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Synthesis and Characterization of Nanocrystalline Zeolite Y

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Abstract

Worldwide attention is being focused on nanocrystalline zeolites and they are replacing conventional ones due to their pronounced potential in many fields. In this study, NaY zeolite has been prepared hydrothermally using sol –gel method and modified to the proton type by ion –exchange process. Characterization is made using X-ray diffraction (XRD), thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR), Atomic force microscopy (AFM), Brunauer –Emmet- Teller (BET) nitrogen adsorption method, Ammonia Temperature programmed desorption (NH_3-TPD) and Scanning electron microscopy(SEM). The effect of aging time, silica to alumina ratio is studied and the results show well defined crystalline structure with nano particle size (70 and 81nm) with surface area of 499m^2/gm.

Keywords: *Zeolite Y, nanozeolite, sol-gel method, characterization.*

1. Introduction

Since their introducing in petroleum industry , zeolites provide the great role in many catalytic and petrochemical processing such as in the yield of high octane gasoline or in water filtration and purification in addition to their impact in producing cleaner fuels and lubricants [1]. Zeolites are aluminosilicates materials having a highly ordered crystal structure. The three dimensional network structured of SiO_4- and AlO_4- tetrahedral which provide the cavities of the zeolite [2]. Often zeolites are prepared hydrothermally from a period of time of many hours to few days. The reaction gel mixture provided from silicon source and aluminum source under alkaline state in different concentration and suitable aging and crystallization time to reach the final crystalline zeolite[3]. According to many function of zeolite such as solid acidity, shape selectivity and loading properties, nano-sized zeolites were prepared and became a challenge to obtain unique properties relative to microcrystalline zeolite having a high surface area and small particle size of less than 100nm [4,5]. Nano crystalline Y zeolite having more acid sites on its surface, large pore volume exhibites higher stability and higher activity in many application such as in fluid catalytic cracking reveal to their good diffusion of reactants and products and producing more gasoline and lighter gas oil [6,7].

Zeolite Y displays the Faujasite type structure having a 3-dimensional structure with pores being perpendicular to each other in the x, y, and z. It is constructed from sodalite cages with crystalline cubic structure having a characteristic pores of 7.4A^o. Also, it can be characterized by its silica to alumina ratio of (4 to 6) which differs than the other Faujasite type ; Zeolite X which have this ratio as 2 to 3 while for type A is 2.

The acidity of zeolite Y is motivated by its application as a solid catalysts, therefore the study of the acid site became inevitable Several methods had been successfully applied to study the active sites present on zeolites, and most of them are based on the adsorption or desorption of gas phase

probe molecules, which are chosen on the basis of their reactivity and molecular size.

NH_3-TPD is a widely used, simple and inexpensive technique for reflecting the surface acidity of a catalyst, where the temperature dependence in the desorption step is related to the acid sites present which may involve physical or chemical adsorption [8].

Preparation of nano zeolites is widely experienced with different methods and precursors. Therefore, microwave and ultrasonic methods, templates and surface modifiers are used mostly in Teflon- lined stainless steel autoclaves [9].

In this study, a facile method for the preparation of nonocrystalline zeolite Y is investigated using clear hydrothermal synthesis. Extensive characterization of the final product was done using (XRD), (TGA) (FTIR), (AFM), (SEM), (NH3-TPD),(BET).

2. Experimental

Sodium aluminate ($NaAlO_2$), sodium silicate solution (Na_2SiO_3) supplied by Sigma-Aldrich, sodium hydroxide (NaOH) by Merck were used. Zeolite Y was synthesized according to sol-gel method. Seeding gel is prepared by adding 19.95 gm of deionized water to 4.07 gm of sodium hydroxide and 2.09 gm sodium aluminate [10,11]. The mixture was stirred in a plastic bottle until dissolved then 28.3gm of sodium silicate solution was added and stirred moderately for at least 10 min. Then the seed gel was aged for different aging time of 24,48 and 72 hr at room temperature. The feed stock gel was prepared by mixing 131 gm of deionized water, 0.14 gm sodium hydroxide and 13gm sodium aluminate. Stirring was continued in 500 ml plastic beaker until the constituents were completely dissolved, then 178 gm of sodium silicate solution was added with stirring vigorously at 1600 rpm until smooth gel was obtained.

The final gel is prepared by adding 16.5 gm of aged seed gel to feed stock gel slowly with mixing up for 20 min. at 1600 rpm..After that the final gel was transferred to a poly propylene bottle and heated in an oven at 95^{o}C overnight. Precipitation occurred until a clear solution was observed indicting complete crystallization. The bottle was opened and left to cool then the wet solid product was set to filtration, washing with distilled water several times until the pH of the filtrate was 9. The product was dried at 110 ^{o}C for 24 hr. and then calcined at 500^{o}C at a rate of 2 ^{o}C /min. for 3 hr.

Ion Exchange Step

The acid form of zeolite Y was obtained by ion exchange with a solution of Ammonium chloride NH4Cl using two concentrations (2 and 4 N) at 70^{o}C. 10 g of zeolite NaY was slurried in 200 ml of ammonium chloride solutions with mixing at 70^{o}C for 2 hr and then left at room temperature overnight. After that the exchanged zeolite was filtered off ,washed with deionized water, dried at 110oC overnight, and then calcined at 525oC for 3 hr.[12].

