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# The effect of hydrothermal conditions on surface properties of synthesized nano SBA-15 using sodium silicate

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## Abstract

A successful synthesis of mesoporous Santa Barbara amorphous (SBA-15) with nanoparticle size was attempted using prepared sodium silicate from Iraqi high silica sand. EO20PO70EO20 copolymer was used as a template at highly acidic conditions (pH < 2). The effect of crystallization temperature of (100, 110, 120, and 130 $^0$C), and crystallization time of (24, 48, 72, and 120 h) on surface properties was studied. The experiments were characterized using XRD, FTIR, AFM, BET, and FESEM. The XRD and FTIR tests represent amorphous SBA-15 without any impurities. Decreasing average particle size distribution increased the surface area, and decreased the porosity (pore volume, and pore size) in all experiments. The texture properties were surface area of 210-756 cm2/g, volume pore of 0.28-0.7 cm3/g, and pore size of 1.94-13.45 nm. The optimum result of surface area was achieved at hydrothermal conditions of 110 $^0$C, and 24 h, while optimum results of pore volume, and pore size were achieved at hydrothermal conditions of 100 $^0$C, and 120 h. Semi-spherical shape of particles appeared at hydrothermal conditions of 120 $^0$C and 24 h. This morphology was transferred to small rods at 130 $^0$C, and to semi-platelets at 120 h.

*Keywords: SBA-15; Sodium silicate; surface properties; Hydrothermal temperature; Hydrothermal time.*

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## 1- Introduction

Porous materials are solids that contain pores, with a pore volume fraction of 0.2-0.95 of the total volume. These materials can be classified into (micro, meso, and macro) porous according to pore size. Due to selective size, shape, and thermal stability, microporous materials are used in limited applications concentrated on the separation of fine chemical particles [1]. Mesoporous materials are preferred for their highly ordered mesostructured, apart from the high surface area, which allows diffusion and adsorption of larger molecules for wider applications than that of microstructure due to literature M41S, and SBA groups are the most famous ordered mesoporous materials. [2, 3, 4].

The first synthesis of mesoporous materials type MCM41 was attempted in 1990. This type has a high surface area with narrow pore size, which makes it suitable for some applications like, hosts for catalysts, drug delivery systems, solar cells, and others. In 1998, Zhao et al., prepared a mesoporous material with a uniform two-dimensional hexagonal structure, named SBA-15, by using a nonionic surfactant called Pluronic 123 as a template in a highly acid media (pH < 2). Compared with microporous MCM-41, this material is characterized by larger pore size, thick wall, and high thermal stability during applications [5, 6].

In recent years many researchers have focused on overcoming the challenges of making a balance between the surface properties of prepared SBA-15 like; nanoparticle size distribution, pore size, surface area, and ordered morphology. These studies were concerned with varying conditions such as pH of media, micelle expanders, added salts, removal of surfactant, silica source, repining and aging temperatures [7, 8]

Synthesis temperature, and silica source, are responsible for the interaction with micelles of P123, so they are responsible for the behavior of the hydrolysis and condensation stages during SBA-15 synthesis, which affect the texture properties of the final product [9-11]. The common silica sources are expensive and toxic chemical materials like; tetramethylorthosilicate (TMOS) or tetraethylorthosilicate (TEOS), and sodium silicate [12].

Several hydrothermal treatments were attempted using TEOS as a silica source at different aging temperatures and aging times. Thahir et al. [13] investigated the synthesis of SBA-15 using TEOS as a silica source at an aging temperature between (100-120) $^0$C, and an aging time of 48, and 96hrs. The optimum texture properties were 560 m2 /g, 1.57 cm3/g, and 11 nm pore size obtained at 100 $^0$C, and 48 h. Kruk and Cao [14] synthesized SBA-15 using TEO as a silica precursor at an aging temperature of 40-130 $^0$C, and aging time of 1-5 days, using hexane as a micelle expander and found that surface area was decreased from 900 to 310 cm2/g, while pore size and pore volume were increased from 5 to 15

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nm, and from 0.7 to 1.7 cm3/g respectively. Generally increasing hydrothermal temperature from 60 to 130 $^0$C and hydrothermal time over 12 h decreased surface area and increased the porosity of the product.

