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Investigation of the Morphology of the SBA-15 Mesoporous Silica as Catalytic Support for Hydrodesulfurization Catalysts

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Abstract

SBA-15 is an interesting mesoporous silica material having highly ordered nanopores and a large surface area, which is widely employed as catalyst supports, absorbents, drug delivery materials, etc. Since it has a lack of functionality, heteroatoms and organic functional groups have been incorporated by direct or post-synthesis methods in order to modify their functionality. The aim of this article is to review the state-of-the-art related to the use of SBA-15-based mesoporous systems as supports for hydrodesulfurization (HDS) catalysts.

Keywords: SBA-15, mesoporous, silica, catalyst support, hydrodesulfurization

Introduction

Nowadays, in the petroleum refining industry, the deep desulfurization of "more dirty" feeds containing refractory S-containing compounds, such as 4,6-dimethyl dibenzothiophene (4,6-DMDBT), is a priority task, due to an increasing demand for ultralow S-containing fuels imposed by more strict environmental regulations [1]. Since it is impossible to achieve deep hydrodesulfurization (HDS) of fuels using classical Co(Ni)Mo(W)/Al2O3 sulfide catalysts [2], it is urgent to develop new catalysts with higher activities, greater selectivity and better resistance to H2S and N-poisoning than those that are being used currently.

The origin of the almost exclusive use of alumina as support has been ascribed to its outstanding textural and mechanical properties and its relatively low cost [3]. However, the presence of undesirable strong metal-support interactions in the alumina-supported catalysts has triggered research devoted to the development of new supports for HDS applications [4,5,6,7,8,9,10,11,12,13]. In this sense, the use of ordered mesoporous siliceous molecular sieves as supports has been intensively investigated [5,13,14].

Ordered mesoporous silicas were first reported in 1992 [15]. Since then, significant progress has been made in their morphology control, pore size adjustment, composition variation and application developments [16,17,18]. During the last two decades, various mesoporous structures have been synthesized, which can be roughly classified into three categories based on the pore types: nearly spherical cage, cylindrical channel and bi-continuous channel [19]. Among different ordered mesoporous silicas, SBA-type silicas are the most frequently studied [13 ,14,20,21,22,23,24,25,26,27,28,29,30,31,32,33,34,3 5,36,37,38,39,40,41,42,43,44,45,46,47,48,49,50,51, 52,53,54,55,56,57,58,59,60,61,62,63,64,65,66,67,6 8,69,70,71,72,73,74,75,76,77,78,79,80,81,82]. SBA-15 silica (SBA = Santa Barbara Amorphous) exhibits interesting textural properties, such as large specific surface areas (above 1000 m2·g−1), uniform-sized pores (in range 4–30 nm), thick framework walls, small crystallite size of primary particles and complementary textural porosity. The advantage of the use of SBA-15 material as support includes also its high surface-to-volume ratio, variable framework compositions and high thermal stability [20,21,22].

Figure 1a,b shows two high resolution transmission electron microscopy (HRTEM) micrographs of our laboratory-synthetized SBA-15 mesoporous silica calcined at 550 °C. As seen in this figure, SBA-15 shows hexagonal pores in a 2D array with long 1D

channels (p6mm plane group) [21]. The channels are interconnected by small micropores. Thus, SBA-15 exhibits mainly mesoporous structure and possesses a small amount of micropores. The large pore size of this mesoporous material can mitigate the diffusion barrier for the reactants and the products. However, pure siliceous SBA-15 has an electronically neutral framework and lacks Brønsted acidity. This problem could be circumvented by SBA-15 modification in order to make this mesoporous substrate more versatile in terms of its possible applications, either as a structural material or support, in absorption processes, separation, catalysis, etc.; or, as reviewed in this case, as support of catalysts used in hydrodesulfurization (HDS) reactions in petroleum refining processes.