The prepared zeolite were characterized by different techniques and compared with commercial type. These techniques include (XRD), (FTIR), (BET), (TGA), (SEM), (AFM) and (NH3-TPD).

The XRD for powder product were measured using Philips diffractometer with Cu target at 2 theta value from 10 to 60 ^{o}. The particle size and morphology were analyzed using SEM at different magnification by oxford instrument. The IR spectra was analyzed using Perkin FTIR in the wavelength range of 400 – 4000 cm-1. With KBr pellet method. BET surface area and pore volume were performed using a micrometrics ASAP 2020,the sample were degassed for 2 hr under vacuum at 250^{o}C .AFM was performed using scanning probe microscope (SPM), TGA test using SHIMADSU TGA-51 Thermo-gravimetric Analyzer and NH3-TPD was obtained using Micromeritics Auto Chem II Chemisorption Analyzer.

3. Results and Discussion

3.1. XRD

The powder diffraction patterns of zeolite samples were scanned using XRD diffract meter in the 2 theta range between 5 -60 degree at scanning speed of 5 deg / min using Cu-Kα radiation source of wave length λ=1.5406 A^o. The formation of zeolite Y phase was confirmed by comparing the diffract grams of all synthesized samples to the diffract grams of the reference zeolite Y[10].

Comparison indicates that the preparation method results is compatible with crystal structure of Y zeolite to conclude that the preparation

method gives a good synthesized indigenousness of Y zeolite [13].

The size of crystallites was correlated using Scherrer eq.1

$$d = 0.94\lambda / \beta \cos\Theta \quad \ldots(1)$$

Where d is the diameter of crystallite , λ is the X-ray wave length ,β is the broadening line at the half maximum intensity which represent the full width at half maximum (FWHM) and Θ is the Bragg angle at which the scattering wave was reflected or scattered at lattice plane producing intense peaks.

The average diameter of crystallite is 29.6 nm for prepared zeolite while for reference is 18nm. All peaks observed can be assigned to Y zeolite structure characterized by 2Θ of standard type equals to 6.3, 15.8 &23.7 degree as shown in Table.1 and 2.

Table 1,
XRD data of Synthesized zeolite NaY.

Synthesized zeolite	2Θ	I / I_o Intensity	FWHM degree
	6.344	100	0.327
	15.97	75	0.247
	23.61	84	0.9162

Table 2,
XRD data of reference zeolite NaY.

Reference zeolite	2Θ	I / I_o Intensity	FWHM degree
	6.181	100	0.501
	15.518	78.7	0.398
	23.478	83.1	0.589

3.1.1 Aging Time

The effect of aging time on zeolite formation was investigated and the XRD patterns of prepared samples were shown in Fig.2and the crystallinity was calculated using eq.2 by considering the ratio of the sum of intensities of 10 major peaks [14,15].

The results showed that 48 hr aging time was preferred in comparison to other aging time of 24, and 72 hr giving higher crystallinity of about 82% while for other samples was 75% and 52% as shown in Fig.1.

$$crystallinity\ \% = \frac{\sum I sample}{\sum I refrence} * 100 \quad \ldots(2)$$

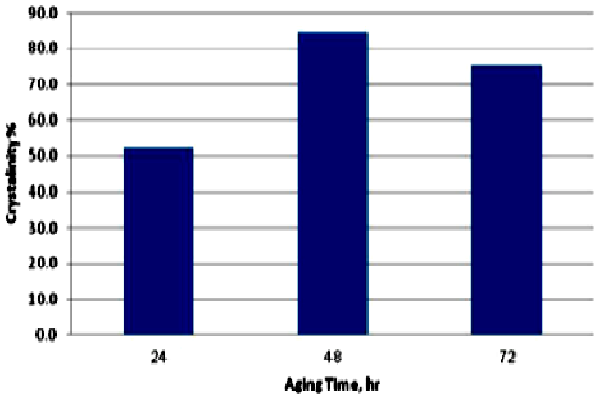


Fig. 1. Crystallinity % of the Prepared Zeolite.

