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Synthesis and Characterization of Nano-crystalline ZSM-5 Zeolite

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Abstract

Nano-crystalline ZSM-5 zeolite was synthesized by hydrothermal method using chelating agent and two gel compositions:CompositionI:Al_2O_3:86SiO_2:5.5TPA:12.7Na_2O:3.4Trien:3320H_2O.CompositionII:Al_2O_3:68SiO_2:5.4TPA: 10Na_2O:2.6Trien:2626H_2O.Study of hydrothermal reaction factors on characteristics of nano- sized zsm-5 has been carried on ,among them are crystallization temperature, crystallization time and concentration of template (TPAOH) solution. Synthesis was accomplished in PTFE lined autoclave (reactor) . The product were characterized by X-ray diffraction (XRD),Atomic force microscopy (AFM),scanning electron microscopy, Fourier transform infrared (FTIR) and Brunauer-Emmet-Teller (N_2 absorption-desorption isotherm).SEM and XRD results indicate that crystallinity of synthesized zeolite increase with increasing crystallization time and temperature. AFM results indicate that a nano-sized level of about 50nm was reached for "particle average diameter". FTIR results showed that the required peaks near "1080,800,550,450 cm^{-1}" have been clearly obtained, Furthermore the results showed strong effect of template (TPAOH) solution concentration on crystallinity and decreasing of particle size towards nano-size level.

***Keywords**: Crystallization , Template, Synthesis, Nanocrystalline, ZSM-5 zeolite.*

1. Introduction

ZSM-5 is a member of MFI-zeolite family with high Si/Al ratio from 10-100+.It is a zeolite with three dimensional channels, and it has two kinds of channels, straight channels with pore opening of 0.54nm x 0.56nm and sinusoidal channels with a pore opening of 0.51nm x 0.55nm[1]. ZSM-5 has been given a great attention by researchers because of its splendid specifications concerning thermal stability, high surface area, strong acidity, and presence of Nano-spaces ,The sizes of intra-crystalline pores and nano- spaces are nearly the same as molecular diameter of light hydrocarbons [2]. Strong acid sites exist on the nanoporous surfaces enable the zeolite to be used as shape selective "catalyst" especially in petroleum and petrochemical industries, such as isomerization, fluid catalytic cracking and also in adsorption processes[2] . The crystal size of zeolite is much larger than the micropore size ,and this will result in a low diffusion rate of reacting hydrocarbon molecules leading to low mass transfer, and consequently lower reaction rate, low selectivity of intermediates and coke formation followed by a short catalyst life time. A methodology of two proposed solutions have been implemented, the first one was the formation of meso-pores in the crystal structure of the zeolite, the second solution was to prepare nano-sized crystalline zeolite, so that the diffusion path for reacting molecules will be decreased as crystal size decrease these two factors are effective in improving catalytic activity[2].

Different methods and procedures have been utilized to synthesize ZSM-5 which involve a hydrothermal crystallization of reactive alumina silicate gels[1,3,4,5,6,10,11,17]. the product properties are highly affected by many factors such as alumina and silica sources[4,11],Si/Al ratio [4,5,11,14],template [1], crystallization time [1,7] and crystallization temperature [1,4,7,8,9].

R. karimi et al[1].used hydrothermal synthesis by stainless steel autoclave lined with PTFE , they investigated the effect of crystallization temperature and time also studies the effect of the type of organic structure directing agent (OSDA) on the synthesized zeolite , a Nano level sized of ZSM-5 has been produced with high crystallinity at temperature range of 170-180 and 120h for crystallization time, .They found that the template (OSDA) content has a great effect on ZSM-5 properties , a ratio of 0.058 for TPAOH/Si was optimum.

M.Abrishamkar et al [3] followed a clear solution method under atmospheric pressure using TPAOH as template, aluminum isopropoxide as alumina source , TEOS as silica source with NaOH and H_2O as mineralizer and solvent respectively , they reported a new path for producing nano-crystalline ZSM-5 Zeolite.

R. Mohamed et al [4] studies the effect of the silica source on the crystallinity of Nano sized ZSM-5 Zeolite ,they concluded that crystallinity of ZSM-5 produced is increased in the following order colloidal silica < TEOS < fumed silica.

R. Mohammed et al[7] studies the influence of the crystallization time on the synthesis of Nano sized ZSM-5 , they found that a time of crystallization of 45h gave the highest crystallization with 55.6 nm for average particle size and the BET surface of 360 m^2/g , using 20wt% TPAOH as template (OSDA).

In this research work tetrapropyl ammonium hydroxide (TPAOH) was used as template .A detailed parameter study to get the desired properties in the final zeolite product has been accomplished including crystallization time ,temperature ,gel composition ,template solution concentration and chelating agent.

