



The static aging effect on the seedless synthesis of different ranges Faujasite-type zeolite Y at various factors

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Abstract

This work focuses on the formation of a pure phase Faujasite-type zeolite Y via a static aging step in the absence of seeds at the ranges chosen for this study. Nano-sized crystals of Y zeolite were successfully obtained when LUDOX AS-40 was used as a silica source for gel composition of range 6 and crystallization was conducted for a period of 4 to 19 hr at 100°C with surface area of 672 m²/g. Moreover, large-crystals with high crystallinity pure phase Y zeolite (with surface area of 682 m²/g) can be obtained here at the above conditions but when hydrous sodium metasilicate was used as a silica source. The most significant outcome of this study is that static aging effectively leads to obtaining a pure phase Y zeolite for all selected ranges at controlled conditions in a seedless synthesis.

Keywords: Faujasite-type, static aging, seedless synthesis, zeolite, hydrothermal treatment

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1- Introduction

Zeolites are microporous crystalline aluminosilicates minerals composed of open-framework structures containing channels and cages of molecular level which are accessible by other molecules [1, 2]. The fully cross-linked open framework of zeolites composed of SiO₄ tetrahedron and AlO₄ tetrahedron linked to each other by sharing oxygen atoms; and zeolites' channels and cages are occupied by water molecules [3]. Also, the cavities and cages of zeolites are occupied by cations such as sodium, calcium, potassium or organic cations to balance the negative charge generated due to the AlO₄ tetrahedron substitutions on the zeolite's framework [4, 5].

Zeolites are different types categorized either according to Si/Al ratio or framework structure [3]. Generally, Faujasite (FAU) zeolites are divided into two types which are zeolite NaX with low Si/Al ratio (below 1.5) and zeolite NaY with intermediate Si/Al ratio (1.5-3) [6] and can be greater than 3 [7].

Zeolite Y is a versatile used zeolite from the Faujasite family featured of a framework constructed of double 6-rings connected through sodalite cages. This connection generates large supercages with 12-ring aperture which is accessible by pores with an average diameter of 7.4 Å [8]. Typically, the unit cell of Y zeolite is cubic ($a = 24.7 \text{ \AA}$) with Fd-3m symmetry and thermally decomposes at 793°C [9].

Zeolite Y is an important intermediate and often high silica zeolite which is broadly used as an adsorbent, ion exchanger, catalyst and others [10-12].

Y zeolite has been extensively used as a catalyst for fluid catalytic cracking (FCC) and as a selective adsorbent for volatile organic and gaseous emissions removal and hydrocarbons purification [3, 15].

The vital role of Y zeolite in many chemical industries is due to zeolites' microporous structure providing excellent shape selectivity, large surfaces, strong Brønsted acid sites, favorable thermal and hydrothermal stability and reasonable low cost [13, 14].

Also, the open negatively charged framework containing easily exchangeable cations makes Y zeolite being utilized for potential environmental application such as heavy metal cations removal from industrial wastewater [16, 17].

Synthesis of zeolites encompasses of preparing aluminosilicate precursor solution from mixing of chemicals (aluminum source and silica source) under the alkaline conditions, aging the aluminosilicate solution and crystallizing the precursor solution for specific times at high temperatures [1, 18]. For some cases, aging step accelerates nucleation, enhances degree of crystallization and increases the silica to alumina ratio.

Aging works on incorporating more silica into aluminosilicate framework even at low level of alkalinity. Aging affects the kinetics of nucleation and crystallization through increasing the population of the final product and reducing the induction period and crystallization time [11, 19]. Kovo and Holmes [20] obtained that aging significantly increased the crystallinity of the final Y zeolite crystals.

Producing zeolite with desired properties depends on various important variables such as, alkalinity, the source of the precursor gel, aging, crystallization temperature and time, and organic additives (template), etc. Choosing the right experiment conditions achieves pure form of the desired zeolite and prevents transition from one structure/symmetry into another structure/symmetry, and emergence of the intermediate phases[8].

