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RESEARCH ARTICLE

Mesoporous SBA-15 Synthesis and Characterization Using Various Dispersing Agents

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ABSTRACT

A successful synthesis of SBA-15 with semi-spherical morphology and suitable texture properties was achieved using hydrothermal technique. Sodium silicate, derived from silica sand from Iraq, was used as a silica precursor, and Pluronic p123 triblock copolymer was used as a template with a strong acid media (pH < 1). The effect of adding (1–3) g of either polyvinyl alcohol (PVA), Polyvinyl pyrrolidone (PVP -k30), or Sodium dodecyl sulfate (SDS) dispersant agents on the texture properties of the synthesized SBA-15 was studied. The SBA-15 sample underwent various characterization techniques, including X-ray diffraction (XRD), Fourier transform infrared (FTIR), atomic force microscopy (AFM), nitrogen adsorption-desorption using Brunauer Emmett Teller (BET), and field emission scanning electron microscopy (FESEM).

XRD analysis showed a single peak in the $2\theta = 20–25^{\circ}$, indicating the presence of pure SBA-15. The FTIR spectroscopy results demonstrated presence of key functional groups like silanone, siloxane, and silanol. Using these dispersant agents had a discernible effect on the dispersion of particles to an average particle size distribution in a nanoscale range producing a high surface area of the prepared SBA-15 material, which ranged between 349 to 871 cm^2/g, while not influencing the pore volume, and pore size, which are ranged between 0.17 to 0.43 cm^3/g, and between 2 to 2.5 nm, respectively. Also, all prepared samples had a nano-scale range.

The optimum texture properties were obtained using 2 g of PVP-k30 dispersion agent. Finally, it was observed that the spherical shape of SBA-15 particles was distorted by introducing more than 1 g of dispersion agents.

Keywords: Dispersing agents, Polyvinyl pyrrolidone, SBA-15, Sodium dodecyl sulfate, Sodium silicate

Introduction

As to the International Union of Pure and Applied Chemistry (IUPAC) materials, exhibiting porosity within the range of 0.2–0.95 can be categorized based on their pore size. These categories include (macro, meso, and micro) porous materials with an organized six-sided polygonal structure and large volumes of porosity. Nanoparticle materials with pore size over 2 nm are synthesized utilizing various silica sources such as kaoline and ash of variable agricultural crops.[1–3] SBA_15 is a well-known example of a mesoporous material.[4,5] It possesses a high capacity for metal loading,[6] a large volume of pores for diffusion limitation of large particles suspended in the inlet[7,8] and a pore thickness over 3 nm that contributes to its hydrothermal stability.[9]

SBA-15 has exceptional characteristics that render it very appropriate for a diverse range of applications, including catalyst utilization,[10,11] water treatment processes,[12] sensor development and drug delivery,[13,14] and advancements in the medical domain.[15]

Although SBA-15 possesses commendable characteristics, such as surface area and pore volume, its textural qualities and morphology can still be improved through modifications achieved by altering the preparation conditions. The pioneering preparation attempt of SBA_15 was undertaken by D. Zhao

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et al.[16,17] utilizing the non-Ionic (P123) in a high acid environment with a pH of 1. Subsequently, numerous endeavors have been made to alter the characteristics of SBA-15 by utilizing diverse synthesis circumstances. In their study, Zuhairi et al.[18] examined the impact of pH on surface area, pore size, and pore volume. They observed a drop in texture properties from (680 – 489) m^2/g, while the diameter of the pores grew from 3.64 nm to 4.88 nm, and pore volume increased from 0.38 cm^3/g to 0.59 cm^3/g as the pH climbed from 1.3 to 3.0. The impact of temperature on the properties of a material was investigated by Hartmann and Vinu.[19] Their findings revealed that an increase in temperature within the range of 100–150 ˚C led to a decrease in surface area from 1100 to 393 cm^2/g and a decrease in pore volume from 1.6 to 1.10 cm^3/g. However, there was an observed increase in pore size from 9 to 11.2 nm. Aumond et al.[20] conducted a study on the removal of surfactant during the drying and calcination process at various temperatures ranging from 300 to 550 ˚C. Their results demonstrated an increase in pore size from 7.7 to 9.6 nm, along with an increase in pore volume from 0.73 to 0.97 cm^3/g.