2. Experimental

2.1. Technical Procedure

Tetraethylorthosilicate (TEOS-Sigma Aldrich) was used as silica source, aluminum is opropoxide (AIP-Sigma Aldrich) as alumina source, tetrapropyl ammonium hydroxide (TPAOH) as template, sodium hydroxide as alkaline agent, triethylenetetramine as chelating agent, and deionized water as solvent. Nano-sized ZSM-5 zeolite was synthesized using twin stainless steel autoclaves lined with polytetraflouroethylene (PTFE), and by hydrothermal reaction of silica source with alumina source in presence of TPAOH as template and Trien as chelating agent .Two compositions was studied and chosen, the first one with silica to alumina ratio Si/Al is 86, and the second is with Si/Al=68.The preparation procedure was kept the same, the differences are in the amount of alumina source(AIP) and Template (TPAOH).

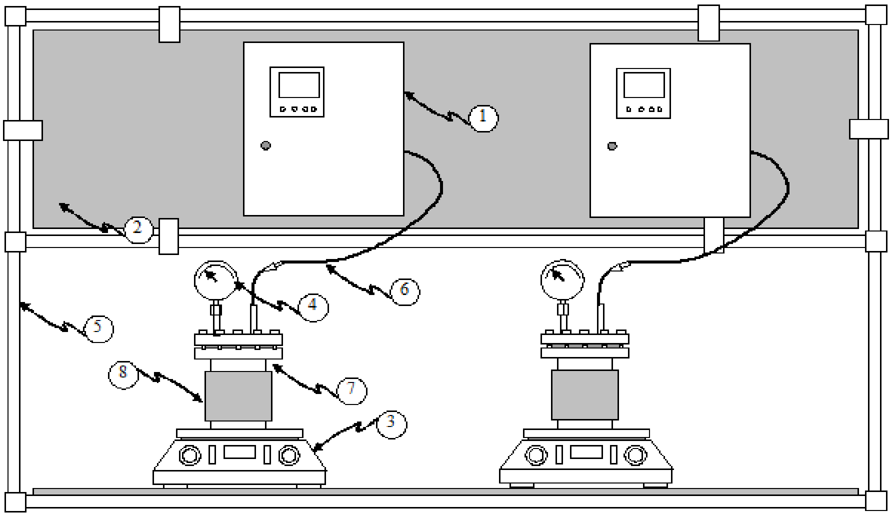
Composition I :
Al_2O_3:86SiO_2:5.5TPA:12.7Na_2O:3.4Trien:3320H_2O.
Composition II:
Al_2O_3:68SiO_2:5.4TPA:10Na_2O:2.6Trien:2600H_2O

2.2. Preparation Composition I

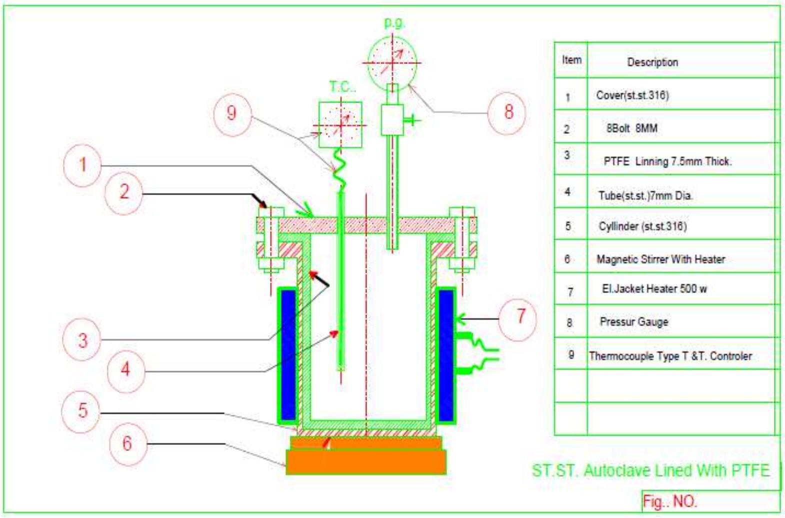
The template TPAOH (2.2g) and TEOS (36g) were mixed with (40ml) deionised water for 1h of stirring. Trien (1ml) was added , NaOH (2.05g) was dissolved in 40ml deionised water and added also to make solution 1. Aluminum is opropoxide (AIP) 0.82g was added to 40ml deionised water and stirred for 1h to make solution 2. The resulting mixture(solution 2) was added dropwise to (solution 1) with vigorous stirring (500rpm) in mechanical stirrer for 120min. The resulting gel was poured into a stainless steel autoclave lined with PTFE(polytetraflouroethylene), the volume of PTFE insert is 200 ml and were filled to about 65% of its inside volume .A stir bar was placed inside the PTFE insert for stirring purpose. The sealed autoclave was equipped with electric heating jacket providing 500w and thermocouple t-type, also pressure indicator for measuring the autogeneous pressure. An electronic board with (PID) to control the temperature inside the autoclave during the hydrothermal reaction ,twin autoclaves were used as shown in Fig.1.

