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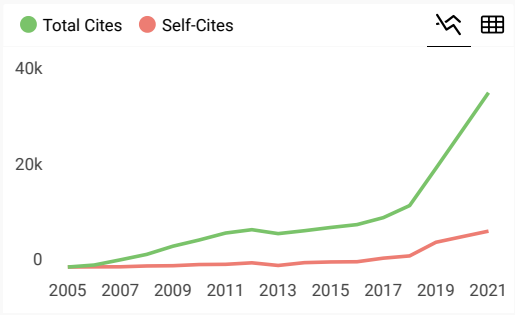

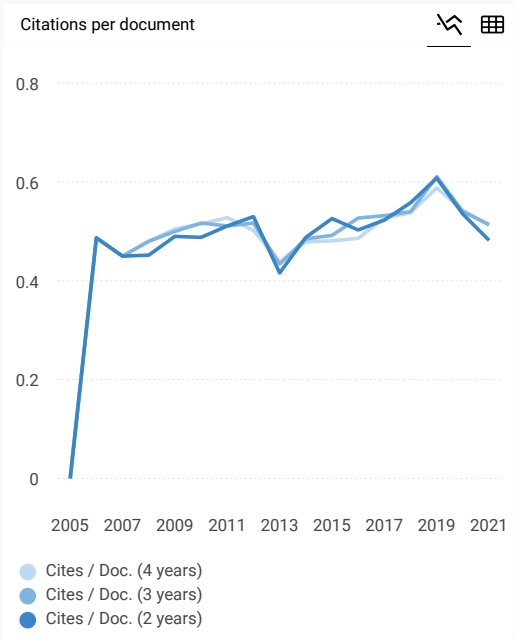

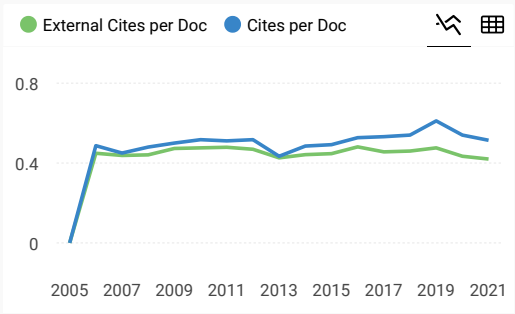

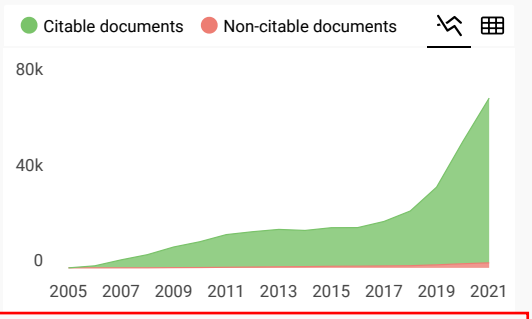

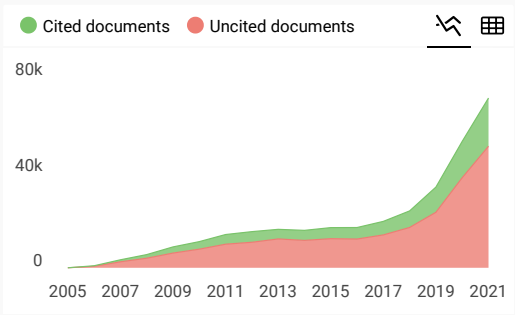

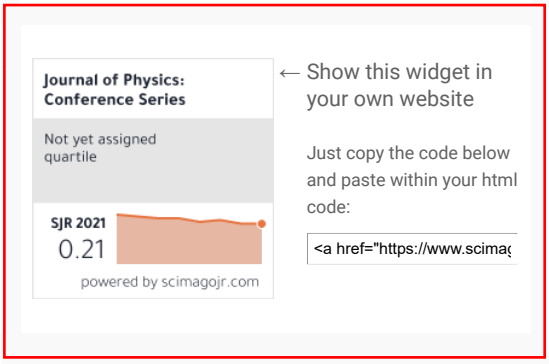

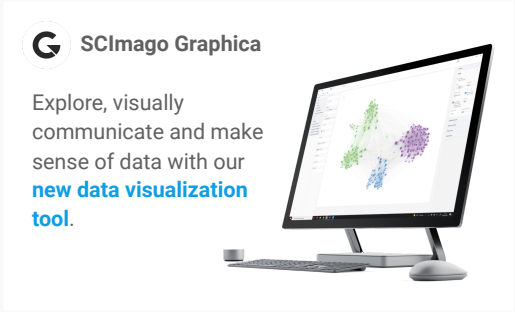

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Characteristics of MSNs synthesized by structure directing method

Characteristics of MSNs synthesized by structure directing method

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Abstract. The high specific surface area (SSA) mesoporous silica nanoparticles (MSNs) are synthesized and characterized in this work. The structure directing method combined with the co-condensation and bi-phasic processes are employed to fabricate the porous particles. Myristyltrimethylammonium bromide (MTAB) is utilized as the major structure directing agent. Triton X-100 and triethanolamine (TEA) are employed as the co-structure directing agents. In order to generate a bi-phasic environment and increase the reaction rate, cyclohexane (CHX) and L-Arginine (LAG) are used, respectively. To swelling of the structure directing agents, the solution of structure directing agents, solvent and catalyst is subjected to sonicate by ultrasonic wave with the frequency of 40 kHz. In the research, as synthesized sample of particles are characterized by different scientific techniques. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are utilized to analyze the morphology and pore formed on the obtained particles. The N_2 adsorption-desorption isotherm testing integrated with the BET and BJH calculation techniques are employed to determine the SSA, mean pore diameter and total pore volume of the resultant particles at relative pressure of 0.99. Results from the tests as mentioned revealed that SSA of particles synthesized by using only MTAB as a structure directing agent, is about 672.30 m^2/g while SSA of particles