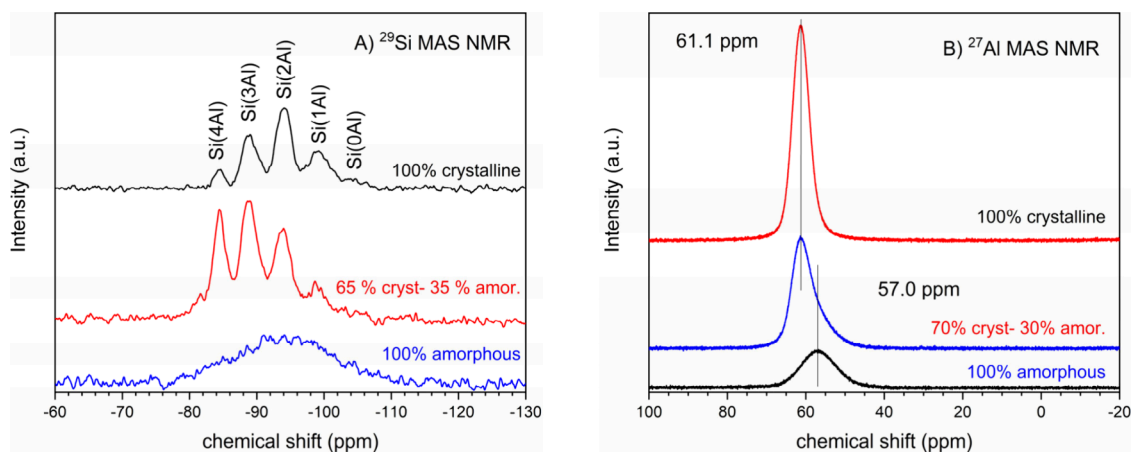


Figure 1.  $^{29}\text{Si}$  MAS NMR (A) and  $^{27}\text{Al}$  MAS NMR (B) of samples after 0.5 h (100% amorphous), 2 h (65% crystalline – 35% amorphous), and 3 h (100% crystalline). Experimental conditions: gel composition  $6.7\text{Na}_2\text{O}/1\text{Al}_2\text{O}_3/12\text{SiO}_2/220\text{H}_2\text{O}$ , crystallization at  $110\text{ }^\circ\text{C}$ , electric heating.