2.3. Preparation of Composition II

The same procedure except that the amount of "template" TPAOH used was (2.9g) and the amount of alumina source (AIP) was (1.025g) .For both compositions and after collapsing the desired time at specific temperature ,the autoclave was left to cool overnight and the solid product was filtered, washed with distilled water many times and finally with ethanol, then the product left to dry at 110 ℃ overnight. Then the material was calcined at 550 ℃ for 5h with temperature ramp 1 ℃/min. The hydrogel compositions and crystallization conditions (time& temperature) of hydrothermal synthesis for various samples are tabulated in Table2.



No.	Item	No.	Item
1	Control Board	5	Stainless Steel Structure
2	Support Board	6	Thermocouple Type-t
3	Magnetic Stirrer with Heater	7	High Pressure Reactor
4	Pressure Gage	8	Electric Heating Jacket



Item	Description
1	Cover(st.st.316)
2	8Bolt 8MM
3	PTFE Linning 7.5mm Thick.
4	Tube(st.st.)7mm Dia.
5	Cyllinder (st.st.316)
6	Magnetic Stirrer With Heater
7	El.Jacket Heater 500 w
8	Pressur Gauge
9	Thermocouple Type T &T. Controler

Fig. 1. Twin autoclaves lined with PTFE.

Table .1
Sample Conditions.

Sample	crystallization time ,h	crystallization temp.^{0}C	Composition	Wt% TPAOH	Si/AL
S1	24	140	I	10	86
S2	72	150	I	10	86
S3	72	160	I	10	86
S4	72	160	I	20	86
S5	72	160	I	40	86
S6	96	160	I	40	86
S7	96	170	I	40	86
S8	80	175	I	40	86
S9	48	150	II	40	68
S10	72	150	II	40	68
S11	96	150	II	40	68
S12	48	160	II	40	68
S13	72	160	II	40	68
S14	96	160	II	40	68
S15	48	170	II	40	68
S16	72	170	II	40	68
S17	96	170	II	40	68
S18	48	180	II	40	68
S19	72	180	II	40	68
S20	96	180	II	40	68

Table.2,
Parameters of samples.

Sample	Ave.particle.diam.,nm	s.surface m²/g	Pore volume cm³/g	Crystallinity%
S1	84.7	155.9	0.111	78
S2	76.3	229.8	0.1477	98.4
S3	80.2	259.7	0.15181	110
S4	73.6	347	0.2007	88.4
S5	79.2	355.1	0.20721	104.7
S6	74	345.88	0.2022	90.3
S7	79.4	380.9	0.225	82.8
S8	74.4	334.13	0.192	95.5
S9	84.43	334.5	0.2016	77
S10	71.98	336.4	0.1878	99.5
S11	90.8	335.86	0.18405	88.8
S12	85.08	360.4	0.2117	84.4
S13	91.2	351.9	0.1959	92.9
S14	65.9	351.76	0.206	96
S15	50.8	365.1	0.2239	114.4
S16	81.68	336.1	0.1997	100
S17	60.5	322.39	0.18852	98.8
S18	86.8	332.8	0.1967	102
S19	63.4	303.96	0.1920	107.4
S20	51.9	327.53	0.18997	95.2

3. Characterization

The XRD patterns of zsm-5 zeolite were determined using a D2 PHASER/ Bruker (Germany 2010) using CuKα radiation Nickel filter (λ=1.54A).Data were collected within the 2Ɵ range of 2° and 50° with a 0.02° 2Ɵ-step and 0.5s per step (30kv and 10mA)X-ray diffraction was implemented to check the required pattern of ZSM-5 and its crystallinity. Fourier Transform Infrared (FTIR) by Shimazdu-IRA AFFINITY-1,using KBr wafer 1% wt zeolite and 99% KBr to check and record the peaks at specified wave lengths corresponding to ZSM-5 In the range of 400-4000 cm-1 wave number. The morphology of zsm-5 zeolite was studied by scanning electron microscopy using Te Scan,Vega ɪɪɪ LM,CZECH. The particle size and morphology on nano-level size were ,determined by atomic force microscopy using an AA3000/Angstrom Advance Inc, USA ,scanning probe microscope(SPM) gave granularity cumulation distribution and surface roughness. Table 2 shows parameters of samples, Average particle diameter measured by , in which the increase in crystallinity is only from 99.5% to 107.4%, in averaging the value of (AFM),specific.surface(BET), pore volume, and crystallinity (XRD) for both compositions.BET surface and pore volume for all samples were measured by surface area analyzer/Q surf series/Italy ,two samples S7 and S20 was repeated by micrometrics ASAP 2020/USA. BET surface is based on BET theory (Brunauer . Emmet and Teller) which described five basic adsorption isotherms . but IUPAC classification added one more adsorption isotherms .BET theory is an extension of the Langmuir theory (monolayer adsorption model) to the multi adsorption model. Adsorption can be defined as the adhesion of atoms or molecules of gas to a surface , the amount of gas adsorption depends on surface area. gas pressure, temperature and strength of interaction between the gas and solids . Nitrogen is usually used in BET surface analysis because of its purity , strong interaction with most solids and availability BET equation for determining surface area in its simplest form.

