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SYNTHESIS AND CHARACTERIZATION OF ZEOLITE NaY USING KAOLIN WITH DIFFERENT SYNTHESIS METHODS

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Abstract—The benefits of using kaolin as a source of aluminosilicate in zeolite synthesis to obtain lower-cost catalysts, adsorbents, or ion exchangers are widely known. Previous attempts to produce zeolite from natural Iranian kaolin resulted in the formation of zeolites A, X, and HS. Zeolite Y plays an important role in the petrochemical industry due to its application in the area of fluidized catalytic cracking; ~40% of gasoline production is obtained using this process.

In the present study, different methods were used to prepare pure zeolite NaY from the Iranian kaolin available. The effects of different parameters such as aging time, crystallization time, kaolin calcination and crystallization temperature, and starting-material composition were investigated in order to obtain improved properties and maximize phase purity. In all cases, the crystal structure and microstructure were studied using X-ray diffraction and scanning electron microscopy. Among different synthesis approaches, the 'guide-agent method' resulted in the formation of zeolite NaY. The synthesis was generally sensitive to changes in kaolin calcination temperature and in hydrothermal synthesis parameters. The optimum parameters to prepare pure zeolite NaY were: kaolin calcination temperature = 680ºC, aging time of guide agent = 48 h without an overall gel aging step, and crystallization at 90ºC for 36 h.

Key Words—Kaolin, Synthesis Methods, Zeolite NaY.

INTRODUCTION

Zeolites are crystalline aluminosilicates with pores of molecular dimensions that are widely used in applications such as separation, catalysis, ion exchange, and adsorption (Song *et al*., 2005). Zeolite Y, a highly versatile member of the faujasite family, plays a significant role in the petrochemical industry (Sang *et al*., 2006) due to its application in the area of fluidized catalytic cracking (FCC) through which ~40% of gasoline production is obtained.

More than 40 natural zeolites have been identified over the past 200 years but no significant commercial uses were found for them until synthetic zeolites were discovered and developed. Modern studies of synthetic zeolites have focused mainly on the use of inexpensive initial or waste materials (Georgiev *et al*., 2013). Zeolites are usually synthesized from low-cost silica-alumina sources in alkaline phases under hydrothermal conditions. The low-cost silica-alumina sources are fly ash and kaolin (Htay and Oo, 2008).

Kaolin is a convenient source of Si-Al for synthesizing low-silica zeolites such as zeolite Y (Chandrasekhar and Pramada, 2004). One of the main routes for preparing zeolite NaY is the so-called '*in situ*' process from kaolin. Two steps are involved in this process: (1) dehydroxylation of kaolin to form an activated material known as metakaolin; and (2) hydrothermal treatment of metakaolin with aqueous alkali to form zeolite (Chandrasekhar, 1996). The keys to this process are the activation of the kaolin and the subsequent hydrothermal synthesis (Liu *et al*., 2003).

Previous attempts to produce zeolite from natural Iranian kaolin resulted in the formation of zeolites A, X, and HS (Farzaneh, 1989). The aim of the present study was to synthesize microsized NaY zeolite because of its properties, *e.g.* its highly catalytic center, large surface/atom ratio, large surface area, large surface energy, and better retro-wear. The FCC catalysts prepared from microsized NaY zeolite with a large Si/Al ratio possess high catalytic activity and can be used in the refining of residual and heavy oils. Use of microsized NaY zeolite has been reported (Bo and Hongzhu, 1998) to improve catalytic cracking selectivity, reduce coke formation, increase the yield of diesel oil, and promote gasoline quality; use in the fine-chemical industry has also been reported.

In the present study, attempts were made, at various temperatures (680−950ºC), using locally available Iranian kaolin, to prepare zeolite NaY from metakaolin. Four different synthesis approaches were tried. In all cases, the crystal structure and microstructure were

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studied by means of X-ray diffraction (XRD) and scanning electron microscopy (SEM).

EXPERIMENTAL

Materials

Kaolin was obtained from Kaolin Shargh Co. (Tehran, Iran). The chemical composition of the natural sample was determined by X-ray fluorescence (Table 1). The NaOH, sodium silicate ($SiO_2 \cdot Na_2O$), and sodium aluminate ($Al_2O_3 \cdot Na_2O$) were obtained from Merck Company (Whitehouse Station, New Jersey, USA) and used as received.

Apparatus

The crystal structure of the NaY powder synthesized was analyzed using CuKα radiation (Philips PW 1800, Amsterdam, The Netherlands).

The Fourier-transform infrared (FTIR) spectra of the samples were recorded using a Perkin-Elmer2000 FTIR spectrometer (Waltham, Massachusetts, USA). The particle size and morphology of the zeolites were examined using scanning electron microscopy-energy dispersive spectroscopy (SEM/EDS), using an XL30 instrument manufactured by Philips (Amsterdam, The Netherlands). The surface area of the zeolites was determined by the nitrogen adsorption method using a BELSORP-MINI II instrument (Toyonaka, Japan) in accordance with the Brunauer-Emmett-Teller (BET) method (Brunauer *et al*., 1938). The thermal stability of the zeolites was studied by thermogravimetric analysis using a TGA/DSC851e device (Mettler Toledo, Columbus, Ohio, USA) with heating rate of 10ºC/min from room temperature to 800ºC in the argon atmosphere.

Synthesis of zeolite NaY

For all four methods, the processes of synthesizing zeolite from kaolin share the same preparation procedure. This procedure began by converting kaolin to metakaolin. To accomplish this, the kaolin was activated at different temperatures; 680ºC for synthetic methods M_1-M_3, and 950ºC for method M_4. The sample was heated from 25 to 680 or 950ºC at a constant rate of 10ºC/min in air. When the calcination temperature was reached, the crucibles were left in the furnace for 3 h. Then, the crucibles were removed from the furnace and cooled in air. The conversion of the kaolin to metakaolin was confirmed by XRD analyses of the starting and thermally treated kaolin samples (Figure 1). The resulting metakaolins were then sieved through a 45 μm mesh and divided into four groups, ready for use in each of the four synthesis methods (M_1-M_4) to prepare zeolite NaY.