fabricated by using MTAB+TEA and MTAB+triton X-100 is 691.45 and 651.93 m^2/g, respectively. A mean pore diameter and total pore volume of the obtained particles are 4.9, 7.5, 3.9 nm and 0.83, 1.30, 0.63 cm^3/g when MTAB, MTAB+TEA and MTAB+X-100 are used as structure directing agents, respectively. Porous particles with uniform size distribution are successfully synthesized and confirmed by SEM and TEM micrographs, respectively. Finally, TEM-EDX confirmed that the synthesized particles are silica (SiO_2) nanoparticles.

1. Introduction

One of the most important materials that many research groups around the world pay attention to research and develop are mesoporous silica nanoparticles (MSNs). This because they have high potential to apply in different fields such as drug delivery, absorbent, high performance composite membrane preparation for purification of water and biofuels and others. To synthesis of MSNs, many of approaches are introduced. One of the most important synthesis methods is co-condensation combined with biphasic condition and using of structure directing agents [1]. Using this synthesis method has resulted a high monodisperse and high surface area MSNs that is suitable for application in different fields such as drug delivery, enhancement of membrane performance and environmental problem mitigation. This research attempted to synthesize the monodisperse MSNs with high specific surface area (SSA) by using the

structure directing method. Different types of surfactant that is used as the structure directing agent or template were employed. Co-condensation method combined with water-CHX biphasic condition was utilized in this research. To the best of our knowledge, myristyltrimethylammonium bromide (MTAB) has not been used as the structure directing agent with Triton X-100 and TEA for the synthesis of porous silica nanoparticles. The synthesis method used for the preparation of MSNs in this research is a facile method that one who non-specialist can synthesize.