St=k(1-p/po)*Va

Where St=total surface area of sample being analyzed K=A constant for nitrogen , assuming STP condition=4.03

p/po=0.294 for gas mixture of 30% N2/70% He

Va=volume of gas (N2) adsorbed

Each cubic centimeter of N2 adsorbed (then desorbed) by the sample is equivalent to total surface area (St) of 2.84 m2 , the specific surface is found by dividing St by the weight of the sample in the cell in grams The complex form of BET equation.

$$\frac{p}{V(po-p)} = \frac{1}{V_m C} + \frac{C-1}{V_m C}\left(\frac{p}{po}\right) \quad [11,19]$$

P=pressure , Kpa

Po=saturation pressure , KPa

V=total volume of gas adsorbed , cm3

Vm=volume of gas absorbed in monolayer , cm3

C=constant and related to the heart of adsorption

$$pore\ volume = \frac{V}{22414} x \frac{Mg}{\rho_g} \quad [11]$$

V=volume of adsorbed gas

Mg=molecular Wight

ρ_g= gas density

For BET surface area analysis a mixture of nitrogen balance helium is passed through the reference channel of the thermal conductivity detector(TCD)to the sample housed within a flow-through glass cell and finally into the TCD analytical channel the signal produced by the TCD detector is collected by the microprocessor control board. The procedure starts with the degassing of the sample with the carrier gas at the programmable temperature when the sample is dried , the operator simply transfer the sample holders to the analytical ports and fit them into place. Pressing the start button will automatically raise the liquid nitrogen flask carriage initiating the analysis.

Fig. 2. BET surface area analyzer.

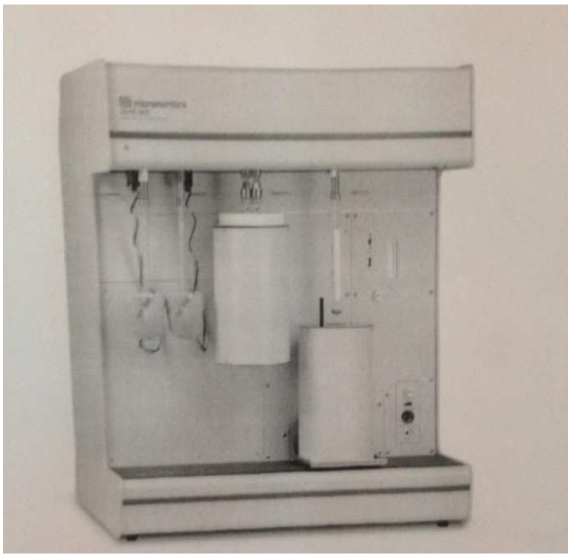


Fig. 3. ASPA 2020 Accelerated surface area and porosimetry system ASTMD3663, ASTMD4222, ASTMD4365, ASTMD4641.

4. Results and Discussion

4.1. Crystallization Temperature

The most important factor in the synthesis of zeolite is crystallization temperature, so a set of samples were prepared for both compositions I & II ranging from 140 ℃-180 ℃ as shown in Table 2, the results show a strong dependency of crystallinity on temperature and these results coincide with previous work[1,4,11]. Comparing sample S9(150 ℃,48h) with S18(180℃,48h) it can be noticed that the crystallinity increased from 77% to 102% , this increase of crystallinity would be less as time of crystallization increase to 72 when comparing sample S10(150℃-72h) and S19(180℃,72h). The average crystallinity with temperature (see Table 3), showed an increase in the average value of crystallinity from 88.4% at 150℃ to 91% at 160℃ and 104.4% at 170℃, keeping almost the same level at 180℃ , indicating that the crystallinity, reached its optimum value around 170-180C , Figs.3 and 4 are XRD patterns and SEM micrographs of ZSM5 products the effect of temperature on average particle diameter and the nano-level size was observed and a tendency to have a decrease in average particle diameter as temperature is increased, and particle diameter decreased from about 82nm at 150℃ to 67nm at 180℃ (see Table 3), these results of nano-level size are associated with a high crystallinity degree, support the hypothesis and results revealed recently by Lupulescu and Rimer[19], which suggested a non-classical pathway of zeolite crystallization by the presence and gradual consumption of nano-particles, while most crystals grow through classical means (the addition of atoms or molecules to the crystal). SEM images show that samples which were prepared at temperature 150 ℃ has amorphous phase which coincide with average results of XRD patterns and crystallinity (88%), and crystallized in spherical form,but for samples prepared at 170 and 180 ℃ crystallized mostly in orthorhombic shape crystals.