Table 1. Chemical analysis of the kaolinite mineral.

Component	Wt.%
SiO_2	57.66
Al_2O_3	30.71
Fe_2O_3	0.15
CaO	0.08
Na_2O	0.04
K_2O	0.01
MgO	0.001
TiO_2	0.079
MnO	0.001
P_2O_5	0.018
L.O.I*	10.88

* Loss on ignition.

Method 1 – alkali fusion method (M_1)

To synthesize zeolite by employing method 1 (Figure 2a), metakaolin synthesized by kaolin activation at 680ºC for 3 h was fused by the addition of NaOH at 550ºC for 3 h. Then, the solid products were ground with a fast mill (Pouyesh Sanat, Abyek, Iran), and sieved through a 45 μm mesh.

The resulting product was mixed with a calculated amount of sodium silicate and water (Table 2) to produce a system with molar composition SiO_2/Al_2O_3 = 5.3, Na_2O/SiO_2 = 0.25, and H_2O/Na_2O = 40. This material was kept for aging at room temperature for 24 h. After 24 h of aging, an aqueous pink gel appeared which was transferred to an autoclave, where the hydrothermal crystallization was carried out at 100ºC for 12 h. After this, the precipitate was filtered, washed several times with distilled water until pH = 9–10 (the pH of the water in equilibrium with the zeolite in the filter had already been measured), and then dried at 100ºC for 12 h.

Method 2 – synthesis without seed (M_2)

To synthesize zeolite using method 2 (Figure 2b), a calculated amount of sodium hydroxide, sodium silicate, and water were added to metakaolin (Table 3). These were mixed in order to achieve the same molar ratios as for method M_1 (SiO_2/Al_2O_3 = 5.3, Na_2O/SiO_2 = 0.25, and H_2O/Na_2O = 40). Then the mixture was aged at room temperature for 24 h to form a gel slurry. On heating at 100ºC, this gel slurry crystallized into zeolite over 36 h. After completion of hydrothermal crystallization, the resultant precipitate was separated and then

Table 2. Raw materials and their amounts used in the synthesis of the M_1 sample.

Raw materials	Amount (g)
Kaolin	32.21
NaOH	5.94
$Na_2O \cdot SiO_2$	71.4
H_2O	32.21

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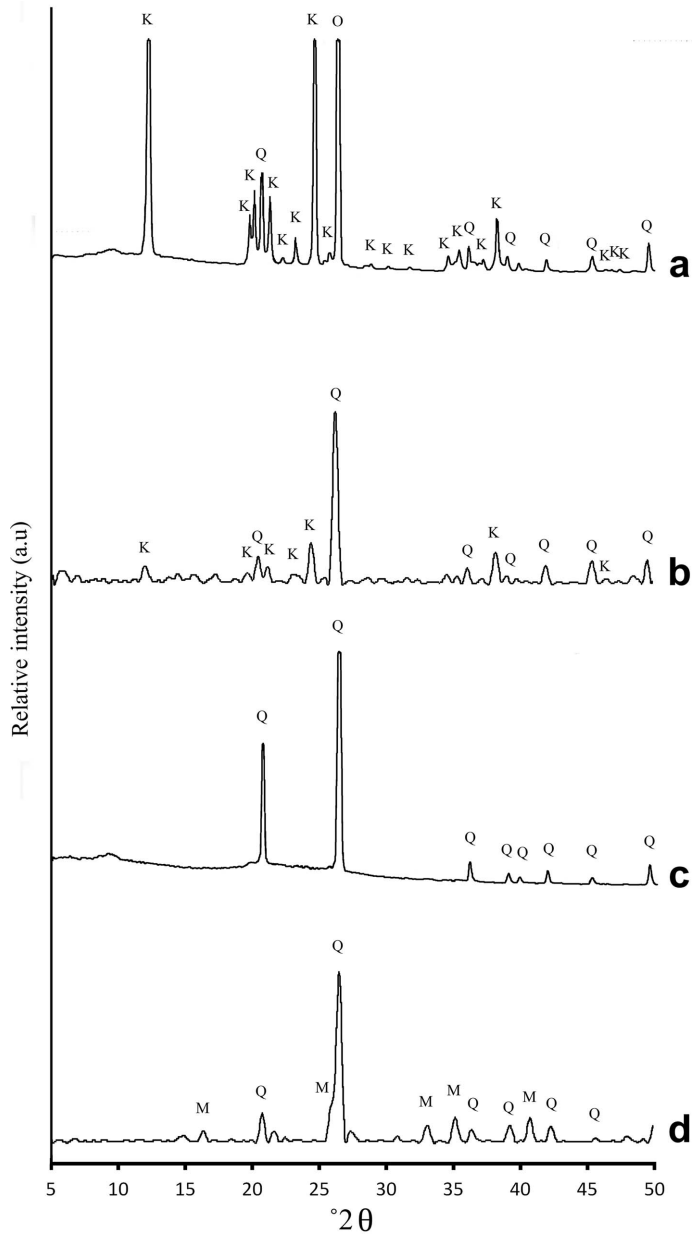


Figure 1. XRD patterns of (a) kaolin, and kaolin calcination obtained from kaolin activation at (b) 400ºC, (c) 680ºC, and (d) 1100ºC for 3 h. K: kaolinite, M: mullite, Q: quartz.

Table 3. Raw materials and their amounts used in the synthesis of the M_2 sample.