To keep environments away from pollution, green synthesis of SBA-15 must be achieved using natural silica sources, and different types of agricultural wastes to prepare sodium silicate [15-17]. However, the effect of thermal conditions on the behavior of the interaction between sodium silicate and the micelles of p123 was still unknown. So, this research was concerned with the effect of aging temperature and time on the texture properties of the prepared SBA-15.

## 2- Experimental materials and methodology

### 2.1. Experimental materials

Prepared alkaline sodium silicate with ($SiO_2$/ $Na_2O$) =1 wt. ratio was used as a silica source. Pluronic (p123) tri-block copolymer with Mw = 5800 purchased from Macklin Co. Shanghai, China was used as a template. For strong acid media during SBA-15 preparation, hydrochloric acid (HCl, 37%) from CDH was used.

### 2.2. Methodology

- Synthesis of SBA-15

Sodium silicate was prepared according to the procedure reported by Kareem, and Hussein [17]. An aqueous solution of sodium silicate was mixed with another solution consisting of suitable amounts of p123 dissolved in an acidic solution of 2 M HCl.

The two solutions reached pH < 2 and were stirred for 24 h at 35 ℃. After that, the mixture was allowed to cool to room temperature. The produced solution was filtered and washed several times with deionized water until Na ions were detected from the powder. The resulting powder was dried at 100℃ for 24 h, calcined at 550℃ for 6 h, and labeled ($M_0$).

The above procedure was repeated to achieve two sets of experiments. The first one consists of varying aging temperatures (110, 120, and 130 $^0$C) with an aging time of 24 h labeled as $M_1$, $M_2$, and $M_3$, respectively, while the second set consists of varying the aging time to 48, 72, and 120 h with an aging temperature of 120 $^0$C labeled as $M_4$, $M_5$, and $M_6$, respectively.

## 3- Experimental analysis and instruments

The identity of prepared SBA-15 was tested using multifunction X-ray diffractometer model Dx2700AB with silicon drift detector SDD, Haoyuan company, China with Cu Kα radiation at 30kv, 20 mA, the tests were attempted at 2Θ angle from (10-80)$^0$. The morphology of prepared samples was estimated using Inspect f50, FEI Company, Holland located at Al-Khora Company, Baghdad, Iraq. The texture properties (surface area, pore volume, pore size) were measured using (BET) Technique with Thermo Analyzer, USA, located at the petroleum research and development center – ministry of Oil, Baghdad, Iraq. The main chemical groups of prepared SBA-15 were obtained using a buker (ALPHA) FT-IR spectrometer in the transmission range between 400-4000 cm$^{-1}$. Finally, the nanoscale range tests were measured using Atomic Force Microscopy (AFM), (Angstrom AA2000). The last two tests were measured at the Department of Chemistry, College of Science, Al-Nahrain University, Baghdad, Iraq.

## 4- Results and discussion.

### 4.1. X-ray diffraction analysis (XRD)

Fig. 1 represents higher XRD peaks using different aging temperatures (100,110,120, and 130 °C) at an aging time of 24h, while Fig. 2 shows the XRD peaks after varying the aging time of 24, 48, 72, and 120 h at an aging temperature of 100 °C.

All the tests were attempted at 2Θ of 10-80, producing a lonely amorphous peak located at 2Θ of 20-25 corresponding to the planner (100) of the standard SBA-15 peaks [18]. Broadening in peaks was clearly shown because of the p123 template, which was released from the powders after calcination [19].