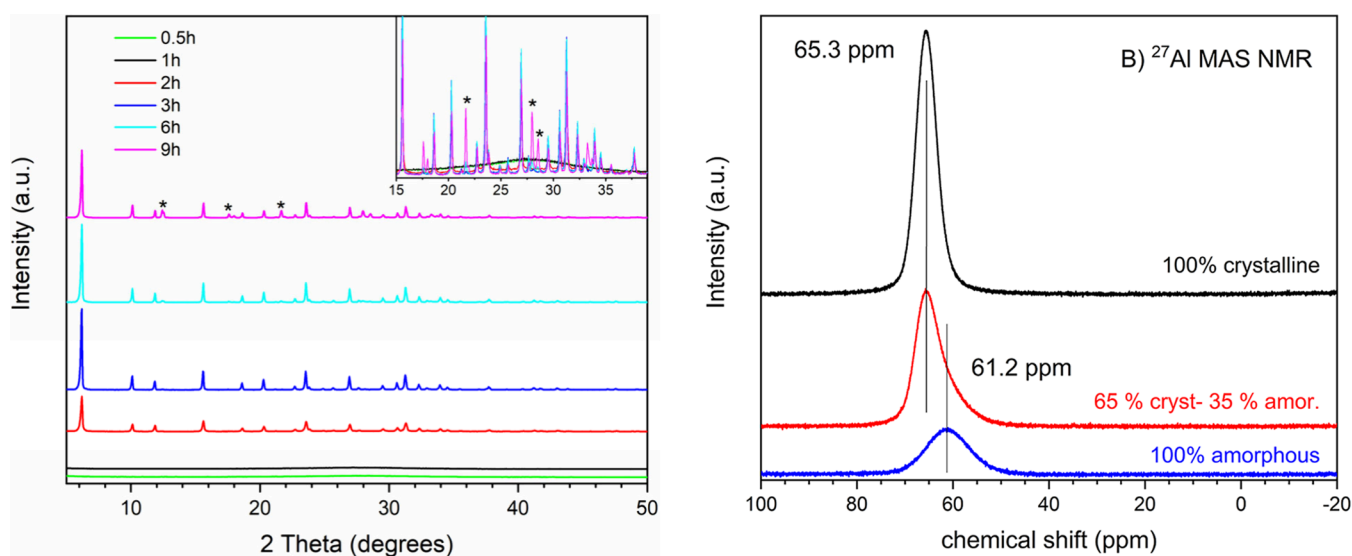


Figure 2. Powder X-ray diffraction patterns and respective crystallization curves for the samples heated at  $110\text{ }^\circ\text{C}$ . Experimental conditions: gel composition  $6.7\text{Na}_2\text{O}/1\text{Al}_2\text{O}_3/12\text{SiO}_2/220\text{H}_2\text{O}$ , electric heating (\* symbols denote NaP1 extra phase).

## RESULTS

### Students Learning Objectives and Outcomes

The synthesis and characterization of NaY zeolites provide students valuable educational experiences by integrating

fundamental concepts from Inorganic Chemistry and Materials Science while offering practical training in zeolite synthesis and routine characterization techniques such as powder X-ray diffraction and infrared spectroscopy. In particular, through

this laboratory approach, students gain deep knowledge in hydrothermal synthesis of microporous materials, crystallization, nucleation and crystal growth, Ostwald ripening, and the concept of crystallinity. Emphasis is placed on introducing and applying core characterization techniques for inorganic materials, namely, PXRD, FTIR, and NMR spectroscopies and SEM-EDS analysis. Moreover, this activity was also built to improve student's soft skills like critical thinking, creativity, and literature research. In that sense, students may also be encouraged to propose and explore alternative synthesis recipes, fostering a more inquiry-driven and research-oriented learning experience. The main learning objectives/outcomes are summarized in Table 1. The assessment plan used to evaluate/validate what the students learned is available in the Supporting Information (section 3).

### Monitoring the Crystallization of NaY Materials

One important learning outcome for students consists of determining the optimal conditions to obtain fully crystalline samples, free from any amorphous secondary phase. In this context, students explore how the various synthesis parameters can influence the nucleation and crystal growth processes. The students also evaluate the crystallinity of their material, which includes the construction and interpretation of the crystallization curves. NMR spectroscopy and PXRD diffraction are used to monitor the crystallization of the NaY materials. Figures 1 and 2 show the  $^{29}\text{Si}$  and  $^{27}\text{Al}$  MAS NMR and PXRD results obtained for the crystallization (at 110 °C) of the synthesis gel with the following composition:  $6.7\text{Na}_2\text{O}/1\text{Al}_2\text{O}_3/12\text{SiO}_2/220\text{H}_2\text{O}$  (Recipe 1). It is important to note that all experimental samples were produced by six different students' groups. Both techniques show that crystallization times of 0.5 and 1 h result in fully amorphous products. However, after 2 h of crystallization, characteristic diffraction peaks of FAU zeolite phase are clearly visible. The percentage of crystallinity calculated is about 65% (see Supporting Information, section 2.2, for further details on the calculation of the percentage of crystallinity for the different samples).

The complete crystallization only happens after 3 h of crystallization. For this sample, the PXRD pattern shows very intense diffraction peaks, and no trace of the amorphous phase is visible, while  $^{27}\text{Al}$  NMR shows a very sharp peak at about 61.1 ppm. The  $^{29}\text{Si}$  NMR spectrum of the 100% crystalline sample shows very well-defined peaks assigned to the different Si(nAl) environments present in the zeolite framework.