2. Materials and methods

Tetraethylorthosilicate (TEOS, $C_8H_{20}H_4Si$, M_W: 208.33 g/mol, density of 0.932-0.934 g/ml at 20 °C) was supplied by Merck. A main structure directing agent, myristyltrimethylammonium bromide (MTAB, $C_{17}H_{38}BrN$, MW: 336.39 g/mol, 98.0% purity) was purchased from Sigma-Aldrich while the co-structure directing agents, triethanolamine (TEA, $N(CH_2CH_2OH)3$, M_W: 149.19 g/mol) and triton X-100 (Extra pure) were supplied by Kemaus (Autralia) and Loba Chemie (India), respectively. Ethanol (EtOH; C_2H_5OH, M_W: 46.07 g/mol, 99.9%) and cyclohexane with purity of 99.5% (CHX, C_6H_{12}, M_W: 84.16 g/mol) were purchased from RCI Labscan. L-Arginine (LAG, $C_6H_{14}N4O_2$, M_W: 174.20 g/mol) that is used as catalyst, was supplied by Himedia (India). Deionized water (DI-water, 18.2 MΩ) was produced by a Sartorius H_2OPRO-DI-T Arium Pro DI Ultrapure Water System. All of chemicals were used without further treatment.

Co-condensation method that is modified from Stöber process [2] combined with water-CHX biphasic condition [1] was utilized for the synthesis of silica nanoparticles. For the step of synthesis, main structure directing agent; MTAB, and co-structure directing agents including TEA and Triton X-100 and catalyst; LAG, were dissolved in DI-water under stirring and then CHX was slowly poured into the solution to create a second phase on the structure directing agent, catalyst and DI-water mixtures. About 15-20 min after the addition of CHX, TEOS was added dropwise and continued stirring for 20 h. Synthesized particles were collected by centrifugation and washed three times with ethanol-DI-water solution. Afterwards, as-synthesized particles were washed again with pure water and dried in electric oven at 80 °C overnight. The process of the synthesis was shown in figure 1.

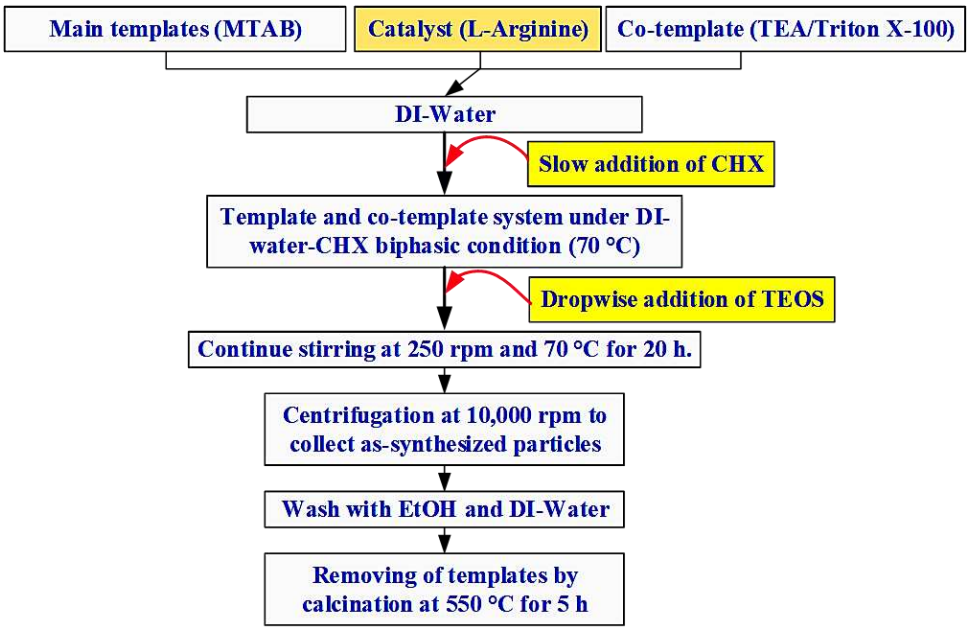

Figure 1. Steps of MSNs synthesis by co-condensation and bi-phasic methods.