The utilization of dispersion agents was seen to be efficacious in enhancing a certain textural characteristic of the manufactured silica support. Prior research has demonstrated that trimethylbenzene (TMB) as a dispersion agent augments pore diameter over 10 nm while reducing surface area concurrently.[21] Using polyvinyl alcohol (PVA) as a dispersant agent was attempted by Ridhawati Thahir et al., who synthesized SBA-15 utilizing triethyl orthosilicate as a silica precursor augments the surface area of 892 cm^2/g using 1 g PVA, and reached to over 1700 cm^2/g after using 2 g, at the same time, the pore volume remains unaffected.[22]

The objective of this work is to examine the impact of several types of dispersing agents, such as polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP-k30), and sodium dodecyl sulfate (SDS), on the properties of prepared mesoporous silica using prepared sodium silicate as a silica source.

Methodology

Chemicals and raw materials

The Iraqi sand, which constitutes 96% of silica, is utilized as a precursor for sodium silicate and was provided by state company for mining industries. Merck, a trusted supplier, provided the sodium hydroxide (99%). The tri-block copolymer, consisting of poly (ethylene glycol), Poly (propylene glycol), and poly (ethylene glycol) with a molecular weight (Mw) of 5800, was procured by Thermo Fisher. 37% (HCl) from Sigma-Aldrich regulated the pH of the samples. Various dispersing agents were employed in the study, such as polyvinyl alcohol (PVA) with a molecular weight of 67000 from CDH, Polyvinyl Pyrrolidone (PVP K30) with 40000 molecular weight obtained from CDH, and Sodium dodecyl sulfate (SDS) with 288.37 molecular weight which provided from Sigma-Aldrich. Additionally, deionized water was used.

Synthesis of sodium silicate

Sodium silicate was synthesized using the blend comprising 96% sand sourced from Iraq and 99% sodium hydroxide for the SBA_15 catalyst precursor. The sand underwent an initial milling process using an appropriate commercial miller. It was then subjected to a washing procedure using deionized water. Subsequently, the sand was dried at a temperature of 100 ˚C for the duration of one night. Finally, the sand was screened using a mesh with a pore size of 63 μm.[22] Then, the sand was combined with varying weight ratios of NaOH to sand (1:1, 1.2:1, and 1.4:1) within ceramic crucibles and subjected to different fusion temperatures (400, 500, and 600 ˚C). The desired outcome was achieved when the experiment was conducted at a temperature of 600 ˚C and a ratio of NaOH to sand 1:1.

Synthesis of SBA_15

Solution A was prepared by dissolving 3 g. nonionic surfactant in 175 g of (2M) Hydrochloric Acid. A solution B was prepared by dissolving 4 g of sodium silicate in 45 g of water.

Solution A and B were combined, resulting in a solution with a pH of 1. The mixture was then aged for a duration of 24 hours at a temperature of 35 ˚C. After that, the solution was subjected to an aging process in an oven set at a temperature of 100 ˚C for 24 hours. Next, the combination was permitted to cool down to the ambient temperature. The generated solution underwent filtration and subsequent washing of deionized water to acquire the white powder. These particles were then subjected to a drying process at a temperature of 100 ˚C for a duration of 24 hours. The SBA-15 powder underwent a calcination process at a temperature of 550 ˚C for a duration of 6 hours and was afterwards labelled as Y_0.

The methodology, as mentioned above, was replicated by introducing various dispersion agents (polyvinyl alcohol, polyvinylpyrrolidone, and sodium

Table 1. Summary amount of dispersing agent.

code	PVA(g)	PVP (g)	SDS (g)
Y_0	-	-	-
Y_1	1	-	-
Y_2	2	-	-
Y_3	3	-	-
Y_4	-	1	-
Y_5	-	2	-
Y_6	-	3	-
Y_7	-	-	1
Y_8	-	-	2
Y_9	-	-	3

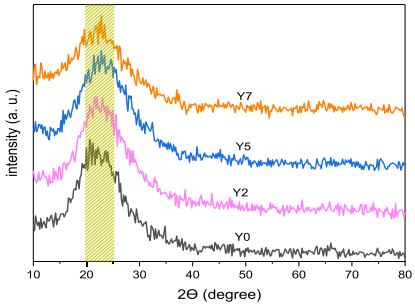

Fig. 1. X-ray diffraction of prepared SBA-15 Y_0, Y_2, Y_5, and Y_7.

dodecyl sulfate) in varying quantities 1, 2, and 3 g of each respective type into the resultant amalgamation. Table 1 presents a summary of the synthesis process for SBA_15.