Interestingly, for longer crystallization times ( $t > 3$  h), the crystallinity of the NaY samples decreases, as a consequence of the appearance of an extra NaP1 phase (see Figure 2 and Figure S4). This result clearly illustrates Ostwald's rule of successive phase transformation. In fact, NaP1 phase can also be synthesized, directly, by slightly modifying the initial gel composition, that is, by decreasing slightly the  $\text{Na}_2\text{O}/\text{SiO}_2$  ratio.<sup>16</sup>

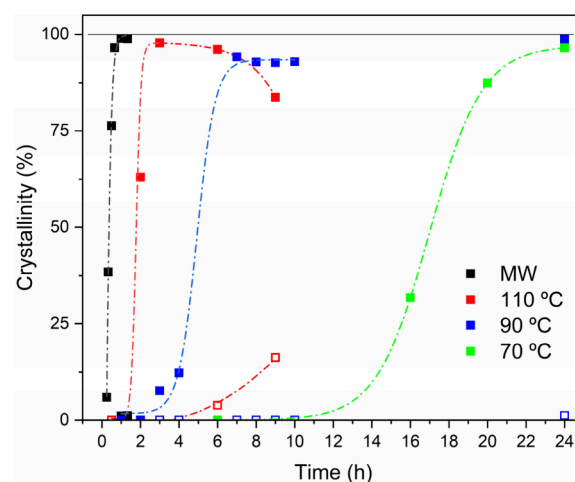
By analyzing both NMR and PXRD data, students can readily determine whether the NaY materials are purely crystalline materials or consist of a mixture of crystalline and amorphous phases. From the crystallization curves, students can (a) identify the induction period, defined as the interval during which no crystalline material is detected, making the separation between nucleation and crystal growth events and (b) establish the crystallization conditions necessary to obtain fully crystalline materials. Additionally, students are introduced to the Ostwald rule, which explains that over extended

crystallization times the thermodynamically most stable phase NaP1 gradually forms at the expense of the initially metastable NaY phase.

### Comparing the Different Synthesis Recipes

Students also learn to identify the different synthesis parameters (crystallization time and temperature, nature of the silicon source, and nature of the heating source) that affect the final physicochemical properties of the synthesized NaY materials. Depending on the class structure (e.g., number of groups and number of students per group) and the laboratory resources available (e.g., equipment, reagents, etc.), other syntheses, proposed by the students themselves, can also be used.

Figure 3 presents a comparison of the crystallization curves obtained for the different crystallization temperatures (corre-



**Figure 3.** Crystallization curves obtained from a gel composition ( $6.7\text{Na}_2\text{O}/1\text{Al}_2\text{O}_3/12\text{SiO}_2/220\text{H}_2\text{O}$ ) and crystallization temperatures of 70 (green squares), 90 (blue squares), 110 (red squares), and 120 °C (black squares) (closed symbol NaY, open symbol NaP1). Note that all the crystallizations were performed with the help of electrical heating, except at 120 °C where microwave was used as an auxiliary heating system.

sponding to Recipes 2 and 3). Each data point on the curves represents a sample prepared by a specific student's group. The construction of the crystallization plots is made possible through the collective sharing of experimental results among all students' groups. In all cases, a typical S-shaped crystallization curve is observed. However, as temperature increases from 70 to 110 °C, the induction period shortens, and the slope of the crystallization becomes steeper. This behavior reflects the enhanced nucleation and crystal growth rate, at higher temperatures, primarily due to the substantial increase in supersaturation of the reaction medium.<sup>17</sup> In the case of microwave-assisted NaY synthesis (see Figure 3), the two factors (MW heating and higher crystallization temperature) combine to severely reduce the NaY crystallization time, i.e., 0.67 instead of 3 h.

The scanning electron microscopy (SEM) images obtained for the different samples as a function of the crystallization temperature are displayed in Figure 4. Using the open-source software ImageJ, students analyzed the SEM images to determine particle size and size distribution. For each fully crystalline sample, measurements were taken from approximately 200 particles to ensure statistical relevance. The three