Raw materials	Amount (g)
Kaolin	29.513
NaOH	3.11
$Na_2O{\cdot}SiO_2$	66.942
H_2O	57.63

washed with deionized water until a pH range of 9−10, and dried at 100ºC for 12 h.

Method 3 − synthesis with zeolite NaY powder seed (M_3)

Preparation method 3 (Figure 2c) was similar to method 2, but differed in that zeolite NaY was used as seeding. The initial precursor was prepared by mixing the required amounts of metakaolin, zeolite NaY, sodium hydroxide, sodium silicate, and water (Table 4) to produce a system with molar composition SiO_2/Al_2O_3 = 5.3, Na_2O/SiO_2 = 0.25, and H_2O/Na_2O = 40, and then the zeolite NaY seed powder (3 g) was added to this mixture.

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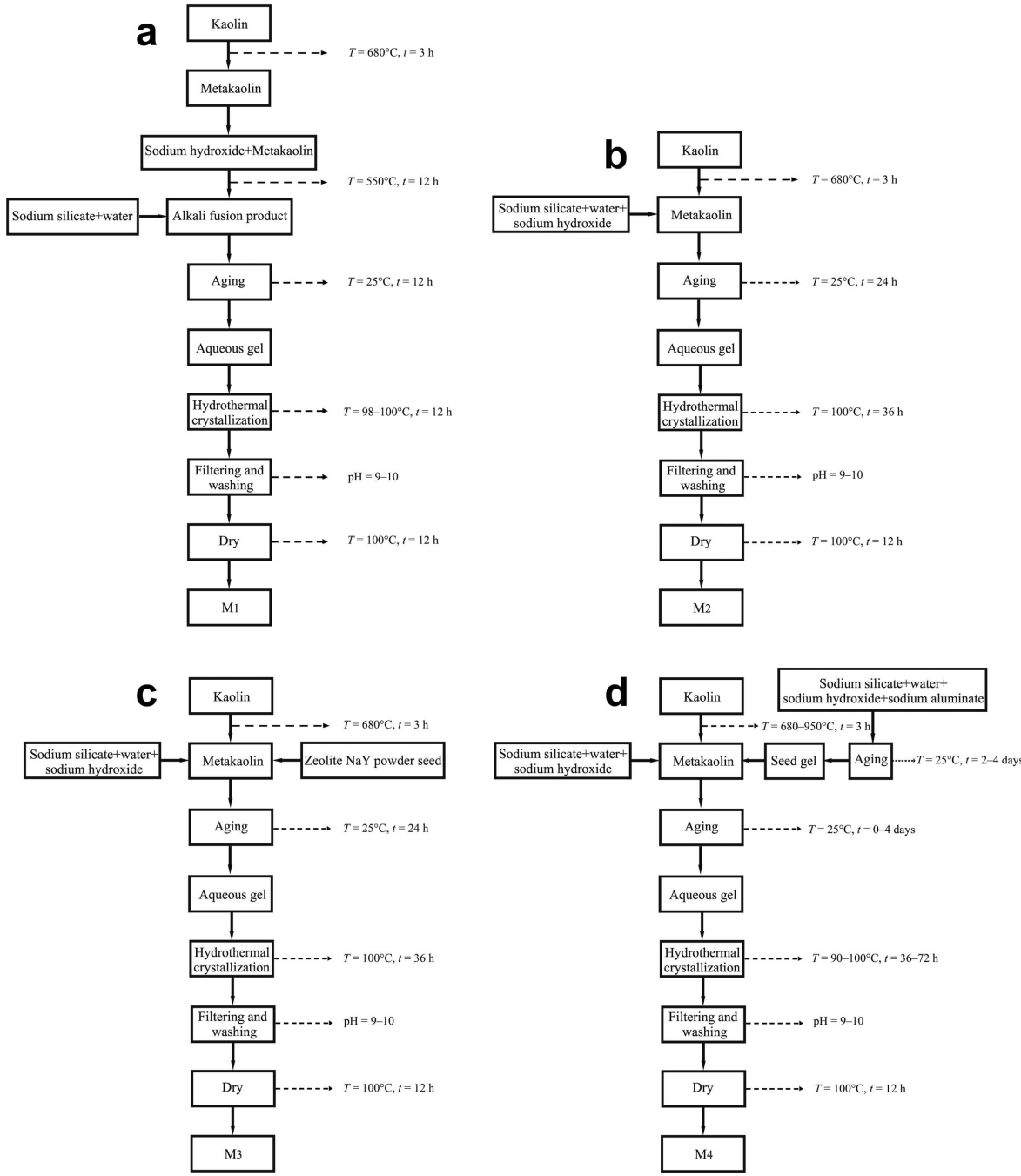


Figure 2. Different synthetic methods used to prepare zeolite NaY: (a) M_1, (b) M_2, (c) M_3, and (d) M_4.

Method 4 – guide-agent method (M_4)

In method 4 (Figure 2d), crystallization of zeolite NaY by the guide-agent method was also used and the effects of the different parameters such as kaolin calcination temperature, guide agent, and overall gel aging time, and crystallization time and temperature on the zeolite NaY preparation were studied in detail.

The metakaolins were prepared by heating the kaolin at 680 and 950ºC for 3 h in a muffle furnace. The guide agent was prepared from a mixture of sodium hydroxide, sodium silicate, and sodium aluminate (Table 5).

Firstly, the sodium aluminate solution was prepared by dissolving a calculated amount of the sodium aluminate in deionized water. This solution was then mixed with the sodium hydroxide and sodium silicate under constant stirring for 15 min. The resulting slurry was aged at room temperature for various times (2 and 4 h) to form a gel slurry.

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Table 4. Raw materials and their amounts used in the synthesis of the M_3 sample.