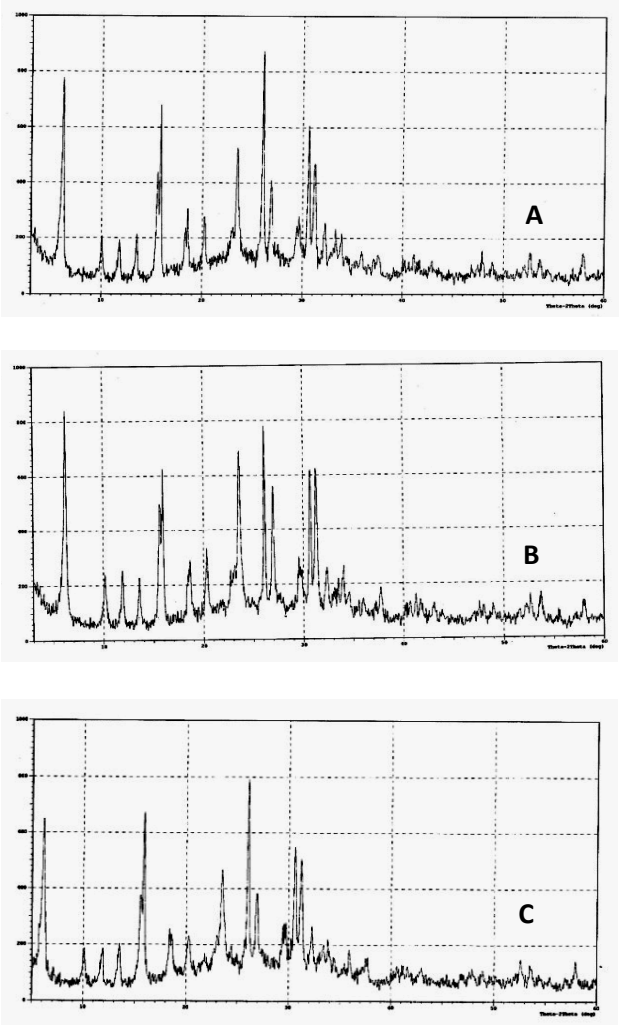


Fig. 2. XRD pattern of synthesis Na- Y zeolite at different aging time (A. 24 hr. , B. 48 hr., C. 72 hr.), X-axis 2theta,Y-axis Intensity .

3.2. Surface Area and Pore Volume

Nitrogen adsorption –desorption were carried out using Micromeritics ASAP 2020 instrument to determine the BET (Brunaure Emmett Teller) surface area and pore volume.

After degassing the isotherms is investigated at relative pressure P/Po from 0 to 1. surface area and pore volume are also measured. BET surface area is calculated according to this equation:

$$S_A = \frac{V_m}{22400} * a_m * N * 10^{-20} \quad \ldots(3)$$

Where SA : surface area m2 /gm, Vm : volume of monolayer m3, am : area occupied by one molecular of nitrogen in monolayer is 0.162 nm2, N: Avogadro's number 6.02*1023 molecules /mole.

It can be seen from the isotherms in Fig. 3 that the sample exhibited behavior of type two according to the classification of BET and this type shows large deviation from Langmuir model of adsorption and the intermediate flat region in the isotherm correspond to the formation of monolayer formation and best surface area and pore volume were obtained at aging time of 48 hr as shown in Table 3.

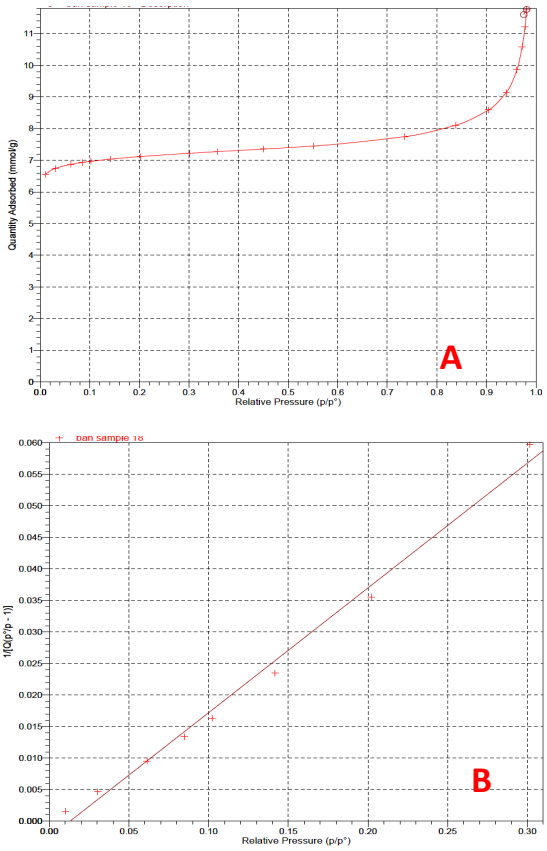


Fig. 3. (A) Adsorption Isotherm of prepared Y zeolite at aging time 48hr, (B) BET plot.

Table 3,
Surface area and Pore volume of prepared Zeolite.

Aging time hr	BET Surface area m^2/gm	Langmuir Surface area m^2/gm	Pore volume m^3/gm
24	201.9	286.63	0.213
48	499.43	707.95	0.38
72	210.6	298.79	0.209

3.3. XRF

XRF was used to determine the chemical composition and structural formula of prepared samples were determined ,and Si/Al ratio was also calculated from the results of this analysis as shown in Table 4.

Table 4,
Chemical composition and silica to alumina ratio of prepared zeolite.

Aging time Hr	24	48	72
SiO_2 wt.%	63.86	59.17	64.63
Al_2O_3 wt.%	22.32	20.10	23.83
Na_2O wt.%	8.966	9.30	7.208
SiO_2 /Al_2O_3 molar ratio	4.86	5.004	4.61

3.4. Ion Exchange

The exchanged technique was used in this process is one step batch impregnation under a constant temperature and different normality to convert NaY zeolite in to HY . Fig.4 shows the mechanism of ion exchange.

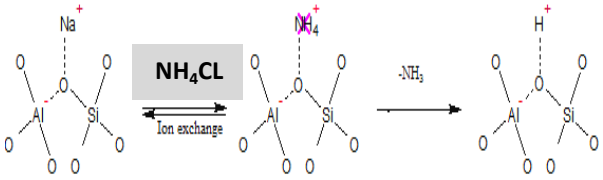


Fig. 4. Ion-exchange of Na-form of zeolite Y into H-form zeolite.

Zeolite is normally hydrophilic especially for small Si /Al ratio, the number of free cations is high so it is favored to increase the Si/ Al ratio by ion exchange.

The number of cations able to interacts with water decreases and the absorption of non-polar compounds molecular such as hydrocarbons leads

to use these zeolite as a catalyst in many petroleum processes such as cracking or reforming.