Samples of the resultant particle were characterized by several scientific techniques. SEM and TEM were employed to study the external and internal morphology and porous structures appeared on the

particles. Specific surface area, pore size and pore volume were determined by the Brunauer-Emmett-Teller (BET) Surface Area Analysis and Barrett-Joyner-Halenda (BJH) through the resulted N_2 absorption-desorption isotherm, respectively. Elemental analysis was also conducted by TEM-EDX to confirm the element type of the resultant materials.

3. Result and discussion

As shown in figure 2, two types of co-template (TEA and Triton X-100) used in this research clearly affect the external morphological structure of synthesized MSNs particles. Smallest and biggest particle sizes were obtained when TEA and Triton X-100 are used as co-template, respectively. This because TEA act as a growth inhibitor for mesoporous particles [3]. Additionally, SEM and TEM micrographs showed that the size of all resultant particles are quite uniform. They also showed that spherical shape nanoparticles with porous structures were synthesized. This recent research confirmed that silica particle size can be adjusted by changing of co-structure directing agent types. In addition to the type of co-structure directing agent or co-template, concentration of co-template in the reaction solution also affect the particle size of the resulting MSNs [3].

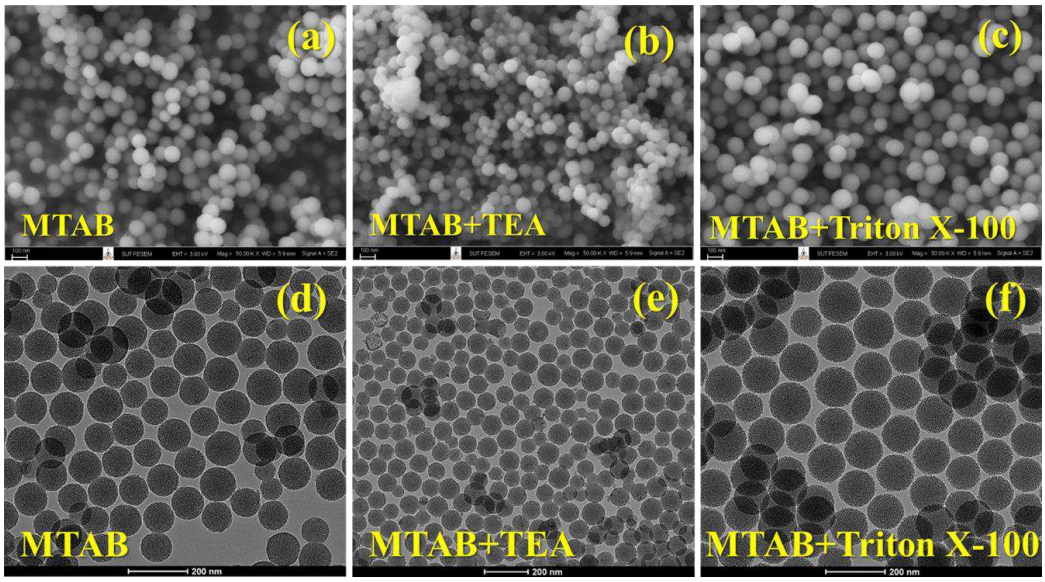

Figure 2. (SEM) External morphology of synthesized MSNs using, (a) MTAB, (b) MTAB+TEA, (c) MTAB+Triton X-100, as structure directing agents and (TEM) porous structure of synthesized MSNs using, (d) MTAB, (e) MTAB+TEA and (f) MTAB+Triton X-100, as structure directing agents.