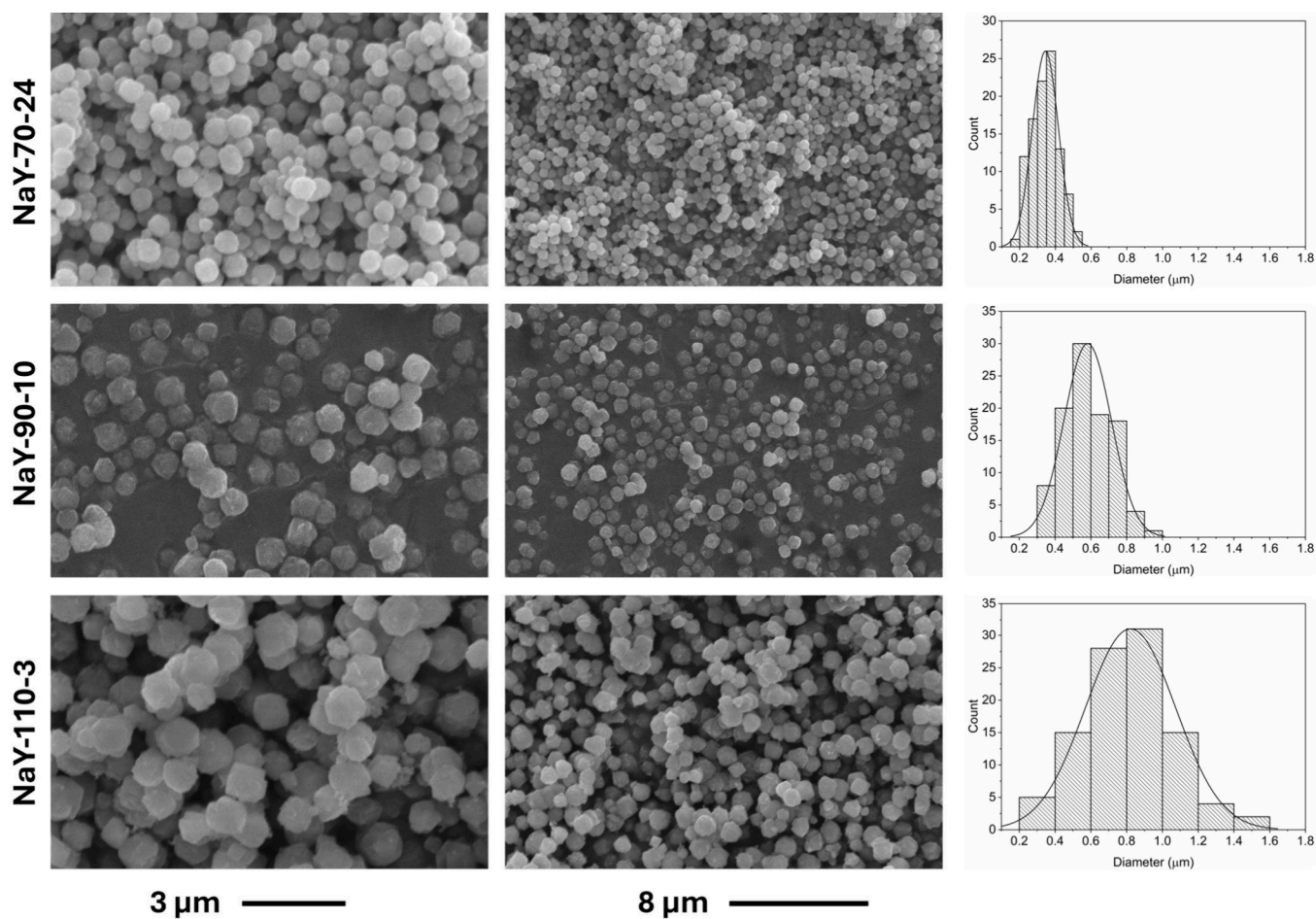


Figure 4. Scanning electron microscopy images of samples NaY-70-24, NaY-90-10, and NaY-110-3 with two magnifications and respective particle size distribution histograms.