4.2. Crystallization Time

To study the effect of this important factor, a series of samples was prepared using gel composition I, and composition II . XRD patterns (Figs.3) at different temperatures (150,160,170,180 ℃) , show that zsm-5 phase is the only one obtained in all experiments and the peak at 2Θ=7-9 and 2Θ=22.5-24.5 are typical for ZSM-5[13]. The crystallinity of samples show a tendency to increase due to increase in crystal nucleation and growth rate , as the time increase in temperatures range of 150 & 160 C but for temperature 170 and 180 C the crystallinity increased up to 72h.

Comparing SEM images of S19 (72h,180 C) with S20 (96h,180 C) show that the increased crystallization time cause to have a lower crystallinity and beginning of losing the orthorhombic shape crystals to spherical. but a little decline observed in crystallinity when time increased above 72h, the crystallinity begins to decrease slightly , this behavior can be explained by the cause of a reverse process of recrystallization occurs leads to this declination , and it seems that the template(TPAOH) begins to lose its performance at higher temperature when the time of crystallization increased above 72h, the reports of AFM showed that the nano-sized level of crystalline ZSM-5 are influenced by temperature and time and as shown in Tables 2 and 3 and Fig.2 . the spectra of (FTIR) for synthesized samples at various crystallization times and temperature are showed in Fig.5 the findings show that the bands near1085CM-1(internalasymmetricstretch), 799CM-1(external symmetric stretch),546 CM-1(double ring vibration) and 452 CM-1(T-O band) and the formation of ZSM-5 phase only.The peak near 550CM-1 is attributed to the five- membered ring of the ZSM-5 zeolite structure (pentasil)[17]. The evidence for getting nano-sized level of ZSM-5 was the peak near 1225CM-1 and it is an asymmetric stretch vibration of the "T-O" bond which is attributed to external linkages between tetrahedral TO4 [18]. In spite of the decrease in Si/AL ratio in gel compositions II comparing with gel compositions I , the effect of increasing the amount of TPAOH ie TPA/Si ratio gave better results concerning nano-sized level.

4.3. Template Solution Concentration

The concentration by weight of template (organic structure directing agent-OSDA,TPAOH)solution was studied by using three kind of solutions 10%wt , 20%wt and 40%wt , using gel composition I, the samples S3 , S4 , S5 represent 10%wt , 20%wt and 40%wt(TPAOH) respectively.

These samples were prepared at the same temperature 160̊C and same crystallization time 72h .the results show that the best result in surface area and pore volume for S5 (355 m2/g and 0.20721 cm3/g respectively) in which 40%wt(TPAOH) was used with very good crystallinity.

4.4. Gel Compositions

Two gel compositions were studied I and II which they are different in Si/AL ratio and more template (TPAOH) introduced in the gel compositions II so TPA/Si ratio is more in gel compositions II . the results reveals (as shown in the Table 2) that a decrease in particle diameter down to 50 nm (S15 and S20) are given by compositions II.