Therefore, several previous works [3, 9, 21, 22] recommended using seeds derived from Y zeolite either in the presence or absence of aging step for synthesizing pure phase Y zeolite. However, utilization of seeds made of counterpart phases to natural zeolites in the preparation adds cost to the cost of the preparation process.

For cost-effective preparation process of pure Y zeolite, this work investigates introducing static aging (ripening) step to prepare Y zeolite with a gel formula chosen from different ranges. Also, it deals with studying the effect of crystallization time and the silica source on the growth of Y zeolite.

2- Experimental Work

2.1. Chemicals

The used silica sources were sodium metasilicates (10.6% wt. Na_2O and 26.5% wt. SiO_2 , Sigma-Aldrich), fumed silica (99.8% wt. Sigma-Aldrich) and LUDOX AS-40 (Sigma-Aldrich). Alumina source was anhydrous sodium aluminate (55-56% wt. Al_2O_3 , Riedel-deHan) and sodium hydroxide (99.9% wt. from Fisher Scientific) was a cation source. Commercial NaY zeolite (Sigma-Aldrich) with Si/Al of 2.28 and an average crystal size of 1268.7 nm was used for comparison with products. Deionized water with conductivity of 2 μS was used for zeolite preparation.

2.2. Synthesis and Characterization of FAU-Type Zeolite Y

Preparation of Y zeolite samples was started with preparing alkali solution from mixing a certain weight of NaOH with a volume of deionized water. The quantities and volume of chemicals used for preparation were specified according to the formula of the mother gel chosen from a previous study[23]. Table 1 displays the chosen ranges for Y zeolite preparation.

Table 1. Ranges of Y zeolite chosen according to[23]

Range	$\text{SiO}_2/\text{Al}_2\text{O}_3$	$\text{Na}_2\text{O}/\text{SiO}_2$	$\text{H}_2\text{O}/\text{Na}_2\text{O}$
2	10-30	0.41-0.6	20-60
3	7-30	0.61-0.8	20-60
6	10-30	1.5-1.7	20-90
9	10-20	1.5-1.7	20-125
11	10-27	0.4	30-50

Then, the synthesis accomplished by adding alumina source to the alkali solution prepared previously and mixed until obtaining a clear solution. After that, silica source was added to the combination and mixed for 15 min. To study the effect of static aging, the produced gel was placed in an autoclave and left to age for 24 hr. The gel then was hydrothermally crystallized at 100°C for 19 hr. Also, the effect of crystallization time was investigated at 100°C for time range of 4-24 hr.

Also, one more sample was prepared by adding Y zeolite seeds to a sample of range 6 (R6) and mixed for 2 hours before crystallization without aging. The seeds were commercial Y zeolite (provided by Sigma-Aldrich) equals to 5% wt of silica source used to prepare the sample. The X-ray pattern, elemental analysis and morphology of commercial Y zeolite are shown in Fig. 1 and Fig. 2.