Porous silica nanoparticles were completely synthesized. Profile of N_2 adsorption-desorption isotherm as illustrated in figure 3 shows clearly pore characteristics on the particles and corresponds to the type IV adsorption-desorption isotherm published by IUPAC [4]. Type IV adsorption-desorption isotherm shows micro/meso-porous on the particles. On the basis of pore diameter on the synthesized porous materials, IUPAC has classified the pore size into three categories. Pores with pore diameters less than 2 nm and greater than 50 nm are called micropores and macropores, respectively. Pores with pore diameters between 2 nm and 50 nm are called mesopores. Materials with pore diameters between 2 nm and 50 nm are called mesoporous materials [4]. SSA of the synthesized MSNs was about 691.45 and 651.93 m^2/g when TEA and Triton X-100 are used as co-structure directing agents, respectively. While SSA of MSNs that is synthesized by no use of co-structure directing agent, was about 672.30 m^2/g. Total pore volume (p/p_0 = 0.990) and mean pore diameter of synthesized MSNs/MTAB, MSNs/MTAB+TEA and MSNs/MTAB+Triton X-100 were about 0.8301, 1.2945, 0.6321 cm^3/g and 4.94, 7.49, 3.88 nm, respectively. Pore size distribution ($d_{p,peak}$) which is analyzed by Belsorp

Adsorption/Desorption Data Analysis Software (Ver. 6.1.0.4, BEL, Japan, Inc.,), of MSNs/MTAB, MSNs/MTAB+TEA and MSNs/MTAB+Triton X-100 was about 2.43 nm.

