



CHAPTER II

LITERATURE REVIEW

SBA-1 mesoporous molecular sieve

Huo *et al.* (1994) proposed that mesoporous cubic silica was synthesized in acidic medium via $S^+X^-I^+$ mechanism where S, X and I stand for surfactant, halide and inorganic species, respectively. Kim *et al.* (1999) also reported that higher acidic condition was favorable for the formation of SBA-1 cubic mesoporous molecular sieve. However, Tanglumlert *et al.* (2007) synthesized SBA-1 cubic mesoporous silica via the sol-gel process using silatrane precursor prepared from fumed silica and triethanolamine (TEA), and found that SBA-1 was synthesized at room temperature using dodecyltrimethyl ammonium bromide (C_nTAB , $n = 14, 16$ and 18) as cationic surfactant template under dilute acidic solution. They also found that the shape of SBA-1 was dependent of alkyl chain length of surfactant. XRD and TEM results show the three-dimensional cubic structure characteristics.

Metal incorporated SBA-1

Vanadosilicate is known to be an excellence hydrocarbon oxidation catalyst using hydrogen peroxide as an oxidant. V-SBA-1 incorporating more than 4 wt% vanadium ($Si/V < 20$) was synthesized by Dai *et al.* (2001) via direct synthesis under strongly acidic condition using tetraethyl orthosilicate [$Si(OC_2H_5)_4$, TEOS] and ammonium vanadium (NH_4VO_3) as silica and vanadium sources, respectively. By using X-ray diffraction (XRD), it was found that structure of V-SBA-1 was less order than that of pure silica SBA-1. N_2 adsorption/desorption measurement showed larger mesopores with diameter of around 40 Å, compared with that of 20 Å for pure siliceous, indicating that V was incorporated onto SBA-1 frame work. UV-vis and raman results revealed that the incorporated vanadium species had tetrahedral coordination and presented in a high dispersion.

Che *et al.* (2001) synthesized a series of Mo-SBA-1 mesoporous molecular sieve with various H_2O/HCl and Si/Mo molar ratios at $0^\circ C$ by direct synthesis method using TEOS as a silica source and ammonium heptamolybdate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$, AHM) as a molybdenum source. For comparison, another

Mo-SBA-1 sample was prepared by impregnating the pure silica SBA-1 with an aqueous solution of AHM. Diffuse reflectance UV-visible and laser Raman spectroscopy results showed that in the absence of water, Mo species consisted of tetrahedrally and octahedrally coordinated monomers and polymers. These results also showed that in the presence of moisture, tetrahedral Mo centers were partially converted to octahedral Mo centers for directly synthesized Mo-SBA-1 and completely converted for impregnated Mo/SBA-1 samples. Che *et al.* concluded that the impregnated samples were better dispersed than the directly synthesized samples.

Dai *et al.* (2001) also synthesized Mo-SBA-1 using the direct synthesis method under acidic condition using TEOS and AHM as silica and molybdenum sources, respectively. Another method to synthesize is impregnation of Mo onto pure siliceous SBA-1. Both synthesized Mo-SBA-1 materials were used as catalyst for partial oxidation of methane with oxygen as an oxidant. The catalytic performance of the directly synthesized Mo-SBA-1 was better than that of Mo-impregnated SBA-1. It was also found that Mo content affected the partial oxidation of methane.

Ti-incorporated SBA-1 mesoporous molecular sieves were directly synthesized under strongly acidic condition by Ji *et al.* (2003) using CTEABr as the template. XRD and SEM results showed well-order cubic structure of synthesized mesoporous materials. UV-visible results revealed that titanium species existed in high dispersion and had tetrahedron coordination. Ji *et al.* (2003) also tested its catalytic performance by epoxidation of styrene as a probe reaction, and found that Ti in SBA-1 framework possessed relatively high activity and selectivity, as compared with one dimensional mesoporous of Ti-MCM-41 and Ti-SBA-15.

Cr-SBA-1 was also synthesized using the direct synthesis method under acidic condition. TEOS and ammonium dichromate ($(\text{NH}_4)_2\text{CrO}_7$) were used as silica and chromium sources, respectively. From XRD pattern, synthesized Cr-SBA-1 showed a well order cubic structure and large mesopores. It also showed that higher amount of chromium incorporated in the mesoporous materials might reduce the structural order. Cr-SBA-1 containing up to 3.75 wt% Cr was formed while still maintaining a fairly well-ordered cubic structure. FT-IR and diffuse reflectance UV-visible results showed that both monochromate and polychromate coexisted on the SBA-1 mesoporous materials. Cr-SBA-1 sample prepared by the direct synthesis

method was used as catalyst for hydrogenation of ethane with carbon dioxide, its catalytic activity was lower than that of Cr-SBA-1 prepared by conventional impregnation. (Zhao *et al.*, 2007).