Characterization

The pattern of phases was determined using an X-ray diffractometer. The experimental setup involved the utilization of a Dx2700AB (x-ray) multifunction diffractometer equipped with a silicon drift detector (SDD) manufactured in China by Haoyuan firm. The diffractometer employed Cu Kα radiation at a voltage of 30 kilovolts and a current of 20 milliamperes. The diffraction data were collected within a scanning range of 2θ angles spanning from 10 to 80 degrees. The surface structure of the produced samples was analyzed using the Inspect f50 microscope manufactured by FEI Company in Holland. The analysis was conducted at AlKhora Scientific office - in Iraq.

The texture properties were determined using the nitrogen adsorption-desorption method employing the Thermo Analyzer from the United States. The measurements were conducted at the Oil Ministry of Iraq. The chemical groups were conducted utilizing the IR-Affinity, Shimazdo, located at Baghdad University. The nanoscale range was tested by an Angstrom Scanning Probe Microscope located at Baghdad University.

Results and discussion

XRD studies

In Fig. 1, we can see the x-ray diffraction synthesis process of SBA-15. The figure shows the process both in the absence of dispersing agents and in the presence of 2 g of each of them. The amorphous shapes produced by the XRD were analyzed at a 2 Θ angle from 10–80°.

The presented figure demonstrates the unique fingerprint of SBA-15, as seen in the characteristic amorphous peak observed at 2Θ = 20–25°. This peak corresponds to the (100) plane with hexagonal planar symmetry (p6mm), which is consistent with established standards.[23]

All the prepared samples show a significant shift in the diffraction angle and the broadening of peaks because of the interaction of P (123) and various quantities of dispersing agents. These agents are canceled out from the structure of silica after calcination, generating a nanoscale of particle sizes.[24]

FT-IR spectral analysis

The function groups of mesoporous SBA_15 like; Si=O, Si-O-Si, and Si-OH groups were identified through FTIR analysis, as shown in Fig. 2. The Si=O bending within the 480 cm^{-1} range corresponds to the stretching vibration of the silanol group. (Si-O-Si) groups observed at 750 and 1020–1120 cm^{-1} range refer to the vibration of siloxane groups.[25,26] The occurrence of the Si-OH group which refers to silanol group formation is suggested by the presence of peaks at 2890 cm^{-1}.[27] The lack of wide bands in the 3400 cm^{-1} range is due to the absence of hydroxyl group in all samples.[28]

Particle size analysis

Figs. 3 and 4 depict the average distribution of particle sizes and three-dimensional images illustrating the topography of the prepared samples. These figures showcase the samples in two conditions: without any dispersing agents and with varying quantities of dispersion agents. The distribution of the particle size of SBA-15 in the absence of additions measures 70 nm. Using 1 g of polyvinyl alcohol (PVA) results in an average particle dispersion of 61 nm. Adding Polyvinyl alcohol (PVA) in quantities beyond 1 g leads to the aggregation and expansion of particles.