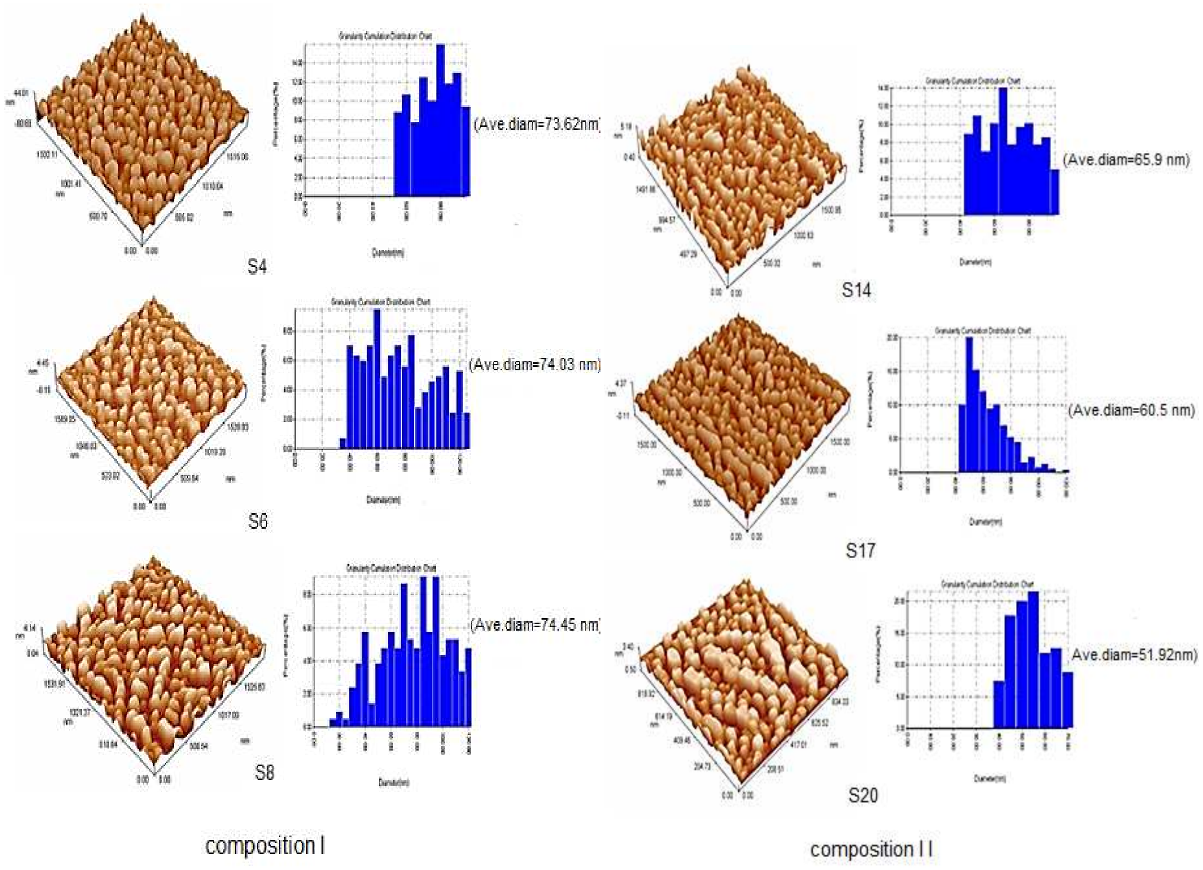
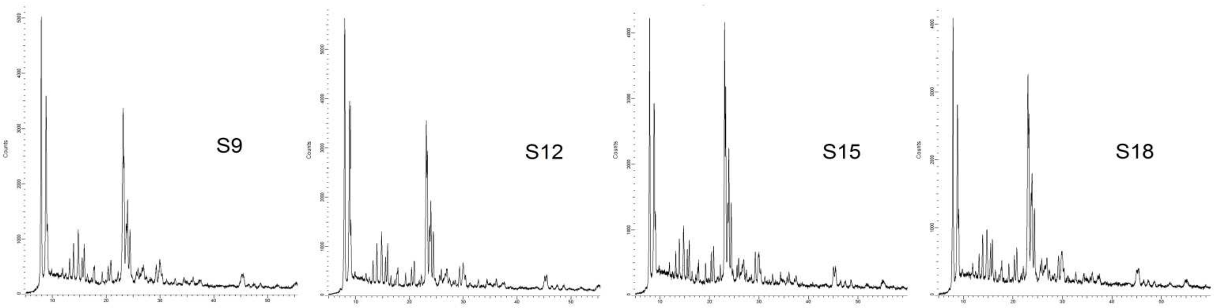


Fig. 4. Atomic force microscopy reports for samples of composition I &II.

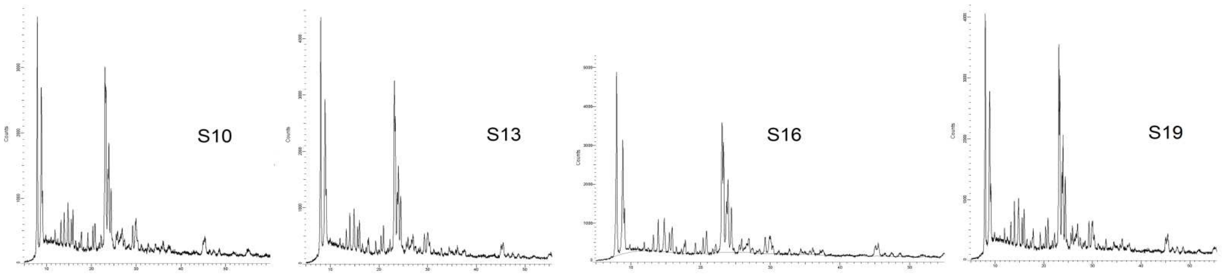
Table 3,
Average parameter value at different crystallization time and temperatures.

