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Synthesis, Characterizations, and Recent Applications of the Silica-based Mobil Composition of Mesoporous Material: A Review

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Abstract

Silica-based mesoporous materials are a class of porous materials with unique characteristics such as ordered pore structure, large surface area, and large pore volume. This review covers the different types of porous material (zeolite and mesoporous) and the physical properties of mesoporous materials that make them valuable in industry. Mesoporous materials can be divided into two groups: silica-based mesoporous materials and non-silica-based mesoporous materials. The most well-known family of silica-based mesoporous materials is the Mesoporous Molecular Sieves family, which attracts attention because of its beneficial properties. The family includes three members that are differentiated based on their pore arrangement. In this review, the major applications of the Mobil Mesoporous Molecular Sieves family, such as catalysts, adsorbents, and drug delivery agents, have been surveyed. Furthermore, the synthesis of the Mesoporous Molecular Sieves materials, the silica sources, the importance of templates, and the mechanisms of the synthesis are discussed herein. Members of this material family are characterized by many physicochemical properties that are closely related to their high silica content, crystalline structure, and pore arrangement. Commonly, the members of this family have large surface areas, high pore volumes, small pore sizes, and narrow and uniform particle size distributions. These properties enable numerous industrial applications and opportunities for scientific studies to further develop existing materials or manufacture new ones.

Keywords: M41S, molecular sieves, surfactant, catalyst, adsorbent, drug delivery agents.

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1- Introduction

Nanomaterials contain at least one structural component on the nanoscale (from 1 to 100 nm) endowing them with many unique properties and enabling a wide range of applications [1]. Porous materials are categorized by the International Union of Pure and Applied Chemistry into three main types: microporous materials that have pore dimensions less than 2 nm, mesoporous materials that have pore diameters between 2 and 50 nm, and macroporous materials that have pore diameters higher than 50 nm [2]. The Mesoporous Molecular Sieves (M41S), Santa Barbara Amorphous (SBA) materials, Michigan State University materials (MSU), Folded Sheets Mesoporous materials (FSM), Korea Advanced Institute of Science and Technology materials (KIT), and Fudan University (FDU) materials are the most common forms of porous silica used in various industries [1, 3]. The discovery of natural zeolites, which are crystalline microporous aluminosilicates marks the beginning of advanced porous materials research [4]. Zeolites are widely used in petroleum refineries and chemical and petrochemical industries [5] because they have major economic benefits and are environmentally friendly [1, 6-14]. New zeolites with layered structures (MCM-22, MCM-36, MCM-49, and MCM-56) have medium pore sizes and various physicochemical characteristics [15]. Layered zeolites refer to a class of promising molecular-sieve catalysts that have unique morphologies, microporous structures, and physicochemical properties [16]. Zeolites have pores that are smaller than 2 nm in diameter, which improves many industrial reactions by increasing the surface area, but the pores are too small to hold a wide range of larger molecules. Diffusion restrictions brought on by smaller pores also have an impact on how well zeolite systems work in various applications [17- 29].

Mesoporous materials have applications in adsorption, separation, catalysis, drug delivery, sensors, spectroscopy for energy storage, and nanodevices working with larger molecules. They are particularly advantageous because of their ordered structure, large surface areas, and high pore volumes [1]. Mesoporous materials are generally divided into two types: silica-based and non-silica-based mesoporous materials [4]. Silica-based mesoporous materials consist of a honeycomb-like silica structure with

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*Corresponding Author: Name: Ammar S. Abbas, Email: ammarabbas@coeng.uobaghdad.edu.iq many empty channels, and non-silica-based mesoporous materials mainly consist of mesoporous metal oxides or different types of metals [4].

Over the years, different materials have been investigated for various uses, but silicon-based mesoporous materials are of particular importance and are promising materials for several applications, such as environmental remediation. This is attributed to their high surface areas and well-defined pore sizes. Additionally, these materials can interact with ions, atoms, and nanoparticles [30].