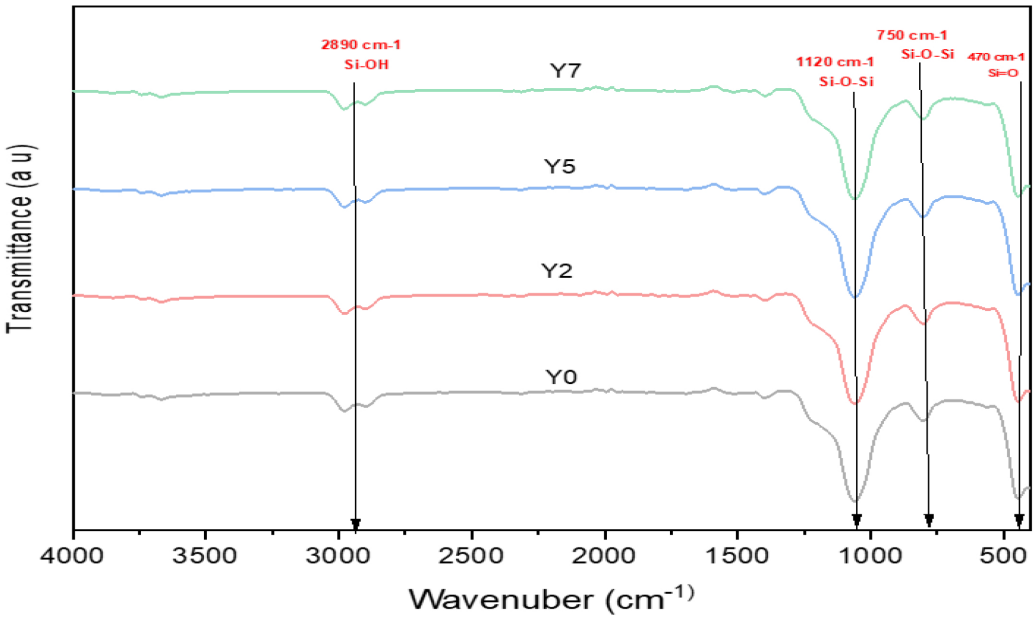

Fig. 2. FTIR of prepared SBA-15 Y0, Y2, Y5, and Y7.

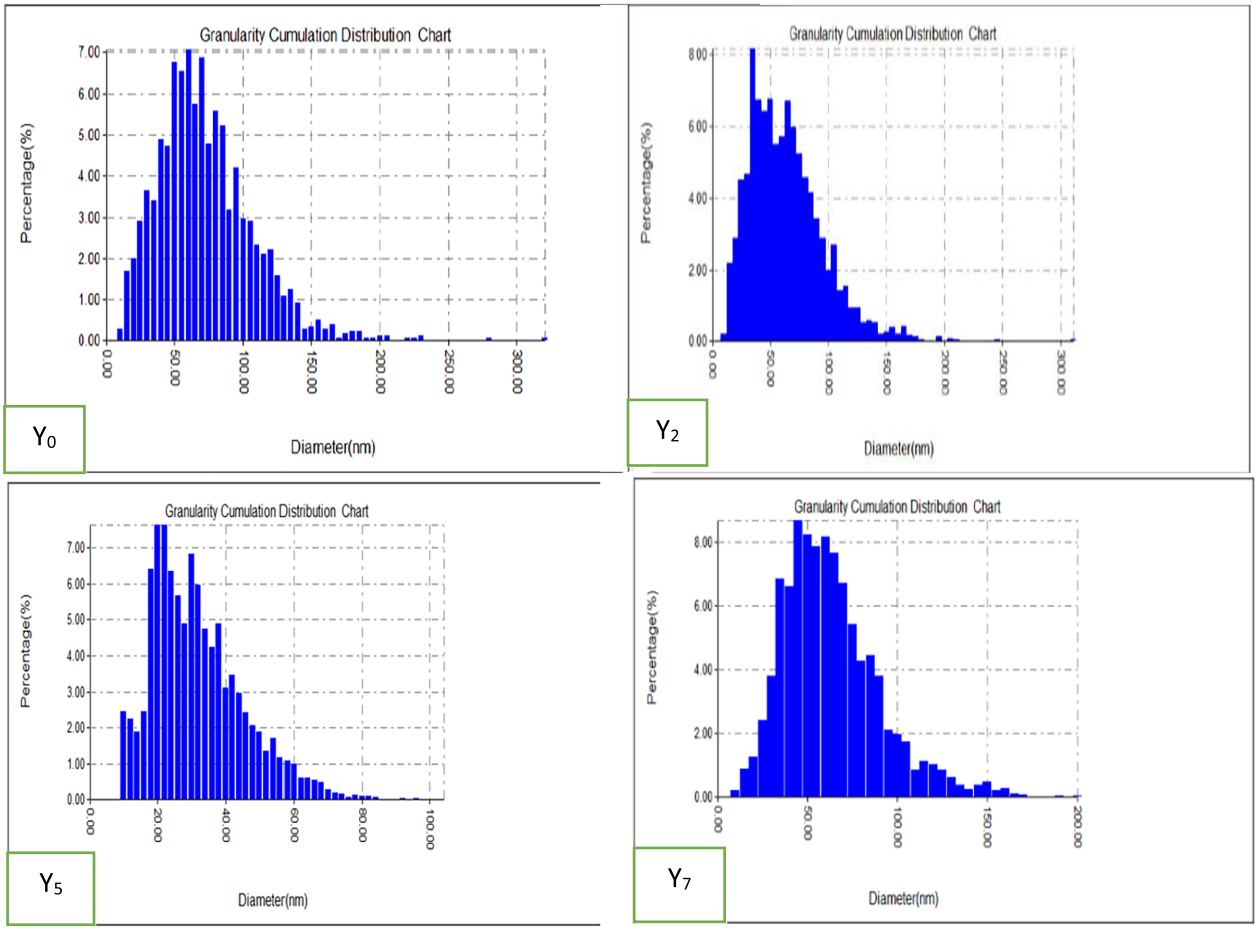

Fig. 3. Particle size distribution for prepared SBA-15 (Y_0, Y_2, Y_5, Y_7).