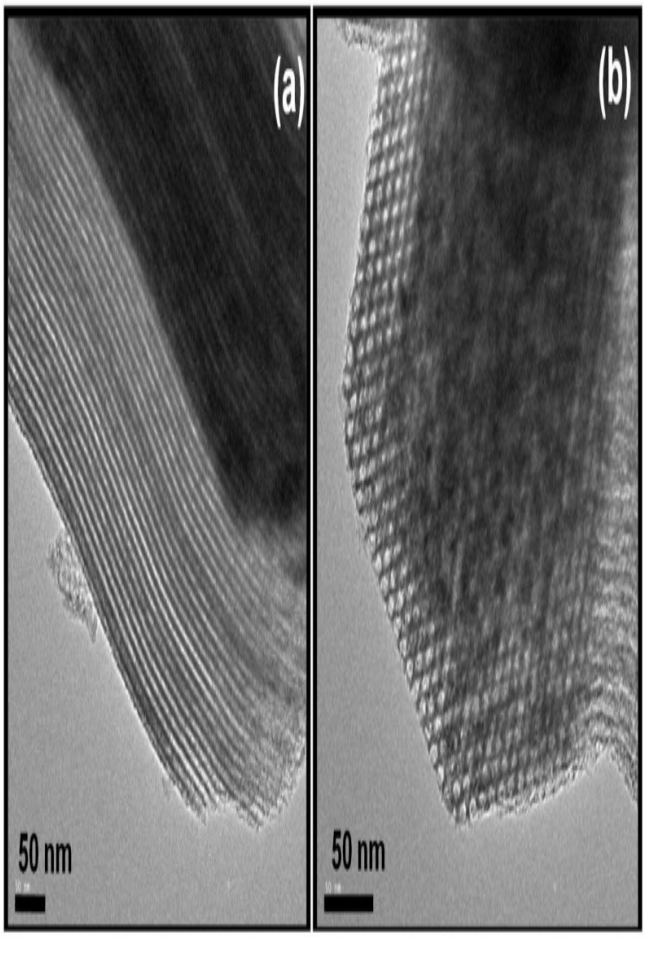

Figure 1: High resolution transmission electron microscopy (HRTEM) micrographs of SBA-15 mesoporous silica. The size and morphology of the highly ordered hexagonal pores in a 2D array (a) with long 1D channels (b) (p6mm plane group) can be observed.

There are many approaches to prepare better SBA-15-supported catalysts, such as changing the support properties by substitution of Si4+ by different cations, functionalization with different groups, etc., changing the active phase component, varying the preparation method, etc. In general, the studies in this field aim to get relationships between different physical and chemical properties of the support and active phases and catalyst performance for hydrotreating reactions, such as hydrodesulfurization (HDS), hydrodenitrogenation (HDN), hydrodeoxygenation (HDO) and/or hydrodearomatization (HDA). Recent revision by Rahmat et al. [22] on the SBA-15-based catalysts focused on their application in biorefinery production. In this review, we are going to show the possibilities of the use of SBA-15 silica as support for hydrodesulfurization catalysts.

Influence of Support

Synthesis of Bare SBA-15

The synthesis of SBA-15 molecular sieve requires the use of triblock copolymer (typically non-ionic triblock copolymer) as a structure directing agent and tetramethyl orthosilicate (TMOS), tetraethyl orthosilicate (TEOS) or tetrapropyl orthosilicate (TPOS) as a silica source [22,23,24,25,26]. In a typical synthesis, the structure directing agent (e.g., Pluronic P123: EO20PO70EO20 from BASF) is dissolved under stirring in a solution of water and 2 M HCl. After this, the required amount of tetraethyl orthosilicate (TEOS) is added at 35 °C. Then, this aqueous solution of triblock copolymer and TEOS is kept under stirring conditions for 20 h for aging. At this stage of preparation, the control of pH is of paramount importance, because the formation of ordered hexagonal SBA-15 with uniform pores up to 30 nm might only occur in strong acidic media, i.e., pH ≈ 1 [23,24,25]. In the case when the pH of a solution will be higher than that of the isoelectric of silica, i.e., at pH 2–6, no precipitation or formation of silica gel occurs. The formation of disordered or amorphous silica was observed for the synthesis carried out at neutral pH (≈7) [25].

Zhao et al. [23,24,25] reported the synthesis of a variety of mesoporous SBA-type silicas using non-ionic triblock copolymers as template. This type of surfactant is very interesting, because it is easily separated, is nontoxic, biodegradable and inexpensive [23]. The synthesis conducted with these surfactants usually occurs in low-pH solutions (pH ≈ 2), where the interaction occurs through an S0H+X–I+ mechanism (S0H+ being the surfactant hydrogen bonded to a hydronium ion, X– the chloride ion and I+ the protonated silica) [24]. The mixture is subsequently aged at 80 °C overnight. Then, the solid obtained is filtered, washed thoroughly with deionized water, dried first in air at room temperature and then calcination is carried out by slowly increasing temperature from room temperature to 500 °C in 8 h and heating at 500 °C for 6 h. The latter step of the template removal is one of the crucial aspects in the synthesis of ordered mesoporous, because the

procedure employed during calcination influenced the final textural properties of SBA-15 material. According to Zhao et al. [23], the calcination at 500 °C led to formation of SBA-15 with interlattice d spacing of 74.5–320 Å between the (100) planes, pore volume up to 0.85 m3·g–1 and silica wall thickness of 31–64 Å.