M41S was discovered in the early 1990s by Kuroda and co-workers and scientists at Mobil Oil Company, marking a milestone in materials chemistry [31]. Mobil scientists utilized long-chain organic surfactant molecules instead of using small organic molecules as a templet during the synthesis of highly ordered mesoporous material [31].

M41S members are toxicologically safe, chemically and thermally stable, mechanically strong, water-soluble, and have high active site concentrations on their surfaces [30, 32]. Another characteristic of such materials is the presence of silanol (Si-OH) groups on the surface [33]. Members of the M41S family attract significant interest because of several distinctive qualities like; uniform and tunable particle size and morphology, high surface area, high pore volume, and uniform distribution of pore size [34], making M41S materials suitable for numerous applications [35]. The active sites of the M41S family members can be modified by adding organic groups, coordination compounds, and nanoparticles. The functionalization broadens the applications of these materials in various fields [36]. The functionalization of these materials is mainly facilitated by the high reactivity and concentration of silanol groups on the surfaces of mesoporous material [30].

M41S originated from the series of materials known as Mobil Composition of Matter (MCM), which includes various members that differ in pore arrangement and structure, namely MCM No.41 (MCM-41), MCM No.48 (MCM-48) [37] and MCM No.50 (MCM-50) [38]. Their crystallographic definition is based on the arrangement of pores, which is generally complex, ranging from one-dimensional to three-dimensional pore structures. MCM-41has a two-dimensional pore structure (hexagonal structure, space group p6mm), MCM-48 has a three-dimensional pores structure (cubic structure, space group Ia3d), and MCM-50 has a one-dimensional pore structure (lamellar structure, space group p2) [39]. Fig. 1 shows the pore structures for the members of the M41S family.

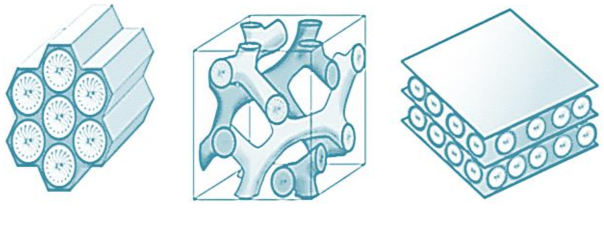
(a) (b) (c)

Fig. 1. Structures of M41S Members: a) MCM-41, b) MCM-48, and c) MCM-50

2- Syntheses of M41S

The characteristics of the M41S members are not only based on the configuration of their pores but also depend on their microcosmic morphology and macroscopic structure [40]. The synthesis approaches used to form inorganic mesoporous materials are based on organic surfactant molecules and inorganic silica sources [4].

The M41S members (MCM-41, MCM-48, and MCM-50) can be synthesized using different silica sources, surfactant types, and operating conditions, including hydrothermal treatment synthesis setups (Teflon autoclaves, reflux setups, and microwaves), temperatures, and reaction times.

The different silica sources include tetraethyl orthosilicate (TEOS) and other simple alkoxides that do not contain any siloxane bonds [41- 42], and colloidal silica [43- 44]. The first synthesis of MCM-41 was achieved using TEOS as a silica source [45]. Other silica-source developed in the synthesis of M41S members include ethyl silicate [46], fumed silica [47- 48], silica gel and sodium silicate [49- 50], silica sol [48], bagasse fly ash [51], iron ore tailing [52] and rice husk ash [53- 55]. Notably, the silica source is one of the costliest factors. Currently, there is a strong effort to identify cost-efficient and reproducible synthesis procedures with high yields [56].

Furthermore, the synthesis relies on the use of organic surfactant molecules that function as templates or structure-directing agents (SDA), through which the source of inorganic silica can condense [4]. The surfactant chemistry and concentration play significant roles in the process [56].

Ultimately, the synthesis of M41S members can be controlled by the type of surfactant, the interactions between the silica source and template molecules, and the particular synthesis mechanism [57]. Different alkyl chain lengths can be used as surfactants such as octadecyltrimethylammonium bromide and cetyltrimethylammonium bromide [58]. The size of the pores, the thickness of the pore wall, and the geometry of the M41S members are all controlled by the surfactant; therefore, the choice of surfactant is critical [59].