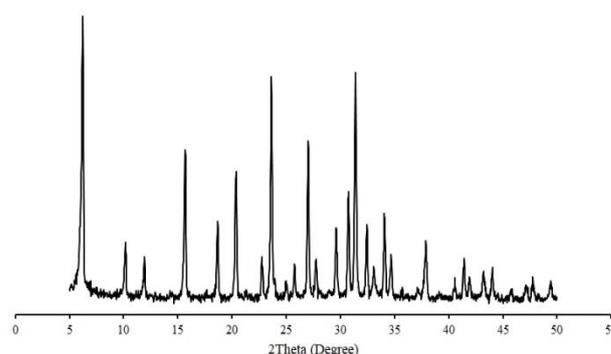


Fig. 1. X- Ray pattern of the commercial Y zeolite

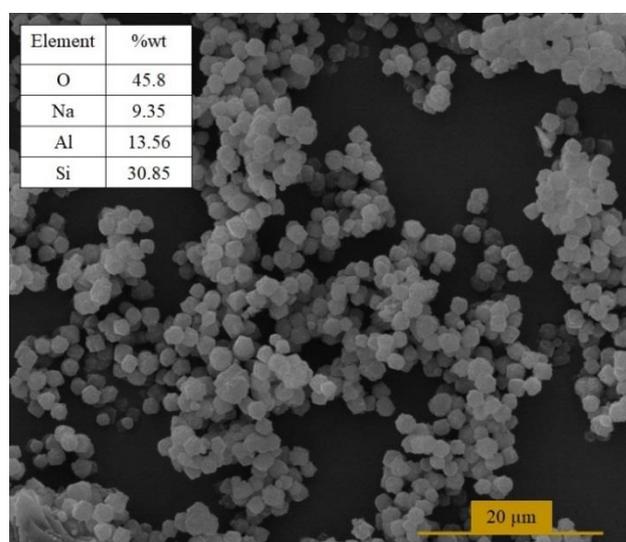


Fig. 2. SEM image of the commercial Y zeolite

Moreover, another sample was prepared according to [24]. This way encompasses preparing seeds gel and feedstock gel.

Then, the overall gel is obtained by adding a certain amount of seeds gel containing 5% of Al in the overall zeolite formula to a specific amount of feedstock gel containing 95% of Al in the overall zeolite formula.

Seeds Gel: 10.67 Na₂O: Al₂O₃: 10 SiO₂:180 H₂O (R5)
 Feedstock Gel: 4.30 Na₂O: Al₂O₃: 10 SiO₂: 180 H₂O (R2)
 Overall Gel: 4.62 Na₂O: Al₂O₃: 10 SiO₂: 180 H₂O (R11)

Once the crystallization completes, the products were separated, rinsed with deionized water to reduce the pH of the outlet water to below 9. After that, the products were dried 70-80°C, collected and characterized.

The phase of product was identified using X-ray diffraction (XRD) conducted via a RigakuMiniflex diffractor meter (CuK α radiation, 30 kV, 15 mA, $k = 1.5418 \text{ \AA}$). The morphology of the products was obtained using Scanning Electron Microscopy (SEM, FEI Quanta 200 Environmental). The composition of the elements composing the specimen was provided by the Energy Dispersive Analysis by X-ray (EDAX). The crystal size of samples was obtained using ImageJ software[25] depending on the SEM images.

The samples prepared from different silica source were also characterized using Fourier transform infrared (IR Prestige-21, Shimadzu) spectrophotometer to obtain Fourier transformed infrared (FTIR) spectrum.

The samples were scanned in the wavelength range of 4000 - 400 cm⁻¹. Also, the surface area was measured for those two samples using Brunauer-Emmett-Teller (BET) method based on the N₂-adsorption/desorption isotherms at -196°C. This characterization was conducted via Micrometrics ASAP 2020 after degassing at 200°C for 1 hr under vacuum pressure.

3- Results and Discussion

The results of this study have been compared to the data of commercial Y zeolite. Also, the crystallinity of all samples was calculated relative to commercial Y zeolite at the strongest peaks of 2 θ of 6.23, 15.65, 23.63, 27.02 and 31.4 according to Equation 1 as given by van Bekkum et al.,[26].

$$\text{Crystallinity \%} = \frac{\text{Intensity of peak samples}}{\text{Intensity of peak standards}} \times 100\% \quad (1)$$

Fig. 3 presents X-ray patterns of Y zeolite produced using three different ways. The three samples exhibited patterns identical to the commercial zeolite with no strange peaks.