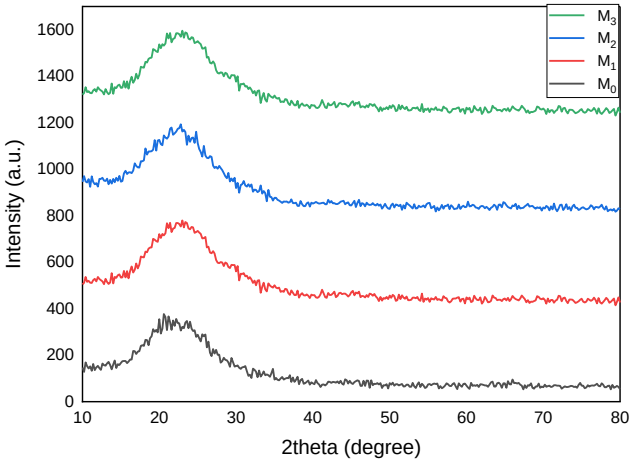


**Fig. 1.** XRD of the Prepared SBA-15 at Aging Time of 100,110,120, and 130°C at 24 h

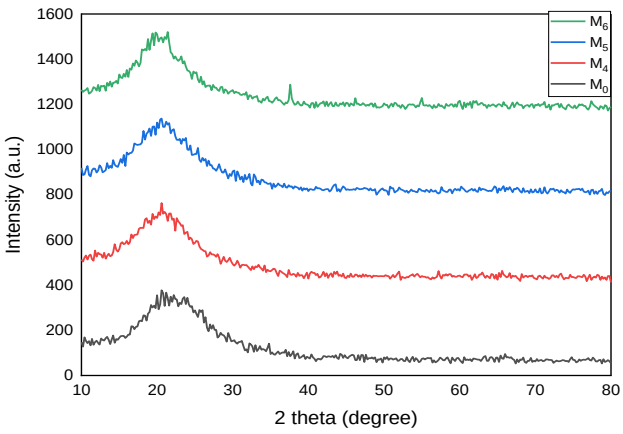


**Fig. 2.** XRD of the Prepared SBA-15 at Aging Time of 24,48,72, and 120 h and Aging Temperature of 120 °C

Also, there is no effect of varying aging temperature and time on d- d-spacing values shown in Table 1, while these variables caused the moving of lonely standard 100 peaks to lower 2$\theta$ values [20, 21].

Increasing aging temperature over 110 $^0$C causes a slight increase in intensity and pore size of prepared SBA-15 at an aging temperature of 24 h. While using aging time over 24 h caused a clear increase in these values, and recorded another small peak called (110) reflection peak at the aging time reached 120 h. This behavior agreed with another work obtained by Calavia, [22].

**Table 1.** Average Particle Size and BET Results of Prepared Samples

| Experiment code | Av. P. size (nm) | S.A. (cm2/g) | P. V. (cm3/g) | P. S. (nm) |
|---|---|---|---|---|
| $M_0$, 100$^0$C, 24 hrs. | 69.9 | 577 | 0.28 | 1.98 |
| $M_1$, 110$^0$C, 24 hrs. | 53.4 | 756 | 0.36 | 1.94 |
| $M_2$, 120$^0$C, 24 hrs. | 55.8 | 741 | 0.4 | 2.16 |
| $M_3$, 130$^0$C, 24 hrs. | 67 | 685 | 0.38 | 2.21 |
| $M_4$, 100$^0$C, 48 hrs. | 80.5 | 511 | 0.48 | 4.17 |
| $M_5$, 100$^0$C, 72 hrs. | 83.9 | 419 | 0.51 | 4.87 |
| $M_6$, 100$^0$C, 120 hrs. | 99.7 | 210 | 0.7 | 13.45 |

4.2. FTIR spectroscopy analysis

The main chemical groups in mesoporous SBA-15 prepared from sodium silicate like; Si=O, Si-O-Si, and Si-OH, using different aging temperatures between (100-130)$^0$C, and different aging times of 24-120 h were observed in Fig. 3 and Fig. 4 using FTIR analysis. The stretching vibration of the silanol groups (Si=O) appeared at a wavelength between 450 and 480 cm-1, while the peaks around 800 cm-1 refer to weak vibration of Si-O-Si groups [6,23,24]. The strong vibration peaks of the same chemical groups were indicated between 1020 and 1120 cm$^{-1}$ [25]. The peak at 2890 cm $^{-1}$ indicates the formation of Si–OH groups found in the experiments using an aging time of 24 h, while this chemical group disappeared after 24 h because of the dehydration of PEO chains in p123 micelles towards the PPO core increasing its volume, which affects the porosity of the prepared samples. [20]. There are no broad bands behind the region of 3400cm-1 because of the absence of –OH groups in all prepared samples [26].