The percentage of the ion exchange, silica to alumina ratio and the concentration of sodium ion before and after protonation are listed in Table.5.

Table 5,
Chemical composition of modified zeolite.

Weight percent ,Wt.%	**Before**	**2 N**	**4N**
Na_2O	9.30	3.58	1.954
SiO_2	20.10	54.84	57.96
Al_2O_3	59.17	17.86	19.01
SiO_2 / Al_2O_3 Molar ratio	5.004	5.22	5.183
% Ion Exchange	-	61.50	78.98

3.5. FTIR

The chemistry of the surface of catalyst including surface acidity was measured using infrared technique. Faujasite structure is characterized by the functional groups and regions found in Fig. 5 The bands that appear in the region between 3365-3489 cm^{-1} reveal to the OH stretching band in the sodalite cage which belongs to the Bronstest acid protons, also called low frequency band ,SiO_4 molecules and Al-OH .The bands in the range of 1010- 1019 cm^{-1} indicate the presence of Si-O, assigned to external asymmetrical stretching while the band at about 600 cm^{-1} is assigned to the internal tetrahedral symmetrical stretching . The peak in about 1600 cm^{-1} corresponds to the bending vibration of water molecules in the zeolite structure. Absorption at about 443 -465 cm^{-1} was assigned to Si – O – Al stretching where Al is in the octahedral coordination. Therefore, it was concluded that the FTIR spectra of the synthesis zeolite matches the typical absorption peaks of commercial type[16,17,18] .

3.6. TGA

Thermal analysis is performed for the synthesized samples of zeolite NaY, HY and commercial type. Thermogravimetric plots are shown in Fig.6. It can be interpreted that as the temperature increases (30-200 ^oC), there was a little weight loss from the samples due to the water evaporation and dehydration. The same trend was found for the three samples. This give an indication of the validity of the calcination temperature ,also after the ion exchange step using NH_4Cl. The percentage of weight loss of the physically adsorbed water for synthesized NaY and HY zeolite are 22.92% and 18.72 respectively while for commercial zeolite is 23.9% which is in agreement of the results in references of published studies [19, 20].

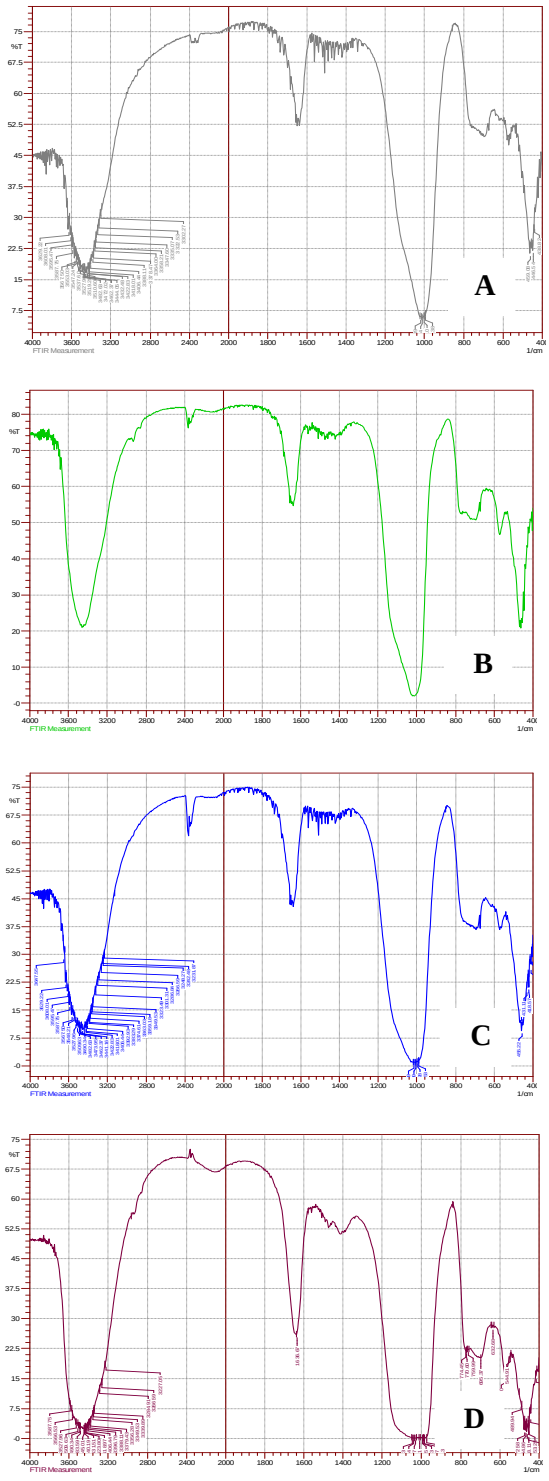


Fig. 5. FTIR of Synthesized zeolite Y at different aging time, (A)24hr, (B)48 hr, (C)72hr and (D) HY Zeolite .