Raw materials	Amount (g)
Kaolin	29.513
NaOH	3.11
$Na_2O{\cdot}SiO_2$	66.942
H_2O	57.63
Zeolite NaY powder seed	3

The overall gel was prepared by mixing the calculated amount of sodium hydroxide, sodium silicate, metakaolin, water, and guide agent to produce a system with molar composition $SiO_2/Al_2O_3 = 5.3$, $Na_2O/SiO_2 = 0.25$, and $H_2O/Na_2O = 40$ (Table 6). Then the mixture was aged at room temperature to form a gel slurry. The gel-like mixture was transferred to an autoclave, where hydrothermal crystallization was carried out at different times and temperatures. The hydrothermal crystallization was also carried out immediately after the overall gel preparation without an aging step. Various guide-agent method conditions ($M_{4\text{-}1}$–$M_{4\text{-}8}$) were applied. To study the effect of the calcination temperature of kaolin on the quality of the final product, three calcination temperatures, 680ºC, 800ºC and 950ºC were investigated for $M_{4\text{-}1}$, $M_{4\text{-}4}$, and $M_{4\text{-}5}$, respectively. The other parameters were kept constant including the aging time of the guide agent (Figure 2d), 0 h of overall gel aging time, crystallization temperature (100ºC), and crystallization time (36 h).

Two different aging times of the guide agent, 2 h and 4 h, were studied for samples $M_{4\text{-}1}$ and $M_{4\text{-}6}$, respectively, under constant conditions including kaolin calcination temperature (680ºC), 0 h of overall gel aging time, crystallization temperature (100ºC), and crystallization time (36 h).

Overall gel aging time is the next parameter that influences the hydrothermal crystallization and so it was evaluated under two sets of different conditions: 0 h in $M_{4\text{-}1}$ and 4 h in $M_{4\text{-}7}$. Other parameters were constant including kaolin calcination temperature (680ºC), aging time of the guide agent (2 days), crystallization temperature (100ºC), and crystallization time (36 h).

Another parameter is crystallization temperature. Crystallization temperatures of 100ºC in $M_{4\text{-}1}$ and 90ºC in $M_{4\text{-}8}$ were examined. The other parameters are kaolin calcination temperature (680ºC), aging time of guide agent (2 days), 0 h overall gel aging time, and crystallization time (36 h).

To investigate the effect of crystallization time, hydrothermal crystallization was carried out at different crystallization times: 36 h in $M_{4\text{-}1}$, 50 h in $M_{4\text{-}2}$, and 72 h in $M_{4\text{-}3}$. Other reaction parameters including kaolin calcination temperature (680ºC), aging time of the guide agent (2 days), 0 h of overall gel aging time, and crystallization temperature (100ºC) were kept constant during this experimental series.

Table 5. Raw materials and their amounts used in preparation of the guide agent for M_4 samples.

Raw materials	Amount (g)
$Na_2O{\cdot}SiO_2$	10.2
NaOH	2.94
$NaAlO_2$	0.652
H_2O	13.26

Table 6. Raw materials and their amounts used in the preparation of the overall gel for M_4 samples.

Raw materials	Amount (g)
Kaolin	30
Guide agent	18
NaOH	1.03
$Na_2O{\cdot}SiO_2$	59.43
H_2O	43.379

RESULTS AND DISCUSSION

Properties of metakaolin

The effect of temperature on the kaolin calcination was examined at temperatures ranging from 400 to 1100ºC. Various XRD patterns of the clay and metakaolins were observed (Figure 1). The clay showed all the characteristic peaks of kaolinite (K) with quartz impurities (Q). On calcination, these peaks disappeared giving a featureless band of X-ray amorphous metakaolin. Kaolin calcination was indicated as complete at 680ºC. Heating at 1100ºC for 3 h resulted in the formation of new phase, mullite (M), which could not transform to zeolite NaY. For these reasons, the influence of kaolin calcination temperature on the formation of zeolite NaY was studied at the temperatures of 680 and 950ºC.

Comparison of the different synthesis methods in the formation of zeolite NaY

Different synthesis methods (M_1–M_4) were used to prepare zeolite NaY. The XRD results (Figure 3) show that the structure of the zeolites prepared from metakaolin depends significantly on the synthesis method chosen. When methods M_1 and M_4 were used, the characteristic peaks of zeolite NaY could exist together with the competitive phase of zeolite P, while, in the product samples M_2 and M_3, only the characteristic peaks of zeolite P were seen.

To prepare zeolite NaY, the guide-agent method was chosen because of the simple synthesis conditions required. Compared with the guide-agent method, the alkali fusion method was more time consuming and

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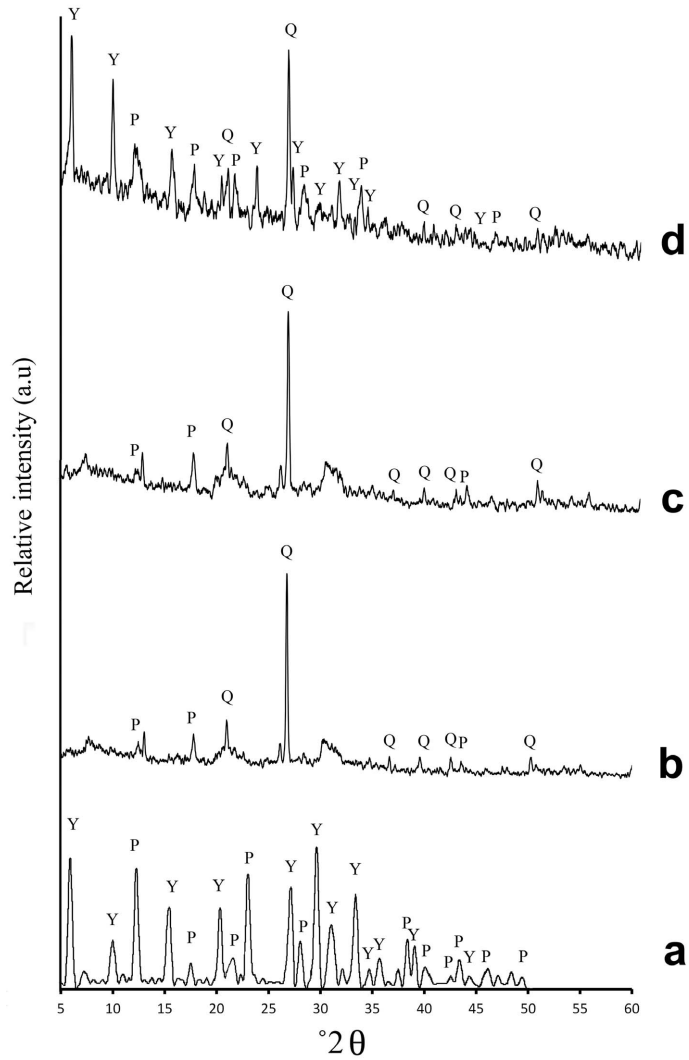