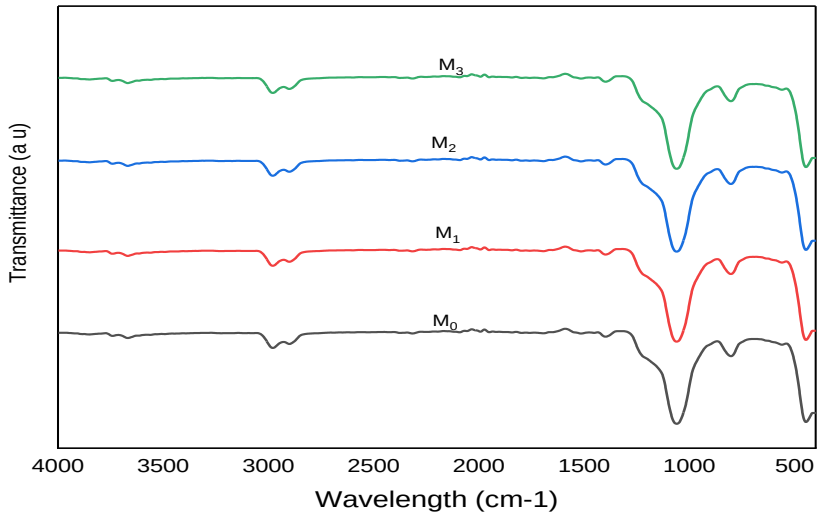


**Fig. 3.** FTIR of the Prepared SBA-15 at Aging Time of 100,110,120, and 130°C at 24 h

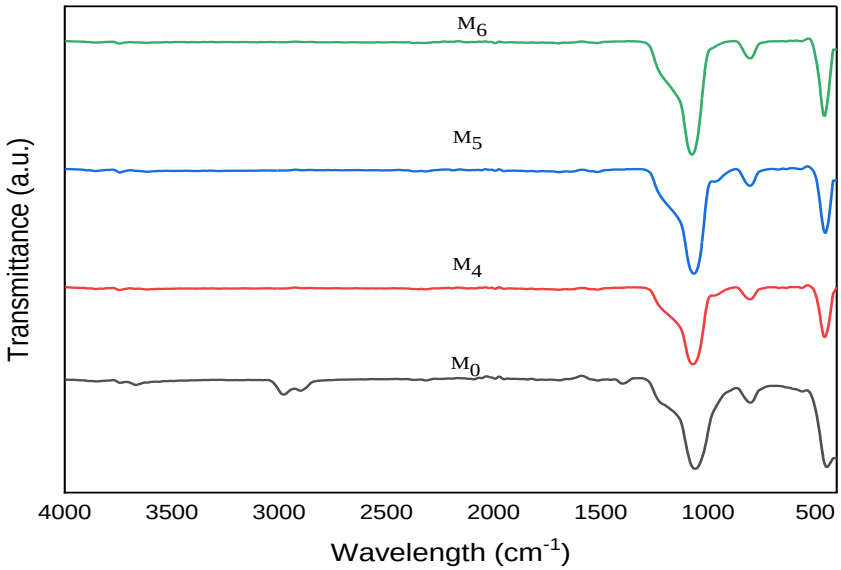


**Fig. 4.** FTIR of the Prepared SBA-15 at Aging Time of 24,48,72, and 120 h and Aging Temperature at 120°C

4.3. Particle size and BET analysis

The average particle size, and surface properties of the prepared samples at aging temperatures of 100-130 °C, and aging time of 24-120 h are listed in Table 1 and Fig. 5. The nanoscale particle sizes were 53-99 nm, while the surface area of 210-756 $cm^2/g$, a pore volume of 0.28-0.7 $cm^3/g$, and a pore size of 1.94-13.45 nm.

Generally, the dispersion of particles (decreasing particle size) increased the surface area and decreased the pore volume and pore size. Increasing aging temperature over 100 °C led to an increase in the nucleation rate and a decrease in the nanoscale particle size after comparison with SBA-15 samples attempted at 100 °C. The nucleation rate had reached its optimum values at 110, and 120 °C producing an average particle size of 53-55 nm, while raising the aging temperature to 130 °C increased aggregation degree and enlargement of its particle size, which reached over 67 nm. This behavior matched the behavior reported by Joni et al. [27]. Although the degree of aggregation has a proportional relationship with aging time, which led to clear enlargement in particle size reached over 99 nm at aging time of 120 h.