However, the sample prepared using seeds gel showed higher crystallinity than a sample prepared using seeds powder, which can be attributed to the controlled conditions used to prepare the seeds gel compared to the conditions used to prepare the commercial zeolite. Also, aging the seeds solution for 24 hr allows formation enough nuclei for growing zeolite with ordered crystals as shown in Fig. 4 (b). Comparatively, the morphology of the sample prepared with powder seeds has irregular shape and size as shown in Fig. 4 (a). While, the sample prepared without seeds after static aging showed full crystallized and regular size crystals.

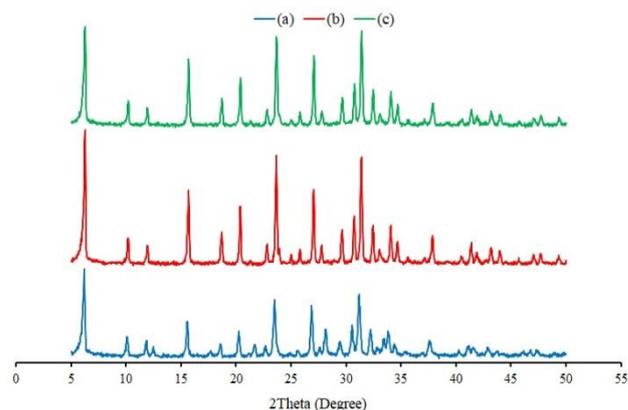


Fig. 3. X-ray pattern of Y zeolite prepared using (a) powder seeds of Y zeolite, (b) seeds gel of Y zeolite, (c) static aging without seeds. Crystallization temperature of 100°C and crystallization time 19 hr

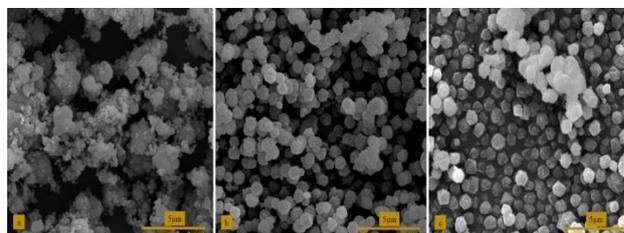


Fig. 4. SEM images of Y zeolite prepared using (a) powder seeds of Y zeolite, (b) seeds gel of Y zeolite, (c) static aging without seeds. Crystallization temperature of 100°C and crystallization time 19 hr

The average crystal size of the sample prepared using seeds powder is smaller than the average size of the sample prepared using seeds gel as shown in Table 2, which can be attributed to the large surface area provided by the seeds powder which promotes the nucleation, but short crystallization with large nucleation rate leads to different size crystals. However, aging the reaction gel for 24 hr promotes the quick formation of numerous nuclei and growing of small size crystals from the same nutrition[27]. Also, the synthesis way did not affect the Si/Al ratio of the produced zeolite as all possessed Si/Al > 1.5.

Table 2. Elements analysis and crystal size of Y zeolite prepared using different preparation ways

Synthesis technique	% wt O	% wt Na	% wt Al	% wt Si	Si/Al	Average crystal size (nm)	Crystallinity %
Powder seeds of Y zeolite	45.8	11.71	15.77	26.7	1.7	2737.75	75.15
Seeds gel of Y zeolite	47.6	7.47	13.61	31.3	2.3	2337.85	Above 100
Static aging without seeds	49.13	9.25	15.625	26	1.66	1545.8	100

The results displaying the effect of crystallization time on the final product are shown in Fig. 5, Fig. 6 and Table 3. The results given by XRD show that crystallization of Y zeolite at 4 hr produced full crystallized product because the pattern has a flat background.

The crystallinity of product started with 77.16% at 4 hr and then it increased with time until it reached to the maximum (100%) at 19 hr; after that it reduced at 24 hr. Ripening process of the gel allows quick gel conversion and crystallization within a short induction time at high crystallization temperature [19].

The average crystal size shown in Table 3 are close for different crystallization time periods because all were crystallized after aging which reduces the crystal size and increases the crystal population.