Figure 3. XRD patterns of products from the different synthesis methods : (a) M_1, (b) M_2, (c) M_3, and (d) $M_{4\text{-}1}$. P: zeolite P; Y: zeolite NaY, Q: quartz.

required a high calcination temperature; the residual fusion product was difficult to remove because it was tough and had to be ground.

Optimization of guide-agent method

To obtain a pure NaY phase, the effects of various parameters such as kaolin calcination temperature, guide agent, and overall gel aging time, crystallization time, and temperature ($M_{4\text{-}1}$−$M_{4\text{-}8}$) on the zeolite NaY formation were investigated.

The effect of kaolin calcination temperature

To investigate the effect of kaolin calcination temperature on the structure of the zeolites, comparative experiments at different kaolin calcination temperatures were carried out (Table 7, $M_{4\text{-}1}$, $M_{4\text{-}4}$, and $M_{4\text{-}5}$). The gel-preparation and hydrothermal-crystallization steps (Table 7) were similar but differed in terms of the kaolin-calcination temperature (680, 800, 950ºC for $M_{4\text{-}1}$, $M_{4\text{-}4}$, and $M_{4\text{-}5}$, respectively).

Clearly, the XRD results (Figure 4) show that when the starting kaolin was activated at 680ºC, the formation of zeolite P was competitive with zeolite NaY. When the kaolin calcination temperature was increased to 800ºC, the intensity of zeolite NaY was decreased and the formation of zeolite P dominated. The XRD patterns of solids obtained in the $M_{4\text{-}5}$ experiment, showed no zeolitic phases. These observations can be interpreted as follows: the kaolin structure transforms significantly to metakaolin ($2Al_2O_3{\cdot}4SiO_2$) at temperatures up to 500ºC. At 925ºC metakaolin layers condense to form a spinel-type phase of approximate composition $2Al_2O_3{\cdot}3SiO_2$ following removal of the silica (Brindley and Nakahira, 1959). On this basis, as the temperature increased to 800ºC, the transformation of metakaolin to the spinel resulted in the Si/Al ratio reduction, and so the

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Table 7. Different conditions for the guide agent-method (M_{4-1}–M_{4-8}).

Sample	Kaolin calcination temperature (ºC)	Aging time of guide agent (days)	Overall gel aging time (days)	Crystallization temperature (ºC)	Crystallization time (h)
M_{4-1}	680	2	0	100	36
M_{4-2}	680	2	0	100	50
M_{4-3}	680	2	0	100	72
M_{4-4}	800	2	0	100	36
M_{4-5}	950	2	0	100	36
M_{4-6}	680	4	0	100	36
M_{4-7}	680	2	4	100	36
M_{4-8}	680	2	0	90	36

formation of the low-silica zeolite P was dominant. On further increase of the temperature to 950ºC, metakaolin transformed completely to the spinel phase which was stable and could not be transformed to zeolite.

The effect of aging time

The effect of the guide agent and overall gel aging time on zeolite formation was also investigated in terms of the reduction of zeolite P formation (Table 7, M_{4-1}, M_{4-6}, and M_{4-7}).

To investigate the effect of aging time of the guide agent, two experiments were carried out for different aging times (48 h and 96 h) without an overall gel aging step. Zeolite NaY was obtained for an aging time of 48 h. Increasing the aging time of the guide agent to 96 h had a negative effect on the preparation of zeolite NaY; zeolite P became the dominant phase.

To study the effect of the overall gel aging time on zeolite NaY formation, a similar reaction was carried out with 48 h of aging time of the guide agent and 96 h of overall gel aging time. The XRD patterns of the comparative experiments (Figure 5) revealed that when the overall gel was aged, only the characteristic peaks of zeolite P were seen. Therefore, in the formation of zeolite NaY, the optimal aging time for the guide agent was 48 h without overall gel aging.