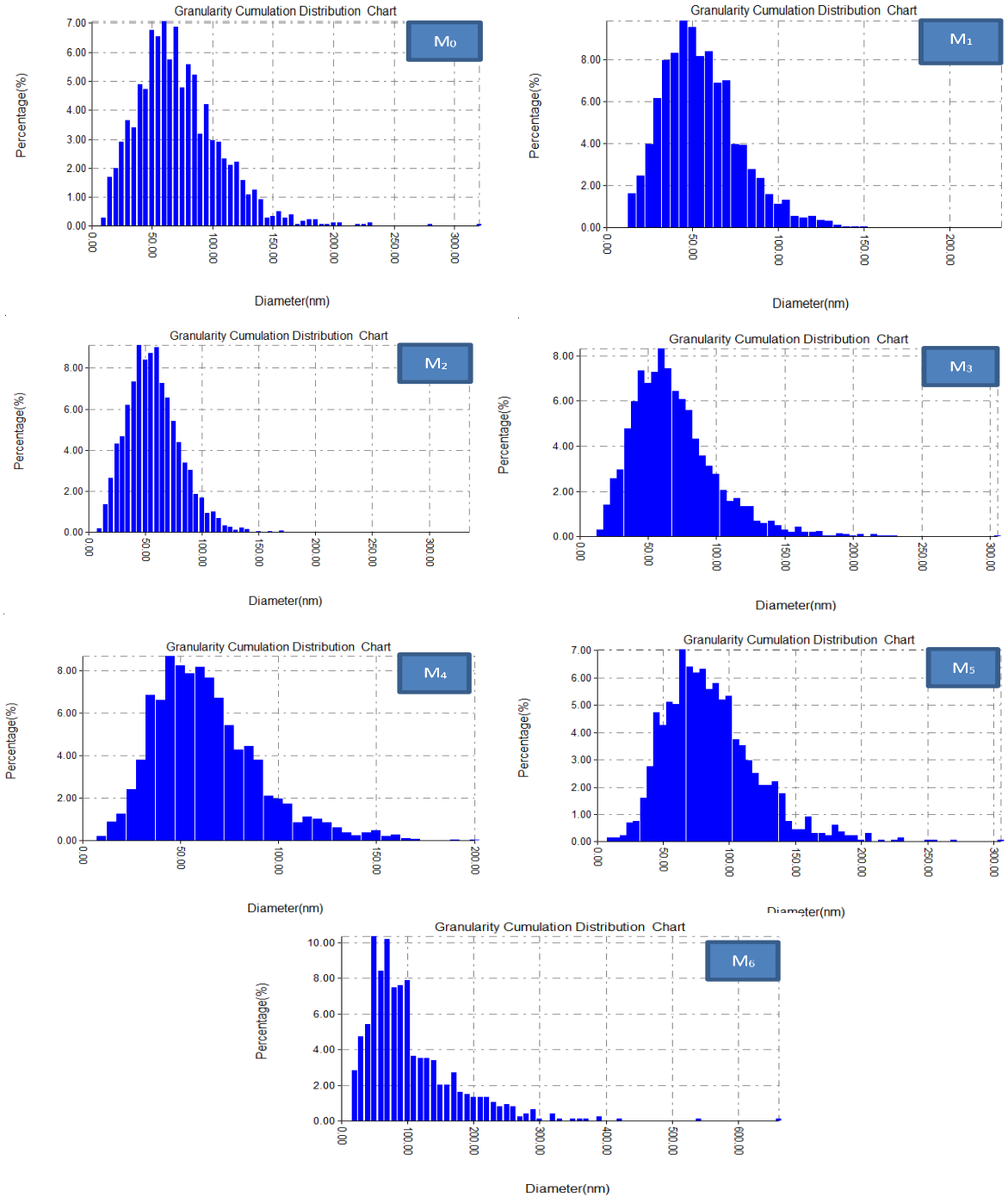


**Fig. 5.** Particle Size Distribution for Prepared SBA-15 at Aging Temperatures of 100, 110,120, and 130$^0$C, and aging time of 24,48,72, and 120 hr

The texture properties of the prepared SBA-15 were influenced by the behavior of reactions between PEO-PPO chains of micelles and silica sources (organic, and inorganic) at different hydrothermal temperatures, and times [28].