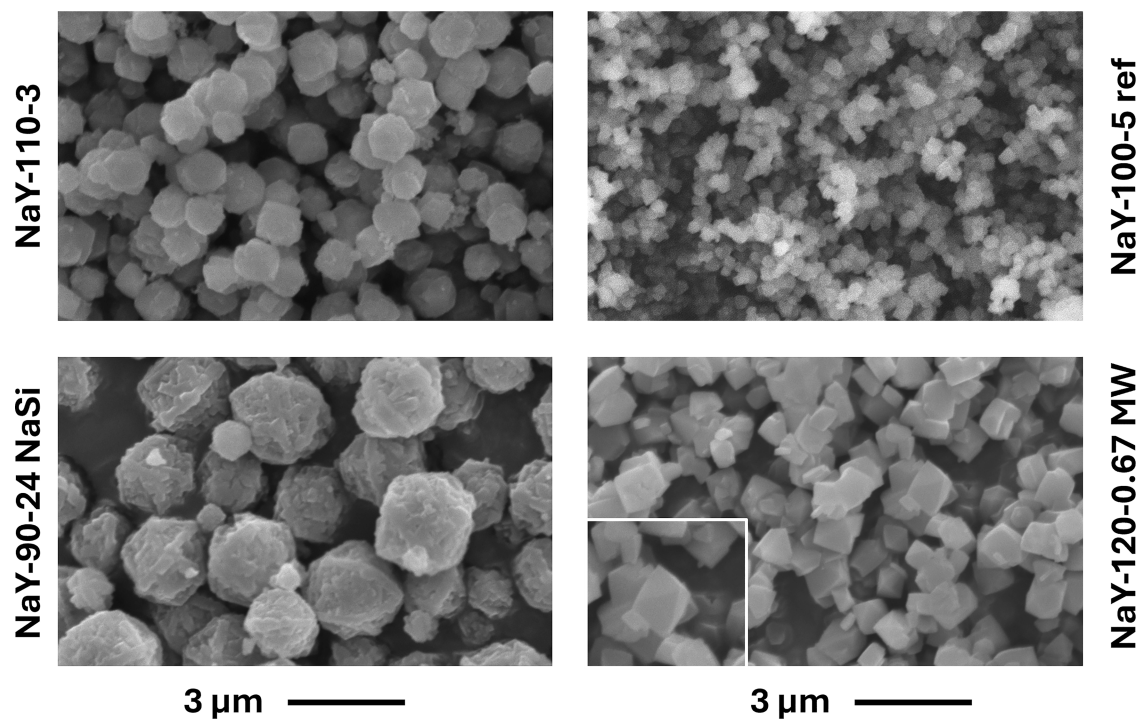


Figure 5. Scanning electron microscopy images of samples NaY-110-3, NaY-100-5 ref, NaY-90-24 and NaY-120-0.67 MW.

**Table 2. Experimental Synthesis Conditions for the Successful Preparation of Fully Crystalline NaY Materials, together with the Final Yield and Particle Size Achieved**

Sample	Si source	Heating <sup>a</sup>	Time <sup>b</sup>	Temperature <sup>c</sup>	Yield <sup>d</sup>	Particles size <sup>e</sup>
NaY-70-24	colloidal silica	CH	24	70	98	0.35–0.4
NaY-90-10	colloidal silica	CH	10	90	99	0.5–0–6
NaY-110-3	colloidal silica	CH	3	110	99	0.8–1.0
NaY-90-24 NaSi	sodium trisilicate powder	CH	24	90	94	nd <sup>f</sup>
NaY-120-0.67 MW	colloidal silica	MW	0.67	120	74	nd <sup>f</sup>
NaY-100-5 ref	sodium silicate solution	CH	5	100	98	0.1–0.2

<sup>a</sup>CH = conventional electrical heating; MW = microwave heating. <sup>b</sup>Time in hours. <sup>c</sup>Temperature in °C. <sup>d</sup>Al<sub>2</sub>O<sub>3</sub> basis. <sup>e</sup>μm (from SEM analysis). <sup>f</sup>nd: not determined.