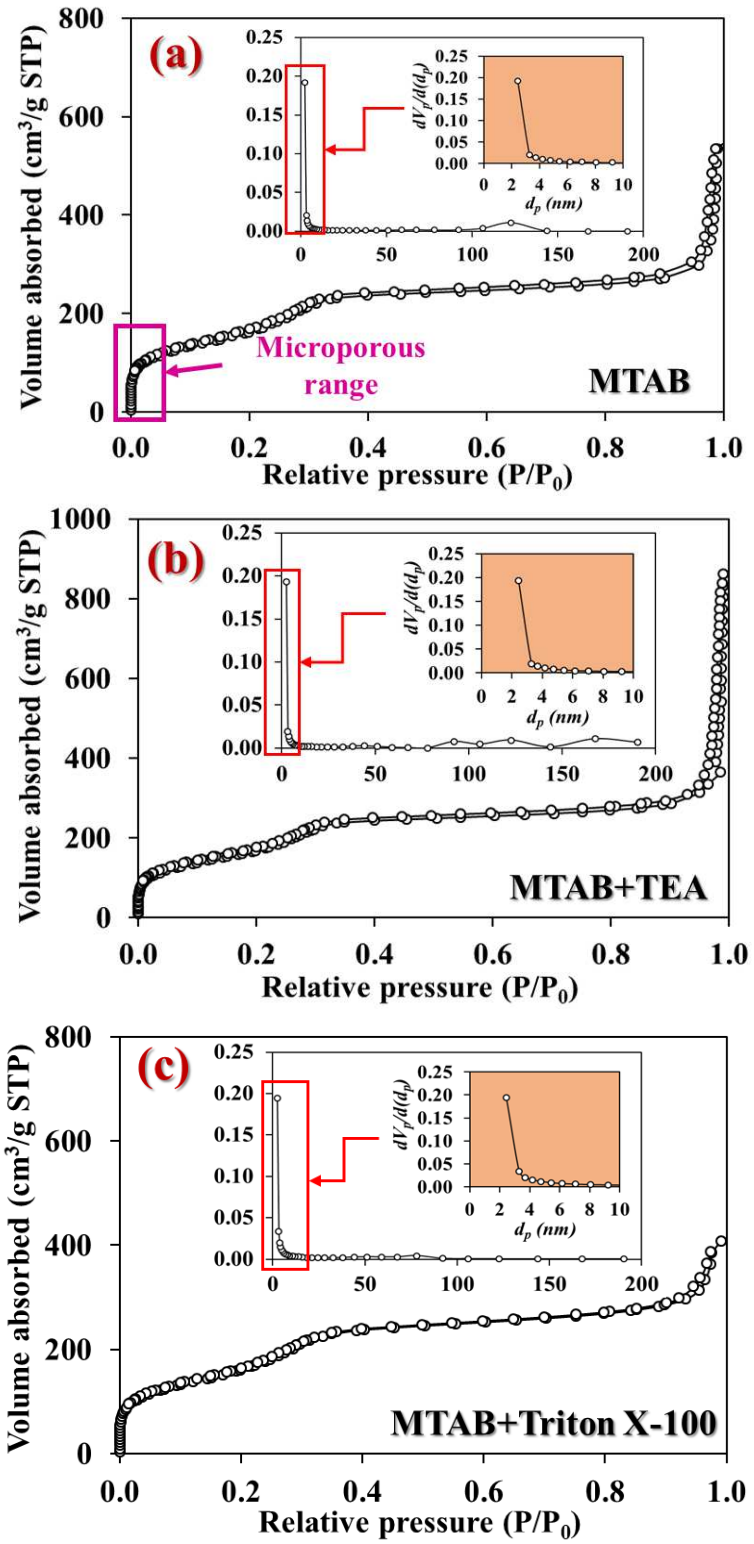

Figure 3. N_2 adsorption-desorption isotherms and pore size distribution of particles using, (a) MTAB, (b) MTAB+TEA and (c) MTAB+Triton X-100 as structure directing agents.

Effect from the size of particle, pore volume and pore size distribution as well as micro-pore and meso-pore filling (as shown in figure 3) [5] lead to the highest and lowest SSA of MSNs/MTAB+TEA and MSNs/MTAB+Triton X-100, respectively. Silica nanoparticles with mesoporous structure and spherical shape is one of the high potential materials to apply for different fields. In this research work, elemental analysis is conducted by TEM-EDX (FEI™ Tecnai™ G² 20). The presence of silica (SiO_2) is confirmed as show in figure 4.

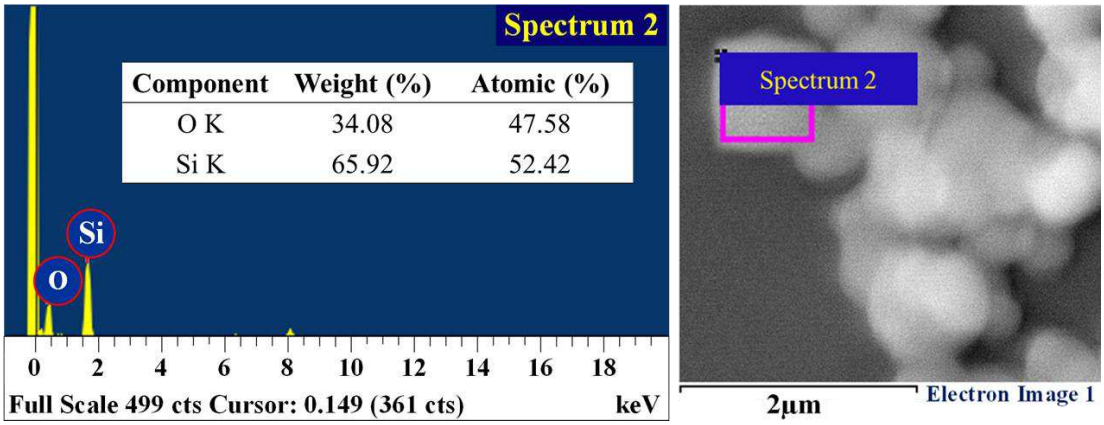

Figure 4. The result of EDX measurement of SiO_2 samples.

4. Conclusion

In summary, mesoporous silica nanoparticles (MSNs) or micro/meso-porous silica nanoparticles (MMSNs) with quite high specific surface area were completely synthesized by co-condensation method combined with a bi-phasic condition and structure directing technique. Co-template types affect the morphological structure, specific surface area, pore volume and particle size of synthesized MSNs. Specific surface area of MSNs decrease and increase when Triton X-100 and TEA are utilized as the co-structure directing agent, respectively. The results also show that specific surface area decrease with increase of MSNs particle size.

Acknowledgements

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