Increasing aging temperature over 100 °C increased the surface area with a small influence on pore volume, and pore size of prepared samples. The results of the surface area reached its high value of 756 $cm^2/g$ at 110 °C. These values were dropped to 685 $cm^2/g$ at 130 °C, while pore volume and pore size obtained in the temperatures of 110-130°C were at the range of 0.4 $cm^3/g$, and 2 nm, respectively. This behavior happened because these experiments were prepared at 24 h which represents a short aging time for PEO dehydration from the silica wall to react with the PPO core region [29]. In comparison with reactions of PEO_PPO Pluronic micelles with inorganic silica source like; TEOS. It had different behavior using almost the same aging temperatures. The microporosity of SBA-15 decreased sharply at temperatures above 100°C due to PEO dehydration from the silica wall to the PPO region increasing the pore size of prepared samples [30]. For example, Hartman et al., [31] synthesized SBA-15 using TEOS as a silica source with pore volume and pore size reaching 1.6 $cm^3/g$, and 9 nm respectively at an aging temperature of 100°C, while these results reached 1.1 $cm^3/g$ and 10 nm at 130 °C.

On the other hand, increasing the aging time from 24 to 120 h for the experiments attempted at 120 °C caused aggregation of particles (increased size) from 55.8 nm to 99.7 nm which decreased surface area from 741 to 210 $cm^2/g$, and increased the pore volume from 0.4 to 0.7 $cm^3/g$, and pore size from 2.16 to 13.45 nm, respectively. The results showed that only time has a great effect on increasing the pore size of SBA-15 particles from the microporous region to the mesoporous one after increasing the hydrothermal treatment time over 24 h. Also, in comparison with previous work conducted by Kruk et al., [14] used TEOS as a silica precursor for the synthesis of large mesoporous SBA-15 with a pore volume of 1.3 $cm^3/g$, and pore size of 16.5 nm at 120°C, and 24 h.

### 4.4. Morphology analysis

The morphology of the surface of synthesized SBA-15 at varying aging temperatures of 100, 110, 120, and 130°C, and aging times of 24, 48, 72, and 120 h is shown in Fig. 6. The morphology of SBA-15 synthesized at 100°C, and 24 h had a semi-spherical shape. This morphology was obtained by Tasfy [31]. Increasing the aging temperature to 120°C did not affect this morphology but caused the dispersion of particles and reduced its particle size to form clusters of semi-spherical particles. Rising the aging temperature to 130 °C caused a deformation of the semi-spherical particle to a small rod shape. This behavior matched another work reported by Serrano et al. [32]. On the other hand, increasing the aging time to 48 h caused agglomeration of the semi-spherical particles. This agglomeration increased with increasing the aging time converting the morphology to semi-platelet at an aging time of 120 h with large voids between particles.

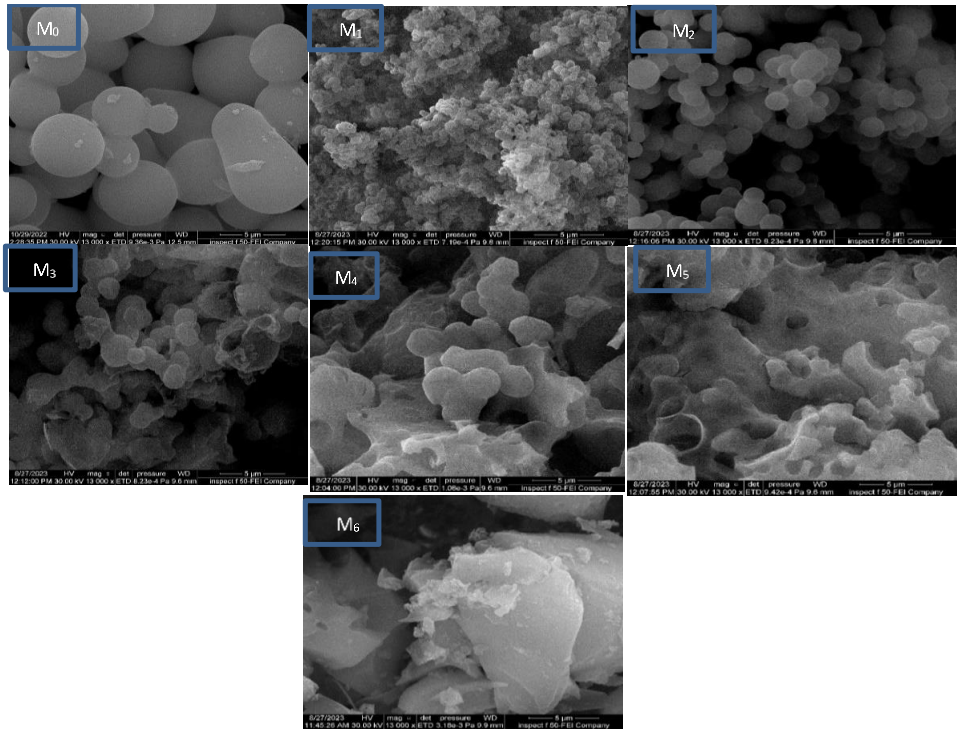


**Fig. 6.** FESEM of Prepared SBA-15 at Aging Temperatures of 100, 110,120, and 130°C, and Aging Time of 24,48,72, and 120 hrs