**Table 3. Determination of the Si/Al Ratio for the Different NaY Samples Using Different Characterization Methods**

sample	Si/Al ratio						σ <sup>f</sup>
	C. A. <sup>a</sup>	EDS <sup>b</sup>	PXRD <sup>c</sup>	<sup>29</sup> Si NMR <sup>d</sup>	IR <sup>e</sup>	Average	
NaY-70-24	1.87	2.03	1.92	1.81	1.89	1.90	0.0811
NaY-90-10	1.93	2.14	1.97	1.97	1.96	1.99	0.0832
NaY-110-3	1.77	2.06	1.82	1.91	1.82	1.87	0.1134
NaY-90-24 NaSi	1.56	1.59	1.47	1.60	1.32	1.51	0.1169
NaY-120-0.67 MW	1.58	1.63	1.45	1.60	1.52	1.55	0.0716
NaY-100-5 ref	1.95	1.93	1.75	1.80	1.95	1.87	0.0942

<sup>a</sup>From elemental analysis. <sup>b</sup>From SEM-EDS experiments. <sup>c</sup>From Breck and Flanigen,<sup>20</sup> using the unit cell parameter  $a_0$ . <sup>d</sup>From <sup>29</sup>Si NMR deconvolution, according to ref 21. <sup>e</sup>From Fichtner-Schmittler et al.,<sup>22</sup> using the maximum in frequency of the asymmetric stretching T–O–T band; <sup>f</sup>Standard deviation. For more information concerning the different Si/Al calculations, please refer to the Supporting Information file.

samples exhibit similar morphology, characterized by irregular spherical particles, although the particle size varies significantly with crystallization temperature. The observed irregularity may indicate the presence of particle aggregates rather than isolated crystals.

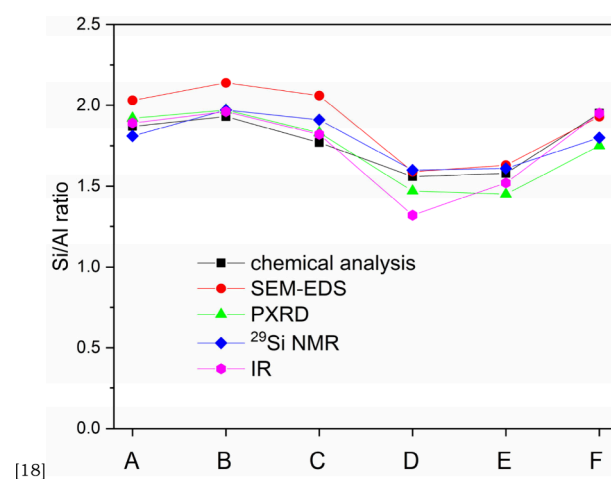
Figure 5 shows the SEM images of the NaY samples obtained from different crystallization conditions (MW or CH heating, Si source, etc.). Here, the results are very different as both particle size and morphology strongly depend on the crystallization conditions. Comparing the synthesis conducted with and without seeding, the results demonstrate the strong effect of seeds in decreasing the particle size during the crystallization step (sample NaY-100-5 ref vs sample NaY-110-3). On the other hand, the use of powder sodium silicate, instead of sodium silicate solution, results in the formation of larger aggregates. Finally, when microwave heating is applied during crystallization, the resulting materials consist of large and well-defined octahedral crystals, morphologically characteristic of NaY zeolites,<sup>18</sup> as illustrated in the inset of Figure 5. In this case, the gel composition is significantly diluted, leading to reduced supersaturation which favors crystal growth over nucleation, resulting in the formation of large single crystals.<sup>17</sup> Table 2 summarizes the different experimental conditions used to obtain 100% crystalline NaY materials together with information about the synthesis yield and particles size.

### Evaluation of the Si/Al Ratios

The third learning objective concerns the determination of the Si/Al ratio, a fundamental parameter of zeolites that critically influences their catalytic, ion-exchange, and adsorption properties. Both chemical analysis and EDS give a global Si/Al ratio while the other three methods, namely, PXRD diffraction (refinement of the unit cell parameter) and <sup>29</sup>Si NMR and infrared spectroscopies give exclusively the framework Si/Al ratio (tetrahedral Al(IV)). However, as seen in Figures 1 and S6, the <sup>27</sup>Al NMR spectra of all of the samples show only a single signal at about 61 ppm, characteristic of tetracoordi-

nated Al species, while no signal from extra framework Al species (around 30–40 and 0–10 ppm) can be observed. This proves that all the aluminum is present as the tetrahedral framework Al(IV) and that all the methods will give the same information, i.e., the framework Si/Al ratio. Table 3 summarizes the Si/Al values obtained by using the various characterization methods. Each sample (entry) was prepared by different student groups. As a valuable exercise, students could compare the Si/Al ratios obtained by each characterization technique across several fully crystalline samples (i.e., those synthesized by different groups) and analyze the variability or dispersion of the results.