Sample code	**Average crystallinity%**	**Average particle diameter ,nm**	**Average BET surface area m^2/g**	**Average pore volume Cm^3/g**
S9,S10,S11 (48,72,96h) And 150 ^{0}C	88.4	82.4	338.5	0.19115
S12,S13,S14 (48,72,96h) And 160 ^{0}C	91.1	80.7	354.7	0.2045
S15,S16,S17 (48,72,96h) And 170 ^{0}C	104.4	69.3	341.2	0.20404
S18,S19,S20 (48,72,96h) And 180 ^{0}C	101.4	67.3	321.4	0.1929

For 48h



For 72h



For 96h

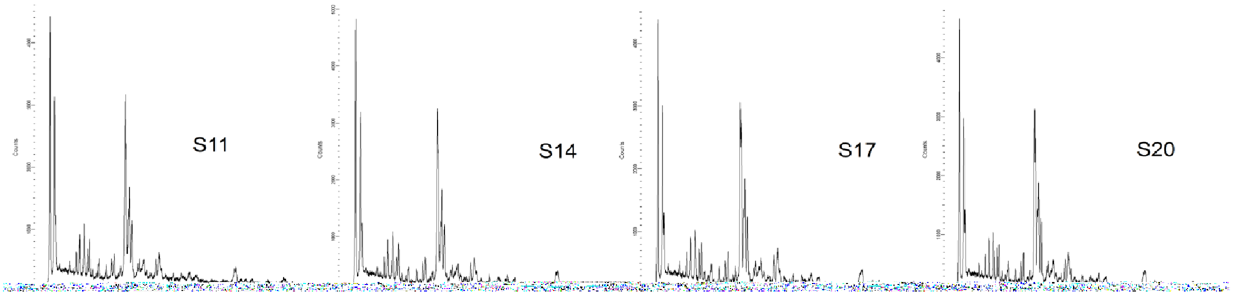
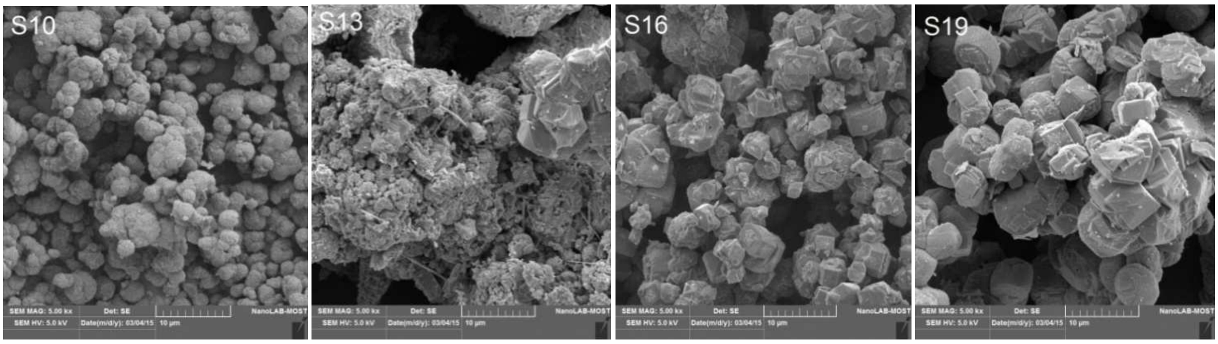


Fig. 5. XRD patterns at different crystallization temperatures (150,160,170 and 180 C) for different crystallization times (48,72,and 96h).

Group 1 for 72h and different temperature 180,170,160,150 C.



Group 2 for 96h and different temperature 180,170,160,150 C.

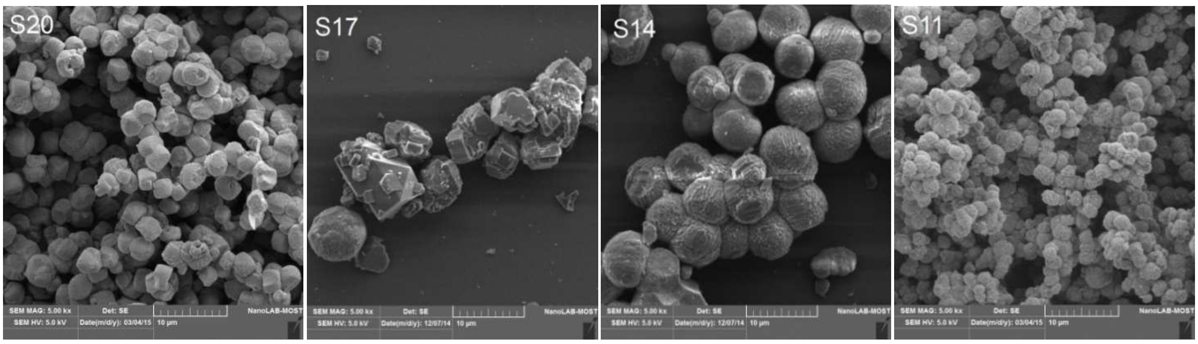
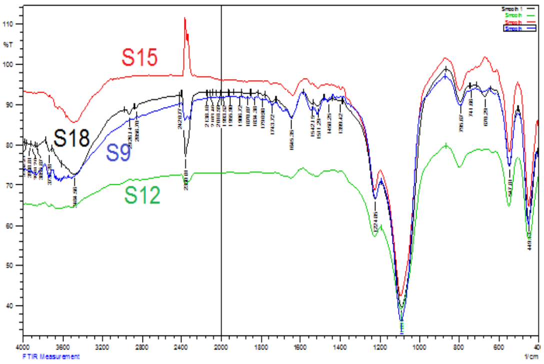