## 5- Conclusions

1. This work shows the successful preparation of SBA-15 using Pluronic p123 surfactant, and sodium silicate synthesized from Iraqi silica sand at variable hydrothermal temperatures and times. The average particle size distribution of all samples was in the nanoscale range.
2. SBA-15 synthesized at 100 $^0$C, and 24 h had microporous properties. These properties were enhanced by increasing the aging temperature over 100 $^0$C, and the morphology was converted from semi-spherical to small rod shape.
3. Mesoporous properties were modified when the aging time was raised over 24 h and reached their optimum values at an aging time of 120 h with a semi-platelet shape.
4. The synthesized SBA-15 with a large surface area, acceptable pore volume, and pore size can be used as a support in variable applications like; catalyst, storage, drug delivery, and others.

## Nomenclature

| | |
|---|---|
| Santa Barbara Amorphous | SBA-15 |
| Mobil Composition of Matter | MCM-41 |
| tetetramethylorthosilicate | TMOS |
| tetraethyorthosilicate | TEOS |
| Poly Ethylene Oxide-Poly Propylene Oxide | PEO-PPO |

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# تأثير الخواص الحرارية على خصائص السطح لل(SBA-15) باستخدام سيليكات الصوديوم

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**الخلاصة**

تم تحضير سانتا باربرا SBA 15 بنجاح بأقطار نانوية وبأستخدام سليكات الصوديوم المصنعة سابقا من الرمال العراقية عالية السليكا. تم أستخدام مادة البلورونك p123 كمشتت وكقالب للتفاعل في جو تفاعل عالي الحامضية (رقم هيدروجيني <٢) وتم خلال التصنيع دراسة تأثير درجة حرارة التحلل المائي بأستخدام درجات حرارة (١٣٠,١٢٠,١١٠,١٠٠) م˚ و بأوقات مختلفة من (٢٤, ٤٨, ٧٢, و١٢٠) ساعة. تم تشخيص العامل المساعد النانوي المحضر بأستخدام أجهزة حيود الاشعة السينية, الاشعة تحت الحمراء, برونر- أيميت- تيللير (أمتصاص وأمتزاز النتروجين), مجهر القوة الذرية و المجهر الالكتروني الماسح. أن فحوصات حيود الاشعة السينية والاشعة تحت الحمراء أثبتت تصنيع عامل مساعد غير متبلور نقي وخالي من الشوائب. وأن أستخدام درجات حرارة عالية فوق ١٠٠م˚ خلال تفاعلات التحلل المائي أدت الى تشتت دقائق العامل المساعد وزيادة مساحته السطحية على حساب أنخفاض حجم وقطر الفجوات.

أن الخواص السطحية للعامل المساعد المحضر تتراوح بين (٢١٠-٧٥٦) سم$^{٢}$/ غرام مساحة سطحية و بين (٠,٢٨-٠,٧) سم$^{٣}$/غرام حجم فجوات و بين (١,٩٤-١٣,٤٥) نانو متر قطر فجوات. الظروف المثالية للتحلل الحراري التي سجلت أعلى مساحة سطحية كانت عند درجة حرارة ١١٠م ˚ ووقت ٢٤ ساعة, أما الظروف المثالية التي سجلت أعلى نتائج لحجم وقطر الفجوات كانت عند دلاجة حرارة ١٢٠م˚ ووقت ١٢٠ ساعة. وأخيرا تم الحصول على شكل شبه كروي للدقائق المصنعة عند حرارة ١٠٠م ˚ ووقت ٢٤ ساعة هذا الشكل يتحول الى شكل العصا القصيرة عند حرارة ١٣٠م ˚ و الى شكل أشباه الالواح عند وقت تحلل حراري ١٢٠ ساعة.

**الكلمات الدالة:** SBA-15، سيليكات الصوديوم، خصائص السطح، حرارة التفاعل، وقت التفاعل.