The template is necessary for inorganic materials to nucleate and build. When the template structure is removed, its geometric characteristics are replicated in the inorganic materials [60]. Three steps are required for this technique to create a mesoporous solid shape: first, is the surfactant self-assembly, second is the organization of an inorganic precursor over this surfactant and the formation of a stable inorganic-organic hybrid, and last is the removal of the organic template to obtain the mesoporous inorganic solid [4].

Surfactants are amphiphilic molecules, which are soluble in organic solvents and water. These molecules have hydrophobic tails and hydrophilic heads, and they can arrange themselves such that the hydrophilic head group interacts with the water and the hydrophobic chain will move above the interface (the interface may be nonpolar liquid or air) [61]. The cohesive energy of the surface is held up because of the presence of amphiphilic surfactant molecules, subsequently lowering the surface tension. The aggregates of surfactant molecules in the solvent have different shapes and sizes depending on the composition and concentration of the surfactants [4].

3- Mechanism of M41S Synthesizing

The M41S synthesis depends on the self-assembled micelles ($SAM_{S)}$ resulting from the electrostatic interactions between the surfactant head group and the inorganic precursor [62]. SAM_S formed between the SDA and the silica source can lead to liquid-crystal phases with hexagonal, cubic, or laminar arrangements [63]. The members of the M41S family can be prepared under hydrothermal conditions. Hydrothermal methods are effective for the synthesis of mesoporous molecular sieves generating products with higher hydrothermal stability, enhanced mesoscopic regularity, and improved pore size [64]. The synthesis of the M41S family is based on the combination of sol-gel, and surfactant-based approaches. Organic-inorganic hybrid materials are often prepared using sol-gel methods [65]. This process involves the preparation of a colloidal suspension, followed by gelation of the liquid suspension, and then posttreatment, to ultimately create an inorganic network or solid oxide phase [66].

M41S materials are produced by the liquid crystal template (LCT) technique. The Mobil research group proposed the LCT mechanism, in which supramolecular assemblies of surfactant micelles direct the creation of the mesophase [67]. The liquid crystalline mesophases or micelles act as templates, rather than individual single molecules or ions [68]. The cooperative self-assembling (CSA) mechanism and true liquid crystal template (TLCT) mechanism are two different pathways in the fabrication of M41S materials [4].

In the CSA mechanism, the liquid solution contains surfactant molecules and an inorganic silica source. Micelles are formed in solution, surrounded by inorganic silica species. Then, the micelles tend to arrange themselves in a specific geometry, and precipitated particles separate from the liquid solution [69]. The cooperative assembly is driven by weak noncovalent bonds such as: hydrogen bonds, van der Waals forces, and electrovalent bonds. When the template is removed, the inorganic silica species polymerize and condense [70]. The CSA mechanism occurs when the concentration of surfactant is lower than the concentrations at which lyotropic liquid crystals form [71]. The pathway for the formation of mesoporous by using templet shows in Fig. 2.