Fig. 6. Scanning electron microstructure images.



for 48h

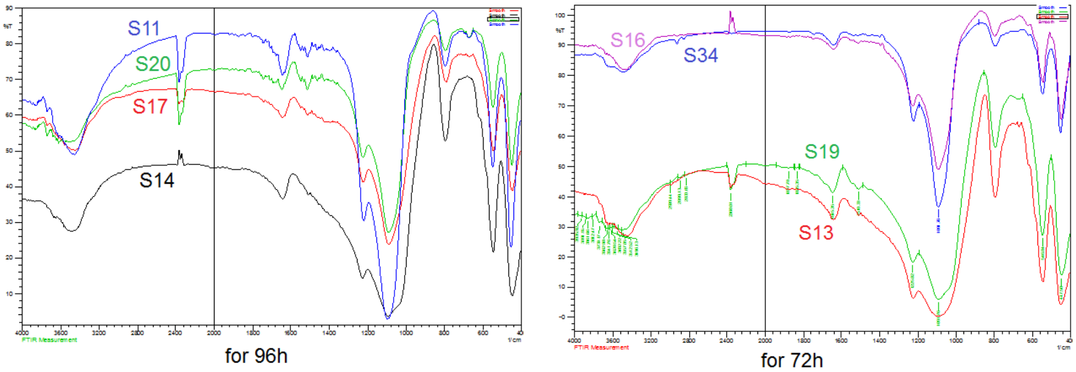


Fig. 7. FTIR spectra for the samples prepared at variuos crystallization time (96h,72h,48h) and temperatures of (180,170,160 and150 C).

5. Conclusion

Nano- Crystalline ZSM-5 was synthesized with considerably matched specifications with theory and previous works. A hydrothermal method was adopted using twin autoclaves carefully designed to meet the requirements(heating, stirring, sealing). The effect of temperature and time of the crystallization also the concentration of template solution on final product specifications like morphology and crystallization were studied. The result showed that the crystallization time, which is the most important parameter, and has a great effect on crystallinity and particle size, crystallization time between 72h-96h gave the best results. Crystallization temperature had also a strong effect on crystallinty, purity and morphology of the prepared ZSM-5 zeolite .it has noticed that an increase in temperature range between (170 C-180 C),tends to get an increase in crystallinity, which is coincided with previous work[1].Template(OSDA)solution concentration showed that the increase of weight percent of the solution from 10% to 20% to 40% has its strong effect on both increase of crystallinity and decrease of particle size to nano-level size.

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تصنيع (تركيب) و وصف الخصائص للزيولايت النانوي-المتبلور نوع ZSM-5

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الخلاصة

الزيولايت النانوي- المتبلور نوع ZSM-5 قد تم تصنيعه (تركيبه) بالطريقة المائية – الحرارية وباستخدام المادة الكلابية تم تصنيع تركيبتين هلاميتين:
التركيبة الاولى: $Al_2O_3:86SiO_2:5.5TPA:12.7Na_2O:3.4Trien:3320H_2O$:
التركيبة الثانية: $Al_2O_3:68SiO_2:5.4TPA:10Na_2O:2.6Trien:2626H_2O$

دراسة تاثير عوامل التفاعل المائي – الحراري على خصائص زيولايت ZSM-5 ذي الحجم النانوي (المتناهي الصغر).من هذه العوامل درجة الحرارة والتبلور و زمن التبلور و تركيز المحلول للمادة العضوية المنظمة للتركيب. التصنيع اجري في اوتكلافيين (مفاعلين) مبطنة بالتفلون الحراري ومزودة بمتحسسين للحرارة و الضغط ومصنعة محليا.اجراء وصف الخصائص تم بوساطة حيود الاشعة السينية,مجهر القوة الذرية والاشعة تحت الحمراء- فورير ترانسبورت وبرونر – ايميت – تيللير (امتصاص و امدصاص للنيتروجين) لقياس المساحة النوعية وحجم المسامات. نتائج الفحص لحيود الاشعة السينية والمجهر الالكتروني الماسح اوضحت بان درجة التبلور للزيولايت المنتج تزداد بزيادة درجة الحرارة و زمن التبلور.واوضحت نتائج مجهر القوة الذرية الحصول على مستوى نانوي وبحدود ٥٠ نانو متر لمعدل قطر الحبيبي.نتائج فحص الاشعة تحت الحمراء- فورير ترانسبورت اكدت الحصول على الحافات المطلوبة والقريبة من الاطوال الموجية١٠٨٠,٨٠٠,٥٥٠ سم-١ فضلا عن ذلك فان النتائج المتعلقة بدرجة التبلور والحجم الحبيبي النانوي اشارت الى تاثير قوي لتركيز المادة العضوية المنظمة للتركيب.
