CHAPTER I
INTRODUCTION

Ordered mesoporous materials, having pore size range between 2–50 nm, exhibit highly ordered pore structure, huge surface area as well as changeable pore size. These properties lead to many applications, such as selective sorption, liquid-phase separation, and effective catalysis. Thus, numerous researchers have paid much attention to develop methods to synthesize those materials to improve their properties.

SBA-15 is one of the well-known ordered materials which belongs to the 2-D hexagonal mesoporous silica with *p6mm* space group. It attracts many scientists due to its large pore size (46–300 Å), thick wall (36–64 Å) (Zhao *et al.*,1998), and high thermal and hydrothermal stability (Zhao *et al.*, 1998).

Generally, SBA-15 has been synthesized in the presence of triblock copolymer surfactant, viz. (poly(ethylene oxide)-poly(propylene oxide)–poly(ethylene oxide); PEO_{20}-PPO_{70}-PEO_{20}), as a structure-directing agent, and tetraethyl orthosilicate (TEOS) as a silica source via sol-gel process in acidic condition. To obtain the highly-ordered structure material, the thermal assistance were used either by conventional autocave method (Zhao *et al.*, 1998) or mocrowave-assisted approch (Newalkar *et al.*, 2000). In both ways, the reaction took long time (11–72 h in autocave at about 100 °C or 2 h in microwave at about 120 °C after stirring at room temperature for about 24 h), and certainly used much energy. The novel-room temperature route was discovered by Samran *et al.* in 2008, they succeeded to prepare SBA-15 at room temperature using a moisture-stable silatrane as a silica source. Surprisingly, using this precursor as a silica source, the well-ordered SBA-15 could be obtained just by stirring at room temperature for 24 h without any heat treatment.

A moisture-stable silatrane is an organosilicate compound discovered by Wongkasemjit *et al.* in 1999. It was synthesized directly from silicon dioxide (SiO_2) and triethanolamine (TEA) using ethylene glycol as a solvent via the "Oxide One Pot Synthesis (OOPS)" process. As being stable toward hydrolysis reaction, it was successful to be used to synthesize many ordered-porous materials, such as SBA-1, Fe-SBA-1 and Ti-SBA-1 cubic mesoporous silica (Tanglumleart, *et al.*, 2007–2008), MCM-41 and Fe-MCM-41 hexagonal mesoporous silica (Thanabodeekij *et al.*, 2006;

Thitsartarn, *et al.*, 2007), titanium loaded TS-1 zeolite (Phonthammachai *et al.*, 2006), etc.

In term of catalytic activity, metal loaded-SBA-15 tends to have higher activity than pure silica SBA-15. For example, Mo-SBA-15 (Melero *et al.*, 2007) showed good catalytic behaviour in the epoxidation of 1-octene in the presence of *tert*-butyl hydroperoxide, Fe-SBA-15 (Zhang *et al.*, 2007) and Ti-substituted SBA-15 (Chen *et al.*, 2004) also showed better catalytic activity for styrene oxidation as well. Therefore, many scientists have developed techniques to incorporate metal into the frameworks of SBA-15, such as a physical-vapor-infiltration, a wet-impregnation, and a sol-gel process. Among them, direct synthesis via the sol-gel method has been effectively used by many groups. Wongkasemjit and coworkers (1999–2009) have successfully synthesized various types of mesoporous materials using this method, for example, Fe-SBA-1 and Ti-SBA-1 cubic mesoporous silica (Tanglumleart, *et. al.*, 2008–2009), MCM-41 and Fe-MCM-41 hexagonal mesoporous silica (Thanabodeekij *et al.*, 2006; Thitsartarn, *et. al.*, 2007), etc.

The objective of this work was to synthesize SBA-15, Fe-SBA-15, Mo-SBA-15 and Ti-SBA-15 with various contents of metals (Fe, Mo and Ti) by a novel synthetic route (a sol-gel process) using silatrane as a silica source, and PEO_{20}-PPO_{70}-PEO_{20} as a template. Ferric chloride, molybdenum glycolate, and titanium glycolate were used as iron, molybdenum and titanium sources, respectively. Finally, the catalytic activity of SBA-15 and Ti-SBA-15 for styrene oxidation was investigated.