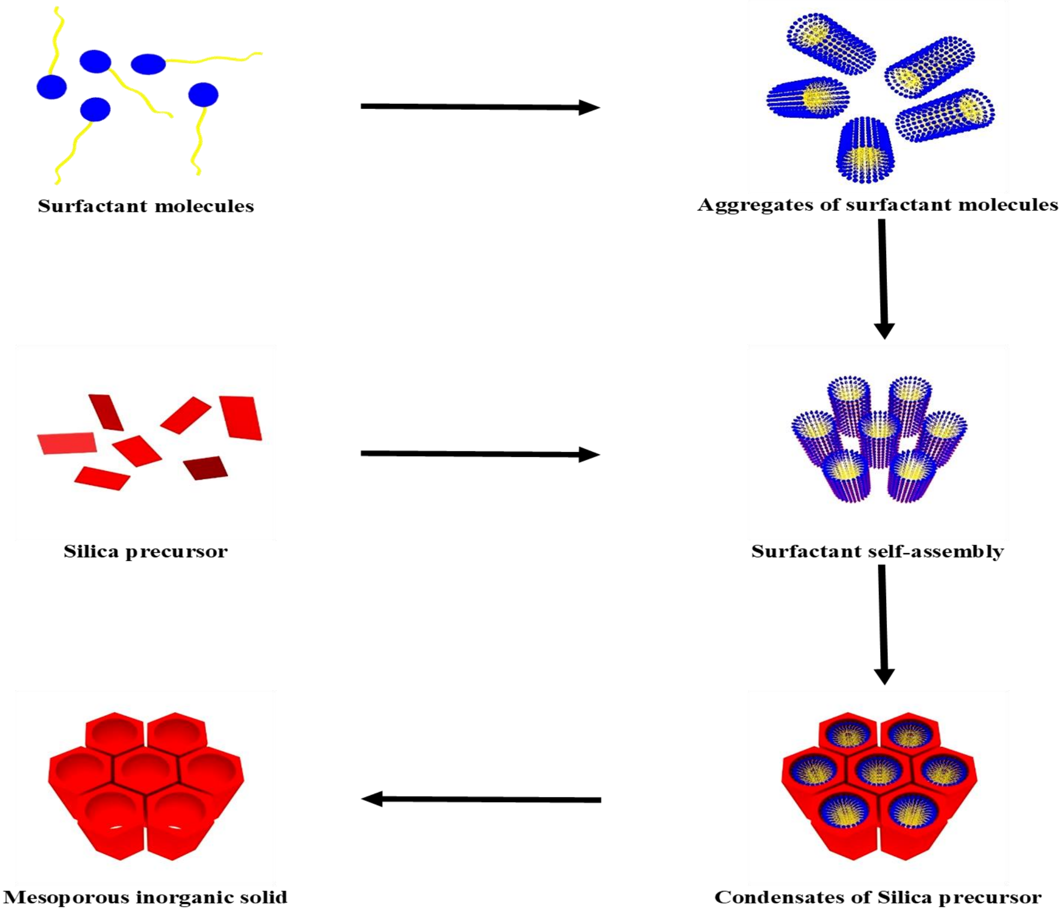

Fig. 2. Pathway for Formation of M41S Family

In the TLCT mechanism, the SAMs and liquid crystal phases form first because the surfactant concentration is relatively high, without the presence of the inorganic silica species. Then, the silica framework polymerizes around the specific geometric arrangement of the template. Finally, the mesostructured silica material is obtained after removing the template [72].

The hydrothermal sol-gel process for the synthesis of M41S materials consists of many steps [4]. Initially, surfactant self-assembly occurs according to the LCT mechanism to form a homogeneous surfactant solution, which is usually aqueous. Then, a silicate precursor or inorganic sodium silicate is added to the surfactant solution which hydrolyzes under basic conditions to form a silicate oligomer solution. Next, these oligomers condense with the surfactant micelles and aggregate to form an inorganic-organic hybrid, which eventually precipitates in the form of a gel [59]. The gel is treated hydrothermally for further condensation, solidification, and reorganization of the material into an ordered arrangement. The ordered mesostructured silica material is obtained when the template is removed by calcination. Finally, the resultant product is cooled, filtered, washed, and dried [4].

In 1992, the Mobil research group presented the synthesis procedure, characterization, and formation mechanism of MCM-41 which has a hexagonal pore arrangement. The authors stressed that microscopy and diffraction results obtained for MCM-41 are strikingly similar to those obtained for surfactant/water liquid crystals or micellar phases [67]. The researchers used sodium silicate, tetramethylammonium silicate, and TEOS as the sources of silica. The quaternary ammonium surfactant compounds were cetrimonium chloride and cetrimonium bromide, which are cationic surfactants. In addition to the hexagonal MCM-41 class, the Mobil research group has synthesized other materials with different geometries by varying the molar ratio of surfactant to silicon. For instance, when the directing agent/silicon ratio is less than 1, the predominant structure is the hexagonal phase. As the ratio increases beyond 1, a cubic arrangement with space group Ia3d can be obtained, known as MCM-48 [45]. For ratios greater than 1.2 and less than 2, MCM-50 can be synthesized [30].