The effect of aging is predominant over the effect of time. This phenomenon occurred for all except for the crystallization of 4 and 14 h, they showed smaller but approximately similar average crystal size (473.35 and 455.4 nm, respectively).

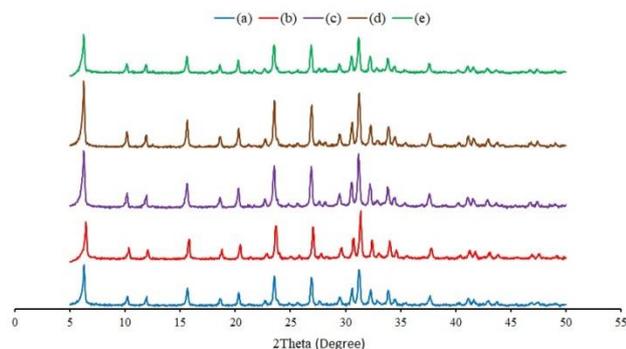


Fig. 5. X-ray pattern of Y zeolite (R6) prepared from LUDOX AS-40 at static aging without seeds conditions and crystallization temperature of 100°C for (a) 4 hr, (b) 9 hr, (c) 14 hr, (d) 19 hr, (e) 24 hr

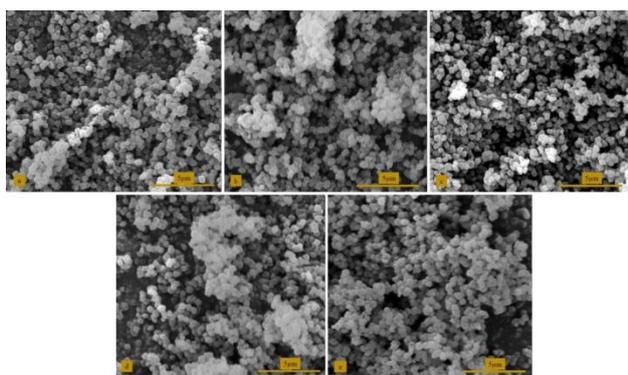


Fig. 6. SEM images of Y zeolite (R6) prepared from LUDOX AS-40 at static aging without seeds conditions and crystallization temperature of 100°C for (a) 4 hr, (b) 9 hr, (c) 14 hr, (d) 19 hr, (e) 24 hr

Table 3. Elements analysis and crystal size of Y zeolite (R6) prepared using static aging without seeds conditions at different crystallization time

Crystallization time (hr)	%wt O	%wt Na	%wt Al	%wt Si	Si/Al	Average crystal size (nm)	Crystallinity%
4	45.56	9.05	16.29	29.01	1.78	473.35	77.16
9	48.04	9.5	15.12	27.34	1.81	558.25	86
14	46.59	8.75	15.98	28.68	1.8	455.4	100
19	49.13	9.25	15.625	26	1.66	568.95	100
24	47.2	8.77	15.54	28.5	1.83	558.65	75.28

Fig. 7, Fig. 8 and Table 4 present the effect of aging on crystallization of different ranges of Y zeolite. XRD patterns shown in Fig. 7 shows successful crystallization was obtained for the chosen ranges because no peak irrelevant to Faujasite appeared for all samples. This result was confirmed by EDAX results presented in Table 4, since all samples possess Si/Al > 1.5; but, they ended with different average size of crystal. The product of R11, R2 and R3 gave larger sizes of 852.8, 820.3 and 811.2 nm, respectively; followed by R9 with an average crystal size of 705.25 nm and the smaller average size was obtained by R6 of 568.95 nm. Accordingly, it is worth to mention that aging helped for obtaining nanosized crystals for the chosen ranges of Y zeolite.