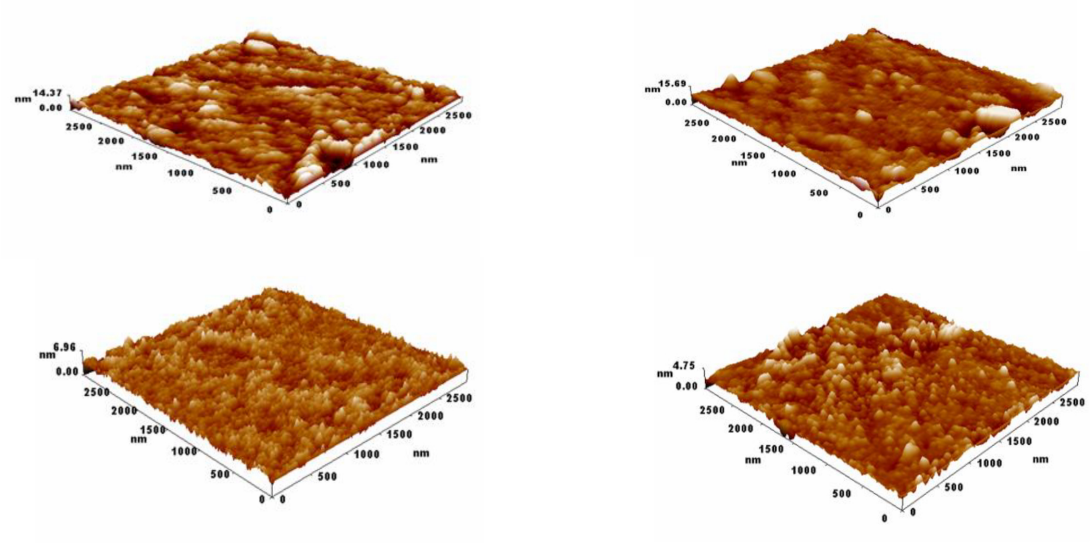

Fig. 4. 3D images of prepared SBA-15 Y_0, Y_2, Y_5, and Y_7.

Table 2. AFM results of SBA-15.

-	Average particle size (nm)	Nanoscale range (%)
Y_o	70	82.43
Y_1	61	88.58
Y_2	82	70.92
Y_3	65	85.85
Y_4	35	99.77
Y_5	31	99.9
Y_6	36	99.29
Y_7	62	91.41
Y_8	73	78.73
Y_9	78	73.95

Aggregates are anticipated to be formed at a PVA addition of 2 g. The experiment revealed a significant dispersion effect at varying concentrations of PVP, with the most positive result observed at a PVP concentration of 2 g, resulting in a particle size of 31 nm and a cumulative nano scale percentage of 99.9. In the context of SDS, it was seen that an escalation in dosage resulted in a rise in the mean particle size, namely from 62 nm–78 nm. This outcome is deemed undesired as it promotes particle aggregation and enlargement, as indicated by previous studies.[29] The attainment of the smallest particle size was accomplished by employing 1 g of polyvinyl alcohol (PVA), 2 g of polyvinylpyrrolidone (PVP), and 1 g of sodium dodecyl sulfate (SDS). Particle size distribution and nanoscale (%) are represented in Table 2.