The effect of the synthesis conditions on the textural and structural properties of SBA-15 materials was studied by Klimova and co-workers [26] by using a statistical model built from a full 23 factorial design at two levels. Textural and structural differences induced by change in the synthesis conditions (temperature of the reaction of gel formation, as well as temperature and time of the aging stage) were discussed in terms of the mechanism of SBA-15 formation in the presence of Pluronic P123. The statistical analysis showed that both synthesis and aging temperatures had a significant influence on the textural and structural properties of SBA-15 materials. Their increase affected in a positive way the Brunauer-Emmett-Teller (BET) surface area, total pore volume, pore diameter and unit-cell parameter, producing, simultaneously, a decrease of micropore area and pore wall thickness. As compared to aging temperature, it was found that the gel aging time is of much lower importance, with the exception of micropore area, which continued decreasing with the increase of aging time [26].

Modulation of Pore Diameter

The control of the support's pore diameter is of paramount importance for SBA-15-based hydrotreating catalysts, which has diffusion limitations of large feed molecules to enter into unidirectional channels of the SBA-15. To study the effect of different pore diameters, the SBA-15-supported catalysts having pore diameters in the range 5–20 nm were screened for hydrotreating of heavy gas oil [27,28,29].

In this direction, Boahene et al. [27] tested FeW/SBA-15 sulfide catalysts with pore diameters in the range 5–20 nm as potential hydrotreating catalysts for hydrotreatment of heavy gas oil. The highly ordered siliceous SBA-15 substrates with different pore diameters were synthesized using hexane as a micelle expander under acidic conditions. It was found that the catalyst with a pore diameter of 10 nm was the best among the FeW/SBA-15 catalysts studied, probably due to the sufficient mass transfer of the reactants through the catalyst's pores, while maintaining a high surface area necessary for metal dispersion [27].

The effect of support pore diameter was reported also for NiMo/SBA-15 [28] and NiMo/Al-SBA-15 [29] sulfide catalysts tested in the hydrotreating of gas oil. A series of binary NiW catalysts supported on SBA-15 with different pore sizes were prepared by Lei et al. [28]. The NiW/SBA-15 sulfide catalysts with different pore sizes were tested in the hydrogenation of a heavy oil (distillation temperature: 320–340 °C) derived from the direct coal liquefaction process. It was found that the pore size of the support has a significant influence on the Ni/W crystallite size and catalytic activity, larger Ni–W crystals being formed on the supports having larger pores. As expected, the catalysts with the largest pores displayed the highest HDN and HDA activities for heavy oil upgrading [28].

For the NiMo/Al-SBA-15 catalysts, Chandra Mouli et al. [29] employed direct and post synthesis modification methods to incorporate aluminum in the framework of SBA-15. In the direct and post-synthesis approaches, the aluminum sulfate and ammonium hexafluoroaluminate were used as a source of aluminum, respectively. In the direct synthesis, the highest pore diameter was limited to 7 nm. The post-synthesis support modification with the ammonium hexafluoroaluminate led to Al-SBA-15 substrate with pore diameter greater than 10 nm. The pore structure of the synthesized SBA-15 did not collapse until 13 nm of pore diameter, as confirmed from the small angle XRD and TEM analysis. The Ni–Mo/Al-SBA-15 sulfide catalysts with different pore diameters were tested in hydrotreating of heavy gas oil carried out in a trickle bed continuous reactor [29]. It was found that HDS and HDN activities increased with the increase in pore diameter until 13 nm, and then decreased, due to the collapse in the pore structure and poor dispersion of metals on the supports, as evidenced from the BET and TEM analysis. As a consequence, the NiMo/Al-SBA-15 sample prepared by the post-synthesis method exhibited the largest HDS activity in the hydrotreating of heavy gas oil [29]. Al3+ Ion Loading SBA-15 support modification with Al3+ ions could be achieved by direct [29,30,31,32,33,34] and post-synthesis modification [35,36,37,38,39] methods. Incorporation of Al during the one-pot synthesis presents difficulties, because the high acidity (pH ≈ 1.5) needed for the creation of ordered pore structure of SBA-15 leads to leaching of aluminum and its coordination in the octahedral state. This problem can be circumvented when Al is introduced by the post-synthesis support's grafting with aluminum isopropoxide in non-aqueous solutions, anhydrous AlCl3, ammonium hexafluoroaluminate or sodium aluminate in aqueous solution.

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