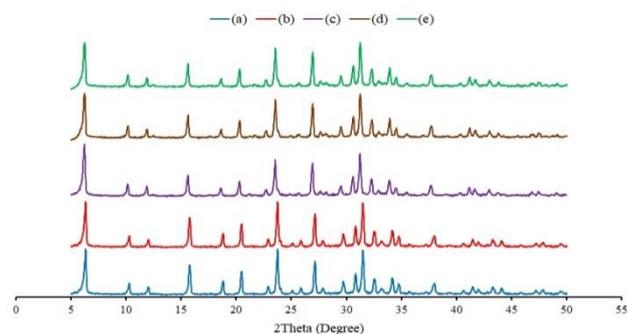


Fig. 7. X-ray pattern of Y zeolite prepared from LUDOX AS-40 at static aging without seeds conditions and crystallization temperature of 100°C for 19 hr. (a) Range 11, (b) Range 6, (c) Range 9, (d) Range 2, (e) Range 3

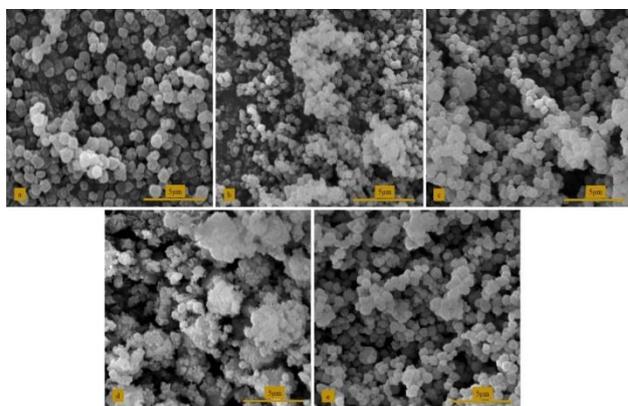


Fig. 8. SEM images of Y zeolite prepared from LUDOX AS-40 at static aging without seeds conditions and crystallization temperature of 100°C for 19 hr. (a) Range 11, (b) Range 6, (c) Range 9, (d) Range 2, (e) Range 3

Table 4. Elements analysis and crystal size of Y zeolite prepared using static aging without seeds conditions for different Y zeolite ranges

Zeolite Y range	%wt O	%wt Na	%wt Al	%wt Si	Si/Al	Average crystal size (nm)	Crystallinity%
Range 11	51.08	7.45	12.55	28.0	2.3	852.8	96.36
Range 6	49.13	9.25	15.625	26	1.66	568.95	90
Range 9	47.56	6.55	13.55	32.34	2.4	705.25	89
Range 2	48.22	9.22	15.11	27.44	1.82	820.3	93
Range 3	46.54	6.84	12.685	33.94	2.68	811.2	61

The results presenting the effect of silica source on the growing zeolite prepared with R6 are shown in Fig. 9, Fig. 10 and Table 5. The XRD pattern shown in Fig. 9 indicates that Y zeolite grown from hydrous sodium metasilicates showed higher crystallinity than this given by LUDOX AS-40. This can be attributed to its occurring with low supersaturation which leads to grow large crystals at the expense of nucleation. Thus, the sample prepared from hydrous sodium metasilicates produced larger size crystals as shown in Table 5 while, using fumed silica as a silica source produced glassy solid with no defined phase, therefore its pattern was not presented here.

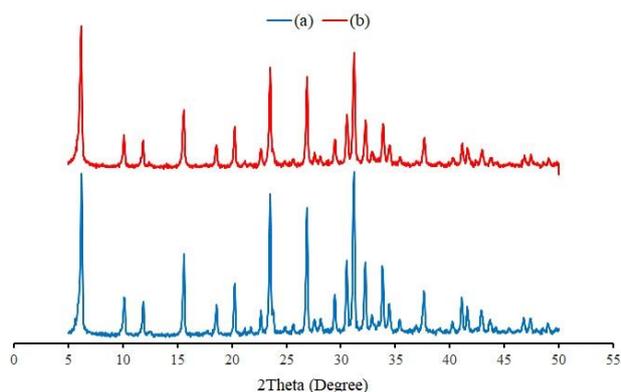


Fig. 9. X- ray pattern of Y zeolite prepared with R6 using (a) hydrous sodium metasilicates, (b) LUDOX AS-40. Crystallization temperature of 100⁰C and Crystallization time 19 hr