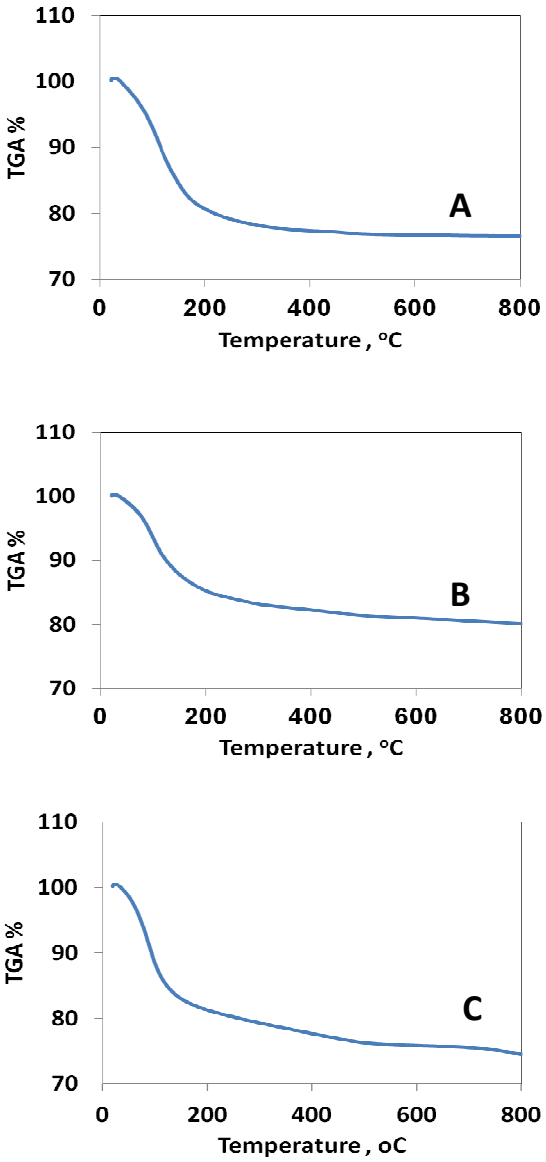


Fig. 6. TGA of Zeolite Y, (A) Synthesized NaY, (B) Synthesized HY,(C) Commercial HY.

3.7. SEM and AFM

Na-Y zeolite crystal size and morphology were observed by SEM. Results show the cubic crystalline micrographs of the synthesized zeolite having small average size of nano sized particles as compared with commercial type and the images show a good agreement with that shown in literature which reveals a uniform particle size with regular shape[11,13,14].

To investigate the surface uniformity AFM technique is used. By comparing the surface of synthesized and commercial zeolite there is no change in surface roughness as shown in Fig.9. It depicts a uniform nano and micro structure and highly crystalline material with uniform pores. The average particles size of the prepared zeolite is 80 nm and for exchanged is 71nm while for commercial type is about 91nm. [21],[22].

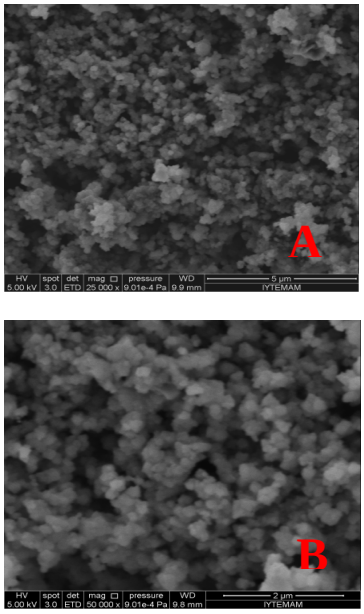


Fig. 7. SEM images of Synthesized zeolite Y, (A)25 000 X, (B) 50 000 X.

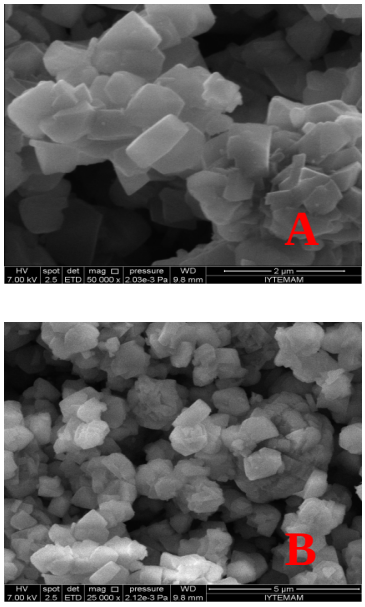


Fig. 8. SEM images of commercial zeolite Y, (A)25 000 X, (B) 50 000 X.

3.8. TPD

Temperature programmed desorption of Ammonia (NH3-TPD) is used to determine the acidity of prepared zeolite samples. All samples are heated up according to steps shown in Figs. 10-13 and the thermal conductivity detector (TCD) is recorded. Data obtained is utilized and

plotted by calculating the area under the peaks shown in these graphs.

Zeolite acid sites are characterized using NH3-TPD ,total amount of acidity and acid distribution are obtained from peaks area ,position and shape. Although NH3-TPD giving an information about the strength of acid site but it cannot distinguish between acids site types as Bronsted or Lewis. Figs.10-13 show NH3-TPD spectra of prepared ,modified and commercial zeolite and all profiles of the sample represent the low peak while for modified samples high peak are also appear and are noticeable than Y zeolite this because of protonation effect .

The results obtained are in agreement with that in literature as the low peak appears at a temperature of about 200 ^{o}C and intermediate appears between 350 – 350 ^{o}C while the high acidity peak represented at temperature between 500 -600 ^{o}C, sometimes it doesn't appears purely because its overlapped or engaged with other peaks. Acidity peak represented at temperature between 500 -600 ^{o}C, sometimes it doesn't appears purely because its overlapped or engaged with other peaks.