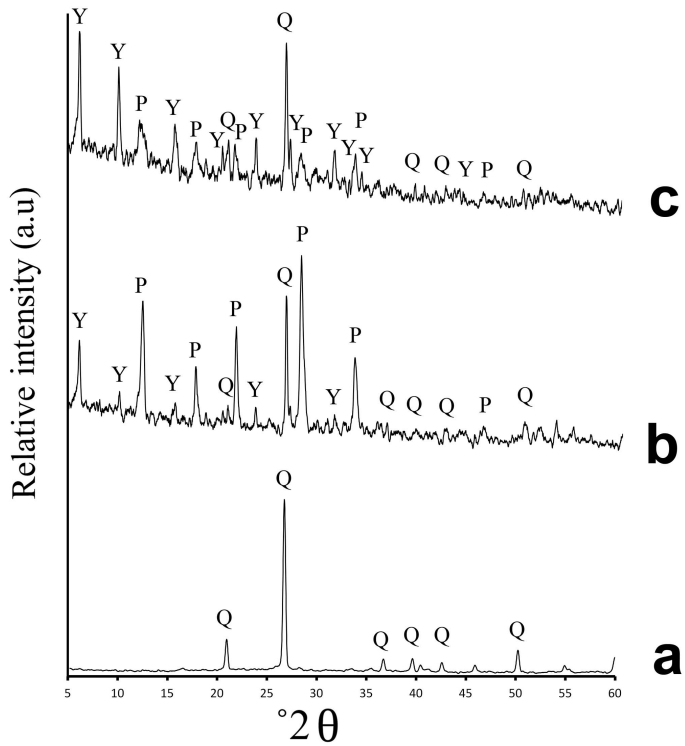


Figure 4. The effect of kaolin calcination temperature on XRD patterns of the products, crystallized at 100ºC for 36 h. Kaolin calcination temperature: (a) 950ºC M_{4-5}, (b) 800ºC M_{4-4}, and (c) 680ºC M_{4-1}. P: zeolite P; Y: zeolite NaY; Q: quartz.

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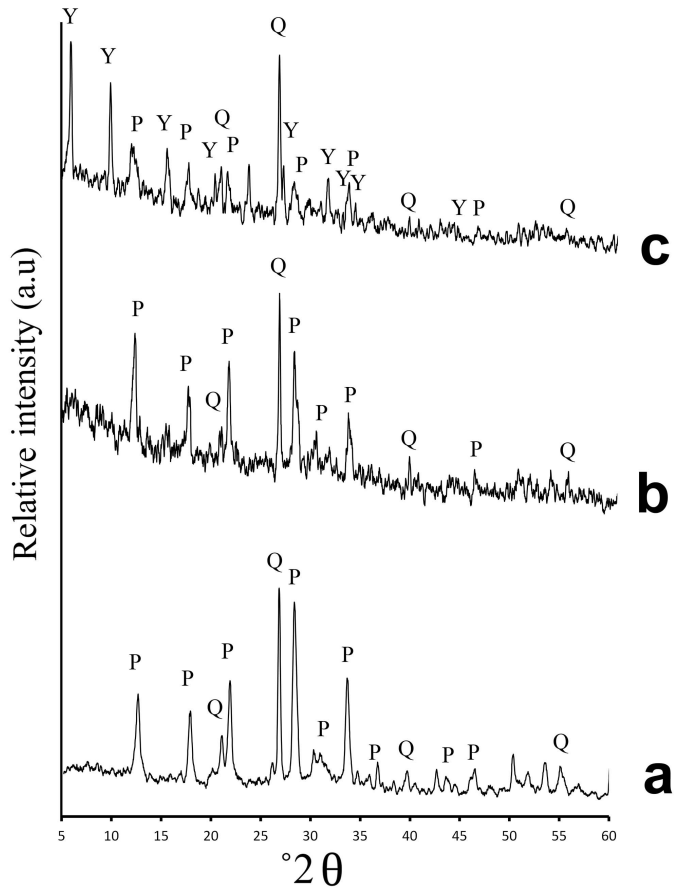


Figure 5. The effect of aging time on XRD patterns of the products, crystallized at 100ºC for 36 h: (a) aging time of the guide agent = 48 h, overall gel aging time = 96 h, $M_{4\text{-}7}$; (b) aging time of the guide agent = 96 h without overall gel aging step, $M_{4\text{-}6}$; (c) aging time of the guide agent = 48 h, without overall gel aging step, $M_{4\text{-}1}$. P: zeolite P; Y: zeolite NaY, Q: quartz.

The effect of crystallization time

Samples $M_{4\text{-}1}$, $M_{4\text{-}2}$, and $M_{4\text{-}3}$ were prepared at crystallization times of 36, 50, and 72 h, respectively, resulting in various XRD patterns (Figure 6). When the crystallization time was 36 h, zeolite P was formed as an impurity phase. Increasing crystallization time was found to favor the formation of zeolite P and pure phase P was formed after 72 h.

The effect of crystallization temperature

Attempts to prepare pure-phase zeolite NaY were continued by changing the crystallization temperature. The hydrothermal crystallization was carried out at 90 and 100ºC for 36 h. A significant increase in phase purity (Figure 7) was achieved by decreasing the crystallization temperature; consequently the zeolite NaY in pure phase was crystallized at 90ºC. As a result of these investigations, the optimum parameters used to prepare pure-phase zeolite NaY were: kaolin calcination temperature of 680ºC; aging time of the guide agent of 48 h (excluding an overall gel aging step), and crystallization at 90ºC for 36 h.

Characterization

Pure synthetic zeolite NaY was investigated here – the sample examined was obtained from method $M_{4\text{-}8}$ using a kaolin calcination temperature of 680ºC, an aging time of the guide agent = 2 days, 0 h of overall gel aging time, a crystallization time of 36 h, and crystallization temperatures of 90ºC.

The thermal degradation of the pure-phase zeolite NaY prepared was induced at a heating rate of 10ºC min^{-1} in an N_2 atmosphere (Figure 8). The DSC curve shows a ~26% weight loss between 100 and 400ºC that can be attributed to the loss of water.