In the absence of dispersion agents, the surface morphology in three dimensions has a semi-spherical form and undergoes deformation beyond the application of 1 g of each dispersing agent. The observed behavior aligns with the findings derived from the SEM research. The effect of PVP on dispersion is more pronounced than that of PVA and SDS. In general, the introduction of polymers into processes has the potential to generate a viscous medium. This might result in the interaction between the polymers and the metal surface, thereby impeding the diffusion of metal ions. Additionally, the presence of polymers can limit the formation of larger particles from smaller ones.[30] Moreover, the composition of the two polymers employed includes a (CH_2=CH) group that reacts with Si Ions and a polyethylene group, inhibiting their aggregation. Due to the two polymers' structure, the length of vinyl groups of PVP-k30 and PVA were 45 and 176, respectively. Based on the findings, it has been observed that the length of polyethylene in PVP-k30 is optimal for Silica particles to prevent aggregation, thereby facilitating good dispersion and achieving optimum nano-scale percentage. Conversely, the length of polyethylene in PVA is excessively long to ensure perfect encapsulation of each particle, thereby increasing the likelihood of aggregation.[31] In the context of SBA-15 preparation, it is observed that both SDS (sodium dodecyl sulfate) and silica ions possess anionic charges, which results in their mutual repulsion and prevents their attraction to one another. Adding 1 g of sodium dodecyl sulfate (SDS) to the PEO in P123 micelles leads to an increase in hydration, resulting in a reduction in the degree of aggregation between molecules and a decrease in particle size. Utilizing a quantity above 1 g of SDS, which augments the negative charge of the micelles, leads to a repulsive effect among the muscles, hence reducing the thickness of the molecular walls and resulting in the formation of aggregates.

Table 3. Texture characteristics of SBA_15.

-	BET Surface. Area. (m^2/g)	P.S (nm)	P. Vol. (cm^3/gm)
Y_o	578	1.98	0.28
Y_1	719	1.92	0.34
Y_2	349	2.00	0.17
Y_3	549	1.99	0.29
Y_4	865	1.97	0.43
Y_5	871	1.96	0.43
Y_6	850	2.03	0.43
Y_7	714	2.2	0.4
Y_8	626	2.50	0.4
Y_9	562	2.50	0.4

Texture properties characterization

The texture properties of all the experiments were determined by BET analysis. The measurements for the samples without dispersing agents and those with varying doses of dispersing agents are presented in Table 3. The measured values for surface area, pore volume, and pore size fell within the ranges of (349–871) cm^2/g, (2–2.5) nm, and (0.17–0.43) cm^3/g, respectively. Initially, using 1 g of polyvinyl alcohol (PVA), the specimen resulted in an augmentation of both the surface area and pore volume compared to the baseline condition (Y0). Conversely, an inverse relationship was observed between the surface area and volume of the pores when exceeding 1 g of PVA. Specifically, a significant decline in textural properties was observed when utilizing 2 g of PVA due to the occurrence of particle agglomeration during the preparation process. Important increases in textural qualities, with the exception of pore size, were found when different quantities of PVP-k30 were utilized. The quantity of 2 g yielded the highest values due to PVP's pronounced dispersion effect.

The observed increase in surface area and pore volume at a concentration of 1 g of SDS can be attributed to the enhanced hydrophilicity in Pluronic micelles. This hydration effect leads to a reduction in particle aggregation and a decrease in the core radius of P123 micelles. The utilization of more than 1 g of sodium dodecyl sulfate (SDS) can lead to the repulsion of micelles and the occurrence of hydrophobic contacts between poly (propylene oxide) (PPO) in P123 micelles and SDS ions. These interactions result in the opening of channels on the external surface, a reduction in surface area, and the maintenance of a high volume percentage of holes.[6]

Based on the findings, it is evident that the manipulation of PVA, PVP, and SDS in varying quantities significantly affected the surface area of the synthesized SBA_15 materials, with little effect on the volume of the pores, while no discernible effect was observed on pore size. This lack of effect can be attributed to the role of these dispersant agents in increasing the reaction between hydrophilic chains of p123 micelles, and inorganic silica ions (increasing hydrolysis reaction), which happened at fixed crystallization conditions (temperature, and time) so that these reactions must need high temperatures and long periods to impede its integration into the silica network. This phenomenon leads to the enlargement of the PPO chain with increasing the pore wall thickness.[6,32] In comparison with other studies using 1–2 g of PVA with triethyl orthosilicate as a silica precursor producing SBA-15 with high texture properties of surface area of 892 cm^2/g, and pore volume of 1.87 cm^3/g using 1 g PVA, and reached to over 1700 cm^2/g, and 1.4 cm^3/g after using 2 g, at the same aging conditions because of the effect of (OH^-) groups in TEOS structure, which enhance hydration-dehydration reactions producing mesoporous materials with superior texture properties.[22]