The high temperature peak can be attributed to the desorption of ammonia from both strong Bronsted and Lewis acid site and on the other hand the low temperature peak can be related to a number of causes such as the adsorption of ammonia on weakly acid site as Bronsted and Lewis, silanol groups and formation of NH+ 4groups.[23, 24, 25].

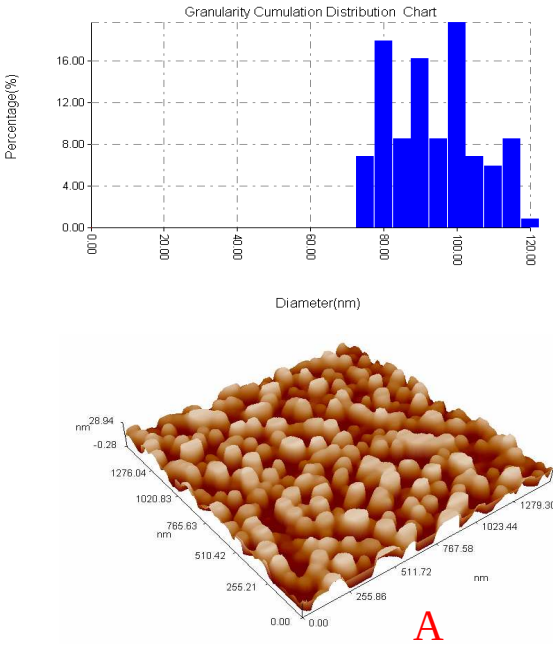
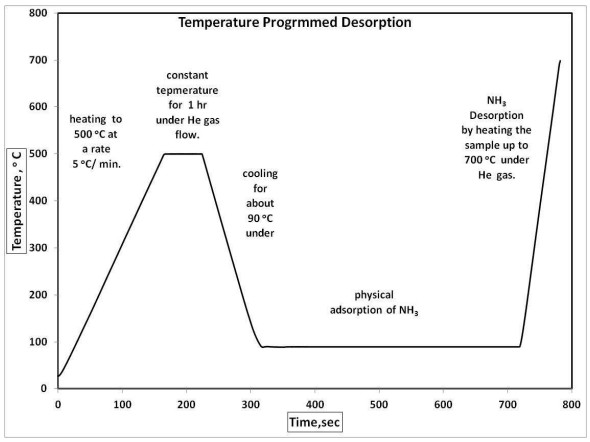


Fig. 9. Particle size distribution and 3-D Dimensional Images. (A) Synthesized zeolite Y (B) Ion exchanged zeolite HY (C) Commercial zeolite.



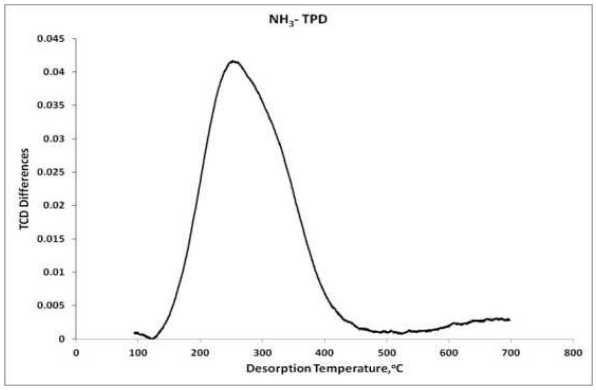
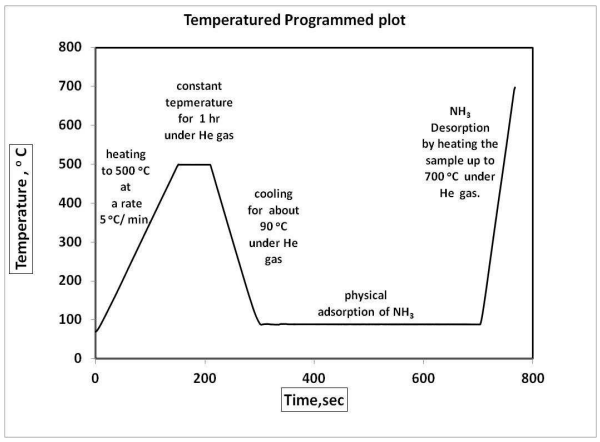


Fig. 10. NH_3-TPD of prepared NaY zeolite.



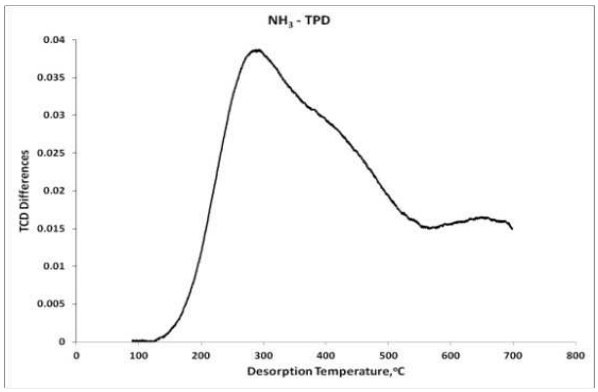
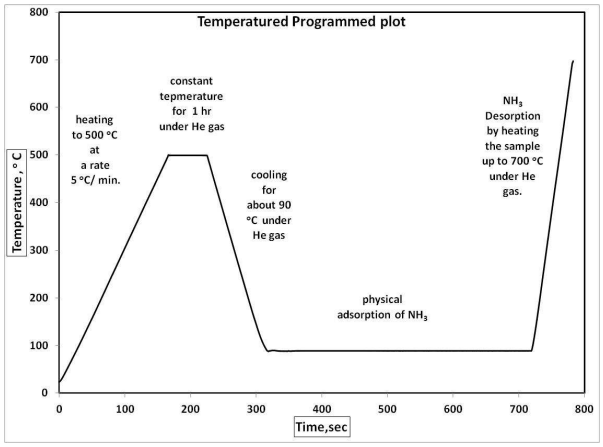


Fig. 12. NH3-TPD of modified HY zeolite, 4N.