Figure 6 plots the Si/Al data from Table 3. Two samples' groups can be distinguished: a first group with a relatively high



**Figure 6.** Si/Al ratio values (obtained from different characterization methods) for the different NaY samples: (A) NaY-70-24, (B) NaY-90-10; (C) NaY-110-3, (D) NaY-90-24 NaSi, (E) NaY-120-0.67 MW, and (F) NaY-100-5 ref.

Si/Al of about 1.9–2.0 and a second one with a lower Si/Al of ca. 1.5–1.6. Logically, the samples prepared with the same gel composition but different crystallization temperatures (NaY-70-24, NaY-90-10, and NaY-110-3, **Recipe 2**) show fairly the same Si/Al ratio, well in line with the values reported by Chaves et al.<sup>13</sup> who used similar chemical gel compositions and crystallization conditions.

Interestingly, **Recipe 1** (NaY-100-5 ref, reference synthesis in ref 5) yields a Si/Al ratio similar to the other recipes, despite the use of seeds during the preparation of the synthesis gel. For the last two samples (NaY-120-0.67 MW and NaY-90-24 NaSi), the Si/Al ratio is lower: 1.5 instead of 1.9 for the other samples. In both cases, the initial Na<sub>2</sub>O/SiO<sub>2</sub> ratio is higher, 16 (for NaY-120-0.67 MW) and 8.6 (for NaY-90-24 NaSi), against 6–6.7 for the other recipes. As a consequence, the higher gel synthesis pH, and hence the higher OH<sup>−</sup> concentration, might favor the formation of materials with lower Si/Al ratio.<sup>19</sup> For all of the samples, the Si/Al ratios obtained from the different characterization methods are indeed very similar, as denoted by the relatively low  $\sigma$  (see **Table 2**). Bearing in mind that all the characterizations were performed with the samples in their hydrated form, it becomes evident that there is a very good agreement between the different techniques in the determination of the Si/Al ratio.

## CONCLUSIONS

Thanks to the versatility and flexibility of the different synthesis approaches explored by our students, NaY materials with distinct characteristics can be readily obtained. These variations include differences in Si/Al ratios, crystal sizes, morphologies, and crystallization times. For example, microwave heating enables the formation of highly crystalline single NaY crystals within just 40 min of crystallization at 120 °C whereas conventional electrical heating requires 10 h at 90 °C to produce polycrystalline NaY particles. In all cases, the synthesis yields remain high, reaching approximately 100% (on the basis of Al<sub>2</sub>O<sub>3</sub>).

This laboratory experiment offers students a valuable opportunity to explore multiple techniques for determining the Si/Al ratio, helping them appreciate the advantages and limitations of each technique and understand their complementarity. Throughout the practical sessions, students are encouraged to think creatively and suggest alternative approaches for both the synthesis and the characterization of NaY zeolites. These open fruitful discussions between students and teachers foster curiosity and encourage collaborative learning. In conclusion, this laboratory experiment not only helps students in applying their knowledge of inorganic chemistry and materials science but also strengthens their practical skills in solid-state reactions and ultimately stimulates and develops their critical thinking.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.5c00851>.

Synthesis of NaY materials, equipment, chemical reagents, safety recommendations before starting, hydrothermal synthesis procedure, complete recipes, and characterization of the NaY materials (PDF, DOCX)

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### Notes

This study falls under a general institutional protocol for classroom activities, and no formal ethics review is required. The authors declare no competing financial interest.

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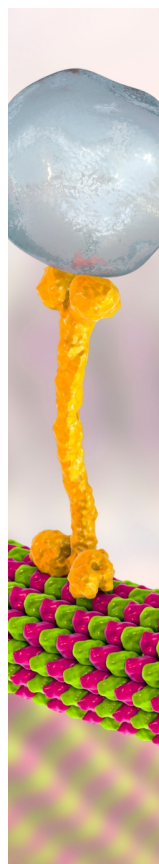
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