Morphology analysis

Fig. 5 displays the surface morphology of some experiments representing mesoporous particles with no additives and with 2 g of (PVA), (PVP-k30), and (SDS) named Y_0, Y_2, Y_5, and Y_7, respectively. In the initial analysis, a comparison was made between the structure of SBA_15 samples prepared with no additives with varying amounts of (PVA). The shape of mesoporous particles with no additives exhibited a hemisphere structure. Using 1 g of PVA leads to particle dispersion and reduction in particle diameter, resulting in well-defined spherical morphology at various magnifications. However, excessive PVA leads to particle merging and distortion of the spherical shape to a lamina one. Furthermore, the use of 2 g and 3 g of PVA causes the formation of clusters of semi-spherical particles. This observed behavior aligns with the findings reported by Wang et al.[29]

Using a PVP dispersing agent leads to enhanced interaction between the template and sodium metasilicate precursor, resulting in a high dispersion of particles within the nanoscale range. However, it should be noted that there is a tendency for the particles to form groups in some places, which ultimately contributes to the development of high textural qualities. The utilization of 1 g of SDS dispersing agent results in the dispersion of particles exhibiting a sphere shape when observed at various magnifications. However, an excessive quantity of SDS leads to the distortion of the particle morphology.

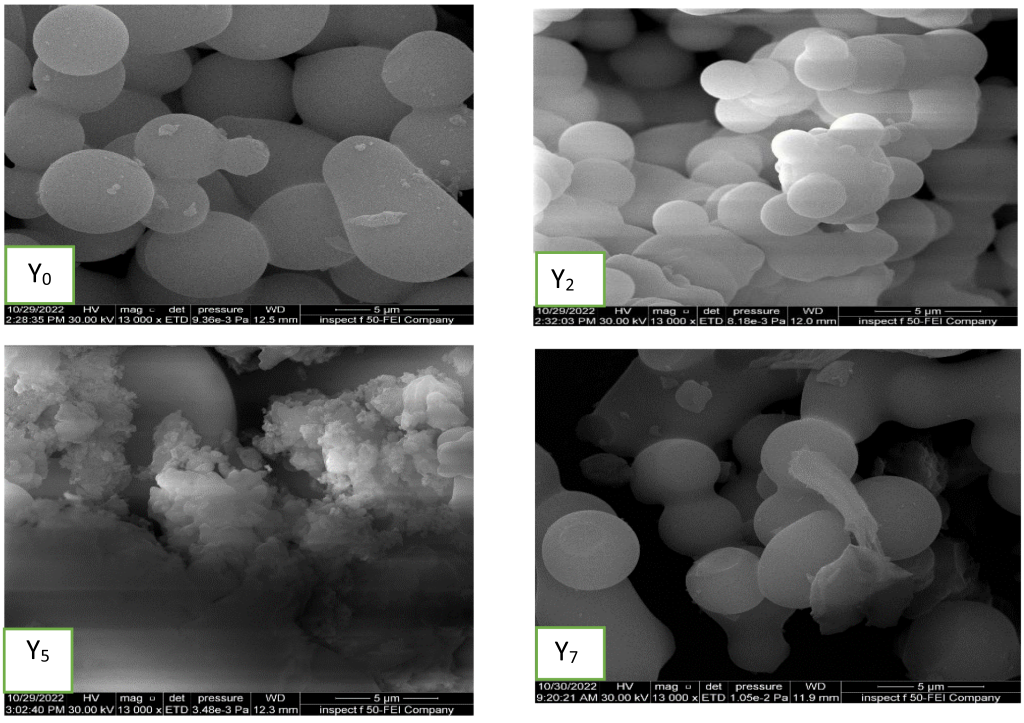

Fig. 5. FESEM of prepared SBA-15 Y_0, Y_2, Y_5, and Y_7.

Conclusion

Mesoporous SBA_15 materials with nano particle size (MSN) was effectively prepared by hydrothermal technique using 1–3 g of either PVA, or PVP, or SDS dispersant agents. Based on the findings about textural qualities, it is evident that using three different dispersion agents had a discernible effect on the surface area with a little effect on pore volume, while the pore size of samples did not surpass a magnitude of 2.5 nanometers. This behavior is happened because of two folds; firstly, the role of these dispersant agents in increasing the hydrolysis reactions between PEO hydrophilic chains of p123 micelles, and inorganic silica ions without dehydration reactions to hydrophobic core of P123 micelles. Secondly all the experiments attempted at fixed thermal conditions (100 °C, 24 hrs.), which may be not enough to encourage the hydrophobic chain reactions inside p123 micelles producing samples with high surface area, and low porosity.