4- Methods for Characterization of the M41S

M41S materials are often characterized by X-ray diffraction (XRD), X-Ray fluorescence spectrometry (XRF), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), and nitrogen (N_2) adsorption and desorption data [30].

XRD is commonly used to identify crystallographic structures, crystallite size, and crystallinity. Although XRD provides direct information concerning the pore architecture [73], the patterns for M41S materials only exhibit peaks in the low-angle range. The ordering lies in the pore structure, and the low-angle diffraction peaks can be indexed according to different lattices. [74]. XRF is used to determine the oxide content in materials, revealing that SiO_2 is the main compound in members of the M41S family, as well as trace amounts of Al_2O_3, P_2O_5, and K_2O_3 [75- 76]. The FESEM images provide information on the morphology of the silica-based mesoporous arrays [30]. MCM-48 consists of fine spherical particles that are approximately similar in diameter, which is similar to MCM-41, whereas the MCM-50 particles are irregular in shape and have a tendency to aggregate [43, 47]. TEM is a powerful tool for visualizing the pore orderings with greater resolution [74]. For example, pores are arranged on the cubic plane for MCM-48 [77], whereas MCM-41 has a hexagonal pore shape is observed for MCM-41 [78]. AFM can be used to study the topography of the materials, the members of the M41S family have narrow and uniform particle size distribution [79]. The N_2 adsorption and desorption data provide more insight into the textural features, such as surface area (S_{BET}), pore size (D_P), and pore volume (V_P), which are generally calculated using the Brunauer–Emmett–Teller and Barrett–Joyner–Halenda methods [80]. Table 1 shows the ranges for different properties of the M41S members.

Table 1. Characterized Properties of M41S Members

M41S member	Pore symmetry	Location of maximum intensity, degree (°)	Silica Content, wt.%	S_{BET}, m^2g^{-1}	V_P, cm^3g^{-1}	D_P, nm	References
MCM-41	**Hexagonal**	2.2- 2.6	94.37- 95.62	730-1082	0.45-1.35	2.20-3.80	[41, 49, 53, 81-83]
MCM-48	**Cubic**	2- 5	> 99	669-1586	0.33-2.58	2.05-4.02	[34, 37, 47, 54, 84- 85]
MCM-50	**Lamellar**	2.1- 2.5	–	–	–	–	[47, 86]

5- Applications of M41S Materials

The M41S family of mesoporous materials are promising candidates for catalysis because they are relatively non-toxic, non-corrosive, non-air sensitive, highly reusable, completely pollution-free, and environmentally benign supports for catalytic transformation in the liquid phase [87]. MCM-41, MCM-48, and MCM-50 have high surface areas, narrow pore size distributions, and efficient adsorption capacity, which facilitate mass diffusion and transport for higher catalytic yields. Furthermore, the textural and chemical properties of their surfaces are adjustable, allowing for the optimization of the product selectivity for target molecules. Moreover, they exhibit useful optical inertia, thermal and mechanical stability, and stabilization of metal complexes, which is necessary to prevent unwanted reactions that can reduce their catalytic activities [88]. Table 2 lists several examples of M41S members used as catalysts for different reactions.

Table 2. Examples of M41S Members used as Catalysts and Support Catalysts for Different Reactions