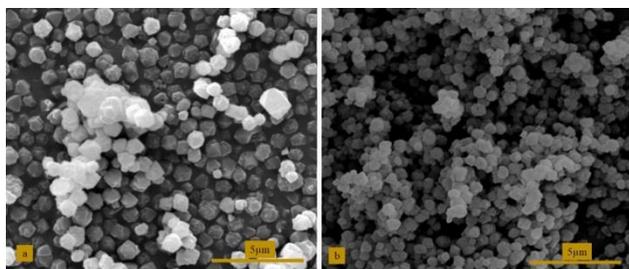


Fig. 10. SEM images of Y zeolite prepared with R6 using (a) hydrous sodium metasilicates, (b) LUDOX AS-40 crystallization temperature of 100⁰C and Crystallization time 19 hr.

Table 5. Elements analysis and crystal size of Y zeolite prepared with R6 using different silica sources

Silica source	%wt O	%wt Na	%wt Al	%wt Si	Si/Al	Average crystal size (nm)	Crystallinity%
Hydrous sodium metasilicates	46.6	8.74	15.8	28.83	1.82	1545.8	Above 100
LUDOX AS-40	49.13	9.25	15.625	26	1.66	811.2	100

FTIR spectra of both samples prepared from hydrous sodium metasilicates and LUDOX AS-40 are shown in Fig. 11 and Fig. 12, respectively. Generally, more bands vibrated for the samples prepared from LUDOX AS-40. Several peaks are common between the samples which are 1384.89, 999.13, 690.52, 617.22, 570.93, 528.50 and 478.35 cm⁻¹.

The figures show broad peaks appeared at 3479.58 and 3468.01 cm⁻¹ due to the presence of O-H stretch. Also, three peaks appeared at the range of 160-3500 cm⁻¹ for the sample prepared from hydrous sodium metasilicates as shown in Fig. 11 and four peaks appeared at the same range here in above for the samples prepared from LUDOX AS-40 as shown in Fig. 12 due to the presence of zeolite water.

The sample prepared from LUDOX AS-40 showed a peak at 1246.02 cm⁻¹ attributed to vibration of internal Si-O-Si and Si-O-Al tetrahedron. Due to the vibration of the same bonds (Si-O-Si and Si-O-Al tetrahedron), both samples showed peaks at 999.13 cm⁻¹. Bands appearing in the range 650-900 cm⁻¹ are related with symmetric stretching vibration of T-O tetrahedron. In the bands range 520-650 cm⁻¹, a group of peaks appeared due to pseudo-lattice vibration. However, the peaks appeared in the range of 400-500 cm⁻¹ are associated with T-O symmetric stretching of the internal Si-O-Si and Si-O-Al tetrahedron.

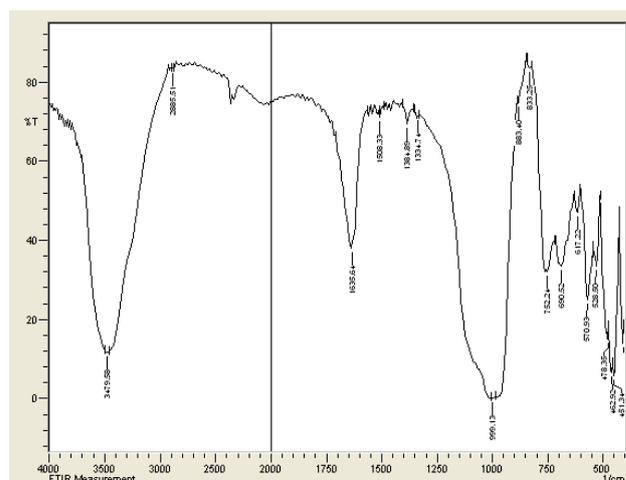


Fig. 11. FTIR spectrum of Zeolite NaY prepared with R6 using hydrous sodium metasilicates and crystallized at 100 °C for 19 hr