By comparing the effect of adding the two polymers (PVA, and PVP), it was concluded that the effect of PVP on dispersion of SBA-15 is more pronounced than that of PVA leads to prevent aggregation, and maximum dispersion of particles reached to 31 nm particle size, this is duo to the optimum length of vinyl chains in PVP structure in comparison with long vinyl chains in PVA structure, which facilitating aggregation of particles.

On the other hand, adding 1 g of (SDS) dispersant agent increasing hydration reactions of PEO chains decreasing the aggregation degree, this phenomenon is happened duo to the repulsive action between SDS, and inorganic silica ions, which have the negative charge, while adding or than 1 g of SDS causes opposite effects, which may be happened duo to reduce the thickness of the molecular walls and resulting in the formation of aggregates. The optimum texture properties of 871 cm^2/g, and 0.43 cm^3/g were obtained using 2 g of PVP-k30 dispersion agent.

Future scope of work will be focused on studying the effect of varying aging conditions (Temperature, and time) on hydration-dehydration reactions between p123 micelles, and silica ions of sodium meta silicate, besides using of different swilling agents to produce SBA-15 with highly mesoporous properties.

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images that are not ours have been included with the necessary permission for republication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- Authors sign on ethical consideration's approval.
- Ethical Clearance: The project was approved by the local ethical committee at University of technology.

Authors' contribution statements

Y. S. K. : Drafting the manuscript, acquisition of data analysis, H. Q. H. : Drafting the manuscript, revision and proofreading.

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تصنيع وتشخيص العامل المساعد (SBA-15) النانوي باستخدام انواع مختلفة من العوامل المشتتة

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المستخلص

تم صناعة العامل المساعد (SBA_15) ذو الشكل الشبه كروي وذو مساحة سطحية وقطر فتحات ممتازة باستخدام الطريقةالحرارية بنجاح . تم استخدام سيلكات الصوديوم المصنعة من رمل السيليكا من الرمال العراقية كمصدر للسيلكا في التصنيع. تم أستخدام البوليمر نوع (P123) كقالب بدرجة حامضية عالية للوسط المائي وصلت الى اقل من 1. تم دراسة تاثير أضافة ثلاثة انواع من العوامل المشتتة على الخواص الفيزيائية للعامل المساعد (SBA-15) المصنع وهي بولي فينايل الكحول (PVA) و بولي فينايل بايرولودين (PVP-k30) و سلفات دوديسيل الصوديوم (SDS) وبنسب وزنية (1-3) غرام .تم تشخيص خواص العامل المساعد بعد أجراء عدة فحوصات مثلXRD, FTIR, BET, FESEM, AFM فحص الXRD بين وجود منحنى رئيسي وحيد في المحور السيني بين 20-25 ثيتا وهذا يدل على نقاوة المادة المصنعة كما بين فحص ال FTIR وجود مجموعة السينالون ومجموعة السيلوكسان و مجموعة السينالول. بينت نتائج فحوصات ال BET و ال AFM أن تاثير العامل المشتتة أنحصر فقط على تشتيت دقائق السليكا والحصول على مساحات سطحية عالية نسبيا مقارنة بالعامل المساعد المصنع بدون اي اضافات حيث ان معدل قطر الدقائق المصنعة كان ضمن حدود النانو مسببة مساحات سطحية تباينت بين 349 الى 871 سم2/g . كما تبين أنه لايوجد تاثير للعوامل المشتتة على حجم الفجوات واقطارها حيث تباينت بين 0,17-0,43 سم3/ غرام و قطر فجوات تراوح بين 2-2.5 نانومتر فقط. النتائج المثالية سجلت باستخدام 2 غرام من PVP-K30 وبشكل دقائق شبة كروي يتحول الى غير منتظم الشكل عندما تزداد اضافة اي عامل من العوامل المشتتة عن 1 غرام.

الكلمات المفتاحية: SBA_15، سيليكات الصوديوم، عوامل مشتتة، بوليفينايل بايروليدون، سلفات دوديسيل الصوديوم.