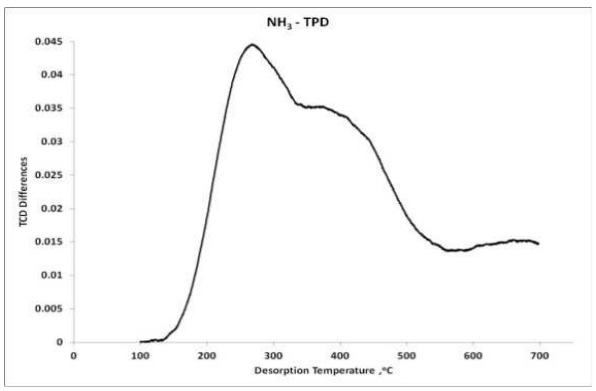
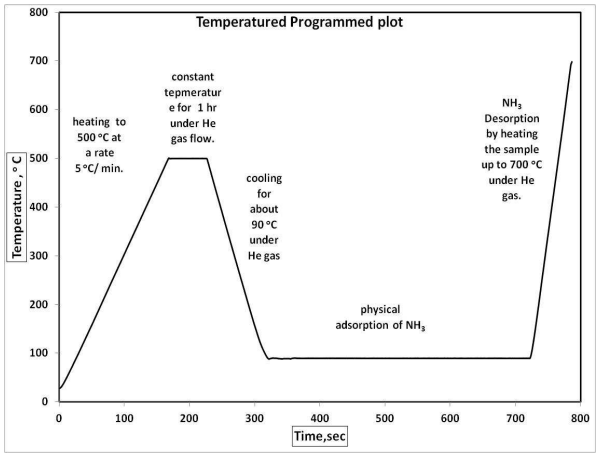


Fig. 11. NH_3-TPD of modified HY zeolite, 2N.



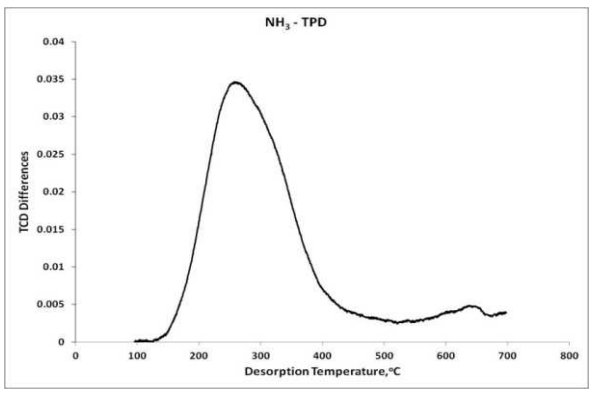


Fig. 13. NH_3-TPD of commercial HY zeolite.

4. Conclusion

Nano crystalline Na-Y zeolite has been prepared successfully using sol-gel method under hydrothermal condition. The decrease in crystal size affects the physical properties of the product such as surface area and surface uniformity as compared with microcrystalline type. Nano crystalline zeolite NaY and HY are prepared and characterized using different methods. Results showed well defined morphology, uniform distribution, high surface area, and stable structure.

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5. References

[1] Mohammed R., Rahimpour M. and Davood I., "Progress in Catalytic Naphtha Reforming Process: A review" Applied Energy, 109, (79 – 93), 2013.

[2] Myada M. Hassan," Improvement of Catalysts for Hydroisomerization of Iraqi Light Naphtha" MSc. Thesis, University of Technology, 2010.

[3] Mahdi F. and Niloofar O.," Controlling Yield of NaY Zeolite Synthesis by Hydrothermal Method" International Journal of Industrial Chemistry , 2, (190 – 195), 2011.

[4] Holmberg B., Wang J. and Norberk J. ," Controlling Size and Yield of Zeolite Y Nanocrystals using Tetramethyl Ammonium Bromide" Microporous and Mesoporous Materials, 59, (13-28), 2003.

[5] Toshev , L. and Valitechev P. , " Nano Zeolite Synthesis Crystallization Mechanism and Application " Chemistry of Materials , 17, (2494 – 2513), 2005.

[6] Song W., Grassian V. and Larsen S. ," Development of Improved Materials for Environmental Applications: Nanocrystalline NaY Zeolite" Enviromental Science and Technology , 39 , (1214,1220), 2005.

[7] Jacobsen C. , Madsen T. and Janssens H. " Zeolites by Confined Space Synthesis – Characterization of the Acid Sites in Nanosized ZSM-5 by Ammonia Desorption and Al/Si NMR Spectroscopy" Microporous and Mesoporous Materials, 39, (393 -401), 2000.

[8] Chester A.W. and Derouane E. G., Zeolite Characterization and Catalysis. A Tutorial, Springer, 2009.