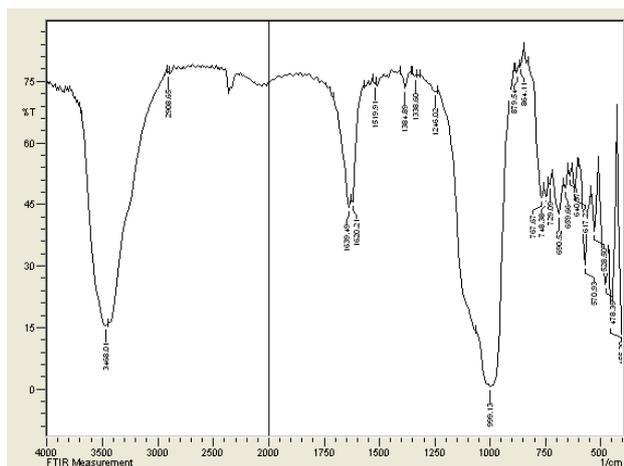


Fig. 12. FTIR spectrum of Zeolite NaY prepared with R6 using LUDOX AS-40 and crystallized at 100 °C for 19 hr

The surface area measured for the sample prepared from hydrous sodium metasilicate was 682 m²/g and for the sample prepared from LUDOX AS-40 was 672 m²/g. This high surface area of these samples which can be attributed to the high crystallinity of samples is industrially favorable and confirms the feasibility of the aging step for Y zeolite preparation. Both samples possess close surface area, but the sample prepared from hydrous sodium metasilicate gave relatively higher surface area which is compatible with its higher Si/Al ratio; in spite of it has larger average crystal size than the sample prepared from LUDOX AS-40.

4- Conclusions

This study presents the possibility of producing pure phase Faujasite-type zeolite Y at the ranges disclosed herein at a static aging condition in the absence of seeds. Wherein, nano-sized crystals can be obtained when LUDOX AS-40 is used as a silica source for gel composition of the preferred range 6 and the crystallization process can be carried out for a period of from 4 to 19 hr at 100°C. Also, high crystalline large-crystals pure phase Y zeolite can be obtained at the same conditions but when hydrous sodium metasilicate is used as a silica source. However, the other selected ranges can offer pure phase Y zeolite at here in the above conditions.

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تأثير التعمير الساكنة على تصنيع مدى واسع من زيوليت FAU بغياب البذور تحت عوامل مختلفة

سما محمد الجبوري

قسم الهندسة الكيمياء-كلية الهندسة-جامعة بغداد-بغداد-العراق

الخلاصة

يركز هذا العمل على تكوين زيوليت من نوع Faujasite النقي باستخدام مرحلة التنضيج الساكنة في غياب البذور ضمن الحدود المختارة. تم إنتاج بلورات بحجم النانو عند استخدام LUDOX AS-40 كمصدر للسيليكا لتكوين الهلام في المدى 2 وبلورته لمدة من 4 إلى 19 ساعة بدرجة حرارة 100 مئوية. علاوة على ذلك ، تم الحصول على بلورات كبيرة من الزيوليت Y عالي النقاوة في الظروف المذكورة أعلاه ولكن عند استخدام مينا سيليكات الصوديوم المائي كمصدر للسيليكا. أهم نتيجة تم التوصل لها من هذه الدراسة هي التنضيج الساكن يؤدي بكفاءة الى الحصول على طور زيوليت Y نقي في نفس الظروف المتحكم فيها لجميع الحدود الأخرى المختارة أيضاً لعملية التصنيع بغياب البذور.

الكلمات الدالة: نوع Faujasite, تعميم ساكن, تصنيع بغياب البذور, زيوليت, معاملة هيدروحرارية