The FTIR spectrum of zeolite NaY (Figure 9) shows the fundamental vibrations of the tetrahedral framework. The peak at 1009 cm^{-1} was assigned to the asymmetric stretching of the Al–O–Si chain of zeolite. The symmetric stretching and bending frequency bands of the Al–O–Si framework of zeolite appeared at 785 and 450 cm^{-1}, respectively (Zendehdel *et al.*, 2010). The appearance of the peaks at 569, 1654, and 3454 cm^{-1} was attributed to the double-ring external linkage, vibration of the water molecule, and

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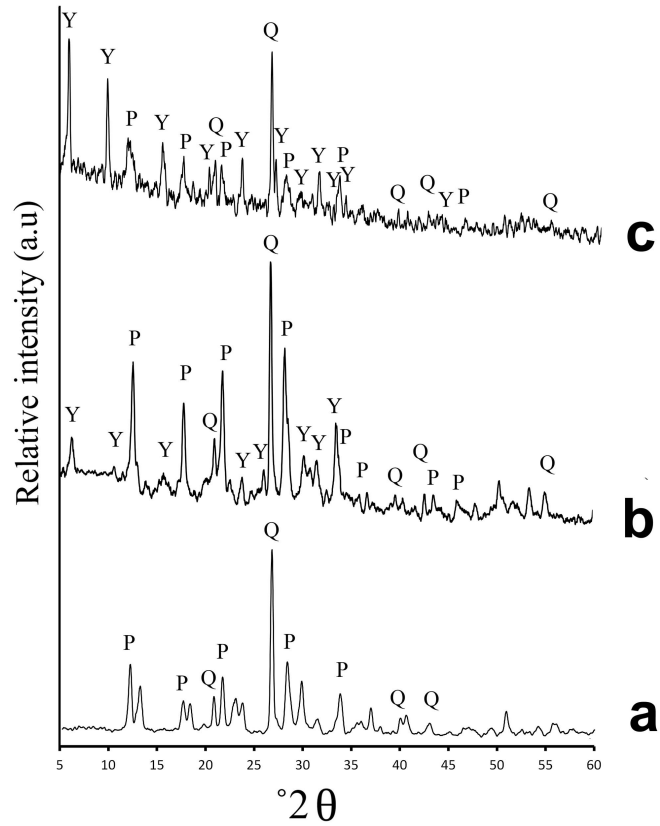


Figure 6. The effect of crystallization time on XRD patterns of the products, crystallized at 100ºC for: (a) 72 h, $M_{4\text{-}3}$; (b) 50 h, $M_{4\text{-}2}$; and (c) 36 h, $M_{4\text{-}1}$. P: zeolite P; Y: zeolite NaY, Q: quartz

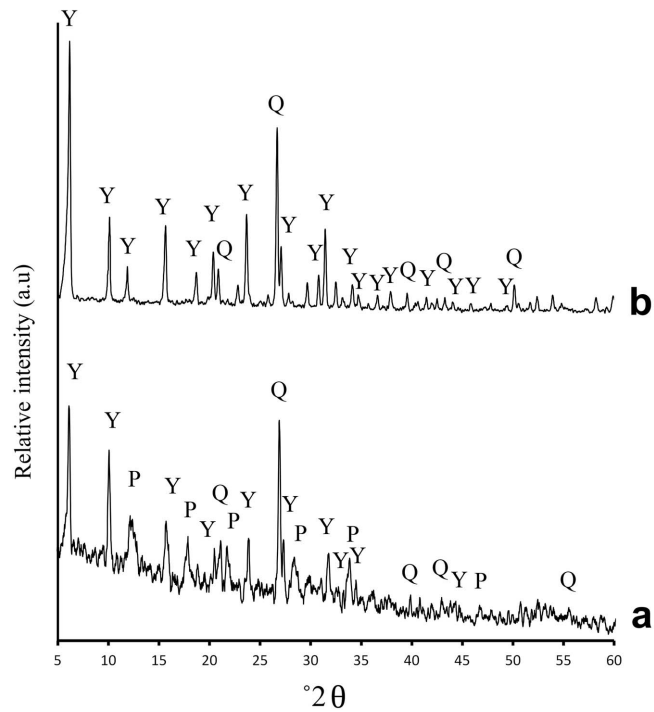


Figure 7. The effect of crystallization temperature on XRD patterns of the products, crystallized for 36 h at: (a) 100ºC, $M_{4\text{-}1}$; and (b) 90ºC, $M_{4\text{-}8}$. P: zeolite P; Y: zeolite NaY, Q: quartz.

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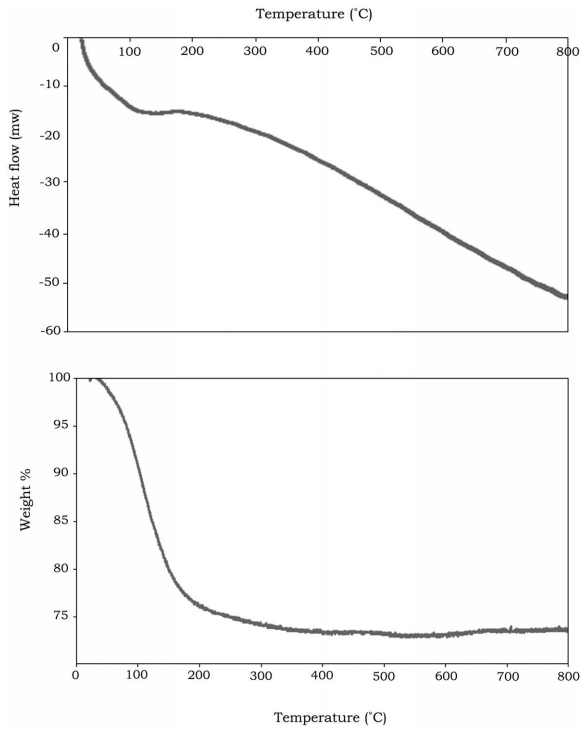


Figure 8. TGA thermogram of the zeolite NaY synthesized by the guide-agent method ($M_{4\text{-}8}$).

OH-stretching, respectively (Khabuanchalad *et al.*, 2008).

The mean value of surface area of the synthetic zeolite NaY was 638 cm^2/g (from BET analysis).