[9] Sadeghi M, Sharifi S.L., Hatami H., Synthesis of nanocrystalline zeolite NaY by hydrothermal method and investigation of its structure and morphology, Int. J.Nano Dimens. 5(1): 91-95, 2014 .

[10] Rahman M., Awang M. and Yuof A. ," Preparation ,Characterization and application of Zeolite Y for water Filteration" Australian Journal of Basic Applied Science , 6 , (50 – 54), 2012.

[11] Nik A. and Alias M. ," Removal of Cr(III) fom Aqueous Solutions Using Zeolite NaY Prepared From Rice Husk Ash" The Malaysian Journal of Analytical Science , (11), (76 – 83), 2007.

[12] Abdual Halim A., Marwan G. and Mohammed N.," Effect of Promoters on Catalytic Activity of the Isomeriztion Catalyst" Iraqi Journal of Chemical and Petroleum Engineering, 9, (9-14), 2008.

[13] Mohammed N. , Ahmed A., Ali F.," Catalytic Cracking of Heptane using prepared Zeolite" Journal of Asian Scientific Research, 2,(927- 948),2012.

[14] Htay M. and Mya O., " Preparation of Zeolite Y Catalyst for Petroleum Cracking " World Academy of Science, Engineering and Technology , 48, (114-120), 2008.

[15] Vaezifar S., Faghihian H. and Kamali M., " Dehydrogenation of Isobutane Over Nanoparticles of Pt/Sn Alloy on

Pt/Sn/Na-Y Catalyst : the Effect of Tin Precursor on the Catalyst Behavior" Iran J. Chem. Eng., 28 , (23-30), 2009.

[16] Kavan M., " Template –Free Synthesis and Modification of LTY,ZSM-5 and LTL Zeolite Catalysts and Investigation of Catalytic Pyrolysiss of Saskatchewan Boundary Dam Coal" MSc. Thesis ,university Gal Gary,2013.

[17] Niken T., Abdual Rahman M. and Subhash B. ," Nanocrystalline Zeolite Y : Synthesis and Characterization " IOP Conf. : Material Science and Engineering., 17,2011.

[18] Yaneri P. and Petit D.," Linde Type a Zeolite and Type Y Faujasite as a Solid-Phase for Lead, Cadmium, Nickel and Cobalt Preconcentration and Determination Using a Flow Injection System Coupled to Flame Atomic Absorption Spectrometry " American Journal of Analytical Chemistry, 4, (8-14), 2013.

[19] Hilal G. ," Liquid Phase Hydrogenation of Citral on Zeolite Supported Monometallic (Ni,Pt) and Bimetallic (Ni, Sn), (Pt-Sn) Catalysts" MSc Thesis , Izmir Institute of Technology, 2005.

[20] Emre K., " An Investigation of Activities and Selectivites of HZSM-5 and H-Ferrite Zeolite Modified by Different Method in N-Butene Isomarization" MSc. Thesis, Izmir Institute of Technology, 2010.

[21] Atheel H., Ahmed Z. and Norli S. ," Investigation on the Catalytic Behavior of Fe Loaded on Encapsulated Titanium for Sonocatalytic Degradation of Amaranth :Characterization and Reusability Study" Modern Research in Catalysis, 2, (100- 109), 2013.

[22] Nabiollah M., Navid R. and Homayon A.," Porosity ,Characterization and Structural Properties of Natural Zeolite–Clinoptilolite- As a Sorpent", Environment Protection Engineering , 39, (139-151), 2013.

[23] Conghua L., Xionghou G., and Zhongdon Z., " Surface Modification of Zeolite Y and Mechanism for Reducing Naphtha Olefin Formation in Ctalytic Cracking Reaction" Applied Catalysis , 264, (225-228), 2004.

[24] Atchariya A. , Kongkiate S., and Sirachay K." Effect of Catalyst Acidity on Thermal Cracking of Ethylene at High Temperature and pressure" International Conference on Chemical , Civil and Environmental Engineering ,Nov. 2014, Singapore.

[25] Tonetto G., Atias J. and de Lasa H. ," FCC Catalysts with Different Zeolite Crystalline Sizes: Acidity ,Structural Properties and Reactivity" Applied Catalysis , 270 ,(9-25), 2004.

تحضير وتشخيص زيولايت Na–Y ذو التركيب البلوري النانوي

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الخلاصة

تتجه انظار العالم الى الزيولايت ذي التركيب البلوري والنانوي لتحل محل الانواع التقليدية لما لها من تطبيقات ملحوظة في شتى المجالات. في هذه الدراسة تم تحضير زيولايت نوع Na-Y بمعاملة الجل البلوري حراريا وتم اجراء عملية التبادل الايوني للحصول على HY زيولايت. تم اجراء الفحوصات التشخيصية مثل الاشعة السينية والتحليل الوزني الحراري والمطياف ذو الاشعة فوق الحمراء وتشخيص الابعاد بمجهر القوة الذرية والمجهر الالكتروني والمساحة السطحية بالامتزاز بالنتروجين وتحليل درجة الحرارة لعملية عكس الامتزاز للامونيا. تم دراسة تاثير زمن التفاعل ونسبة السليكا الى الالومينا. اثبت النتائج فعالية عملية التحضير ونجاحها في الحصول على تركيب مسامي بلوري بقياس ٧٠ و ٨١ نانومتر ومساحة سطحية ٤٩٩متر ^{٢}/ غم.