Catalyst	Promoter	Chemical reaction	Reference
MCM-41	–	Cracking of polyethylene	[89]
MCM-41	–	Esterification of glycerol with acetic acid	[81]
MCM-48	–	Degradation of linear low-density polyethylene	[90]
MCM-48	–	Esterification of acetic acid and ethanol	[91]
MCM-41	Co-Mo	Hydrodesulfurization of light cycle oil	[92]
MCM-41	Dialkylsilane groups	Esterification of glycerol with fatty acids	[93]
MCM-41	Nickel	Hydrocracking of coker wax oil	[41]
MCM-41	$CuCl_2$	Oxy-carbonylation of methanol	[94]
MCM-48	$CuCl_2$	Oxy-carbonylation of methanol	[94]
MCM-48	Sulfonic acid	Alkylation of toluene with benzyl alcohol	[95]
MCM-48	Alumino-silicate	Sulfur Removal from tyre-derived oil	[76]
MCM-48	Fe_2O_3	Esterification of acetic acid and ethanol	[91]
MCM-48	Pd	Hydrogenation of olefin	[96]
MCM-48	Tungstophosphoric acid	Esterification of palmitic acid and cetyl alcohol	[97]

Silica-based mesoporous materials can also be used as a strong support matrix for metals or metal oxides in catalytic applications [98]. Catalyst supports help to increase the efficiency of the supported metals or metal oxides by acting as the catalytically active center [99]. M41S members offer particularly high thermal stability in catalyst synthesis. Their catalytic function is achieved by the incorporation of active sites in the silica walls or the deposition of active species on the inner surface of the material. The advantages of using ordered mesoporous solids as catalyst supports are their relatively large pores and high surface areas, facilitating mass transfer and providing a high concentration of active sites [81]. Finally, the supported catalysts show high activity, a low degree of metal leaching, and ease in recycling when assembled on silica-based mesoporous materials [99].

Adsorption is also recognized as one of the useful applications of M41S materials because of their effectiveness and flexibility [100]. Furthermore, the M41S family has received considerable attention because of their high surface area, slightly higher pore diameters compared with the diameters of the adsorbate, and effective performance as adsorbents [73]. Moreover, their adsorption capacity can be enhanced by modifying their surface chemistry using various functional groups [101]. MCM-41 composites with imprinted nickel ions through co-condensation were used as mesoporous adsorbents to remove Ni^{+2} cations from wastewater, with high recovery. The adsorption rate and capacity were up 95% [100]. In addition, MCM-41 and MCM-48 expanded with N-N dimethyldodecylamine can effectively remove the cations Cd^{+2}, Co^{+2}, Cu^{+2}, and Pb^{+2} in an aqueous solution [102]. Regarding dye molecules, the chemical stability and low degradability of their aromatic structures are considered the major challenges for removing them from the environment [73]. However, MCM-48 has been used to remove Congo red anionic dye, and the optimum adsorption time was 25 minutes [103].

As additives, the M41S materials can improve the tribological, chemical, and thermomechanical properties of polymer materials. The high porosity of the mesoporous silica allows it to host a high amount of polymer chains [104]. Vinyl-functionalized MCM-48 has been polymerized with polystyrene, and the resulting polymer exhibited enhanced mechanical properties compared with the original polystyrene [105]. Ethylene polymerization with the addition of MCM-4, can improve the tensile strength and elongation at break by 28.3% compared with pure polyethylene [106].

Drug delivery systems are the most recent adaptation of the M41S family. An efficient delivery system should transport the desired drug molecules to the targeted cells or tissues and release the drug in a controlled manner [107]. M41S are applied as delivery agents because they have tunable particle size and morphology, uniform and tunable pore size, high surface area and pore volume, facile surface functionalization, stable physicochemical properties, and thermal stability [108]. Moreover, some mesoporous silica materials are characterized by biodegradability and biocompatibility [109]. Cytometry assays revealed that synthesized silica has no cytotoxicity against human peripheral blood mononuclear cells. Accordingly, the drug-loaded nanostructures can be applied via different routes, such as wound dressing [110]. Specifically, MCM-48 has been applied as a drug delivery agent, and the release kinetics of ibuprofen have been examined [111].

6- Conclusion

M41S are remarkable because of their uniform and tunable particle size and morphology, high surface area, high pore volume, and uniform pore size distribution. Additionally, these substances possess characteristics such as being non-toxic, chemically and thermally resistant, mechanically strong, soluble in water and having a high concentration of active sites on their surfaces. M41S differ in pore arrangement and structure which have been named MCM-41, MCM48, and MCM-50. These different members of the M41S family can be produced using different silica sources, surfactants, and synthesis conditions.