Figure 10 is an image of the typical kaolin used, which has a flakey structure. Figure 11 is an image acquired from the kaolin calcined at 680ºC. Clearly, the surface of the flakey structure is partially melted and represents the transformation from kaolin to metakaolin. The microstructure of the metakaolin is amorphous and

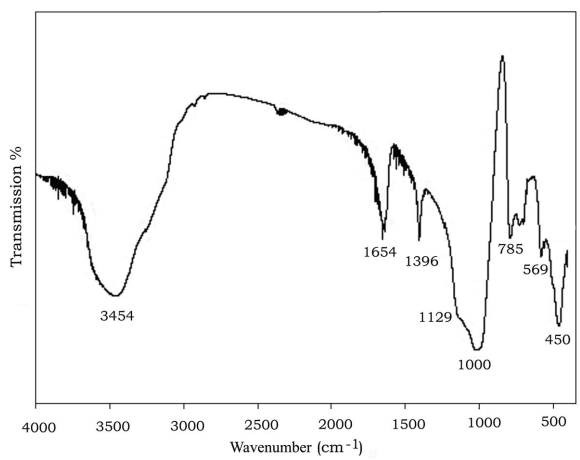


Figure 9. FTIR spectrum of the zeolite NaY synthesized by the guide-agent method ($M_{4\text{-}8}$).

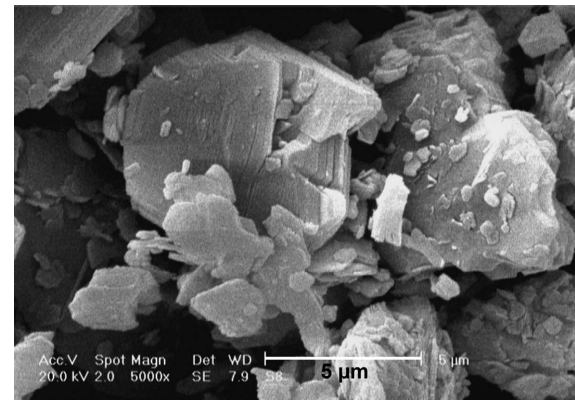


Figure 10. SEM image of kaolin.

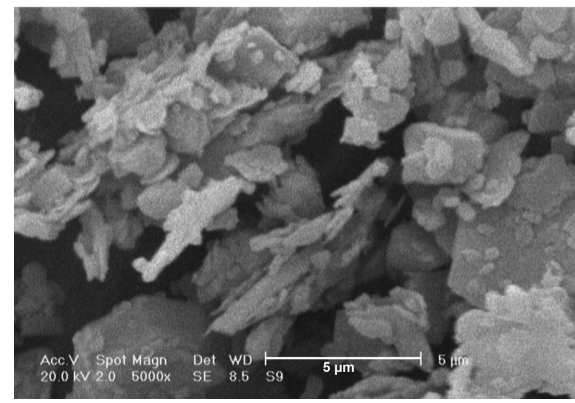


Figure 11. SEM image of calcined kaolin at 680ºC.

the XRD trace (Figure 1c) shows no peaks of any crystalline phase other than quartz (an impurity), which confirms the formation of the amorphous phase. During kaolin calcination, no other crystalline phases such as kaolinite and illite remain. Therefore, in the synthesis step, the zeolite crystals have must have been formed on

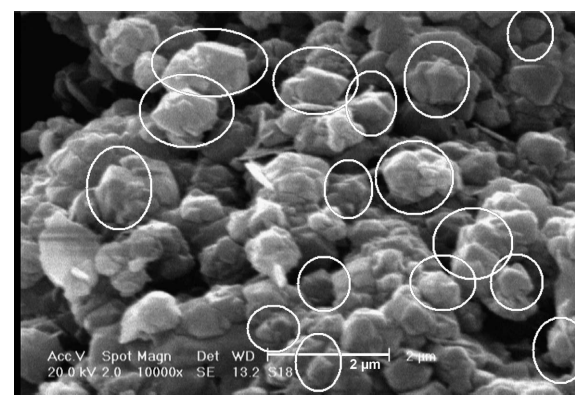


Figure 12. SEM image of the zeolite NaY synthesized by the guide-agent method ($M_{4\text{-}8}$). Circles identify subhedral grains with octahedral habit.

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Table 8. EDS spectrum of the zeolite NaY synthesized by the guide-agent method ($M_{4\text{-}8}$).

Element	Weight fraction	Mole fraction
Na	0.022	2.71
Al	0.1425	14.65
Si	0.835	82.64

the amorphous background. The SEM image of the synthetic zeolite NaY (Figure 12) shows octahedral crystals of zeolite NaY (which are marked with circles) ~0.8 μm wide.

To check the composition of the zeolite NaY synthesized, analysis by EDS was undertaken (the EDS spectra were acquired from the circles indicated in Figure 12). The Si/Al ratio of 2.8 obtained is in the range for zeolite NaY (Table 8). In addition, the strong Na signal from the EDS pattern indicates the successful formation of zeolite surface NaY and the absence of other impurities.

CONCLUSION

Pure zeolite NaY, with uniform, octahedral crystals up to 0.8 μm wide, was synthesized successfully from Iranian kaolin. The method consists of three steps: firstly, the guide agent was prepared; secondly an overall gel was prepared; finally, hydrothermal crystallization. The synthesis was generally sensitive to changes in kaolin calcination temperature and hydrothermal synthesis parameters. The formation of zeolite P was competitive with zeolite NaY during preparation.

The optimum parameters to prepare pure zeolite NaY were a kaolin calcination temperature of 680ºC, an aging time of the guide agent = 48 h with no overall gel aging step, and crystallization at 90ºC for 36 h.

Three other synthesis methods were tested, including alkali fusion, synthesis with NaY seed powder, and synthesis without seed. The results revealed that the synthesis method exerts a significant influence on the structure of the zeolites prepared from metakaolin.

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