Essentially, the synthesis and control of mesoporous materials depend on the surfactant type, synthesis method, and interaction of the silica source with the template molecules. Organic surfactant molecules can act as templates or SDA, around which the inorganic silica precursor can condense. Furthermore, the self-assembly between the SDA and the silica precursors can lead to a liquid-crystal phase with a hexagonal, cubic, or laminar arrangement. M41S materials are produced by the LCT technique, which includes two different pathways such as the CSA and TLCT mechanisms.

Multiple methods have been used to characterize M41S materials, such as XRD, XRF, FESEM, TEM, AFM, and N_2 adsorption-desorption data. The M41S members have diffraction peaks in the low-angle range, their main chemical compound is SiO_2 (> 94 wt.%), and they mainly consist of spherical particles. The pore arrangement is hexagonal for MCM-41, cubic for MCM-48, and lamellar for MCM-50. Generally, these materials have a large surface area (reaching 1586 m^2g^{-1}), high pore volume (1.35-2.58 m^3g^{-1}), and small pore size (2.20-4.05 nm), with a narrow and uniform particle size distribution. MCM-41 and MCM-48 can be used as catalysts, support catalysts, water treatment agents, and drug delivery agents. In contrast, MCM-50 has attracted less attention and has not been used in applications. The surface activity of mesoporous materials can be enhanced by the functionalization of these materials with organic or inorganic functional groups, which are used in different applications such as catalysts or adsorption processes.

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التوليف والتوصيف والتطبيقات الحديثة لتكوين موبيل القائم على السيليكا لعائلة للمواد المسامية: مراجعة

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الخلاصة

المواد المسامية القائمة على السيليكا هي فئة من المواد المسامية ذات الخصائص الفريدة و التي تمتاز ببنية مسامية مرتبة، و مساحة سطحية و حجم مسامي كبيران. تغطي هذه المراجعة الأنواع المختلفة من المواد المسامية (الزيوليت و الميسوبوروس) و تستعرض الخصائص الفيزيائية التي تجعلها ذات قيمة في الصناعة. يمكن تقسيم المواد المسامية إلى مجموعتين: المواد المسامية القائمة على السيليكا و المواد المسامية غير القائمة على السيليكا. تعتبر عائلة المناخل الجزيئية ميسوبوروس هي أشهر عائلة من المواد التي تحتوي على السيليكا، و التي جذبت الانتباه بسبب خصائصها المفيدة. تضم العائلة ثلاثة أعضاء متميزين بترتيب المسام لديهم. في هذه المراجعة، تم استعراض التطبيقات الرئيسية عائلة المناخل الجزيئية المسامية موبيل ، مثل المحفزات و الممتزات وعوامل إيصال الأدوية. علاوة على ذلك، تمت مناقشة عمليات توليف مواد المناخل الجزيئية ميسوبوروس و مصادر السيليكا و أهمية القوالب المستخدمة و آليات التوليف. يتميز أعضاء هذه العائلة بالعديد من الخصائص الفيزيائية و الكيميائية التي ترتبط ارتباطًا وثيقًا بمحتواها العالي من السيليكا، و بنيتها البلورية، و ترتيب المسام فيها. بشكل عام، يمتلك أفراد هذه العائلة مساحات سطحية كبيرة، و أحجام مسامية عالية، و قياس مسام صغيرة، و توزيعات ضيقة و موحدة لحجم الجسيمات. تتيح كل هذه الخصائص لافراد هذه العائلة الاستخدام في العديد من التطبيقات الصناعية و تفتح الباب على مصرعيها لمزيد من التطبيقات و الفرص لدراسات علمية أكثر لمواصلة تطوير المواد الموجودة أو تصنيع مواد جديدة.

الكلمات الدالة: M41S، المناخل الجزيئية، مخفضات التوتر السطحي، المحفز، الممتزات، عوامل إيصال الأدوية.