CHAPTER I
INTRODUCTION

Mesoporous materials are those with pore in the range of 20–500Å in diameter. They have huge surface areas, providing a vast number of sites where sorption process can occur. These materials have numerous applications in catalysis, separation, and many other fields (Estermann *et al.*, 1991; Hoang *et al.*, 2005; Trong-On *et al.*, 2001; Kubo *et al.*, 2007). Synthesis of these materials is of considerable interest and is constantly being developed to introduce different properties.

Since the discovery of the so-called M41S (Kresge *et al.*, 1992; Beck *et al.*, 1992) silicas, much research work has been concentrated on this new class of mesoporous materials. Originally, this family has been classified into three subgroups: a hexagonal (MCM-41), a cubic (MCM-48), and a lamellar phase (MCM-50) (Sayari *et al.*, 1996; Vinu *et al.*, 2003). Among the mesoporous silicas, SBA family has been attracted much attention because of thicker walls, better hydrothermal stability and larger pore size than M41S. It is well known that SBA-15 material is analogous to the hexagonal assemblage of cylindrical micelles in the amphiphilic surfactant. The synthesis of SBA-15 mesoporous silicas is achieved by the use of surfactant micelles as structure directing agents in a sol-gel process. Amphiphillic surfactants, poly(ethylene glycol)-block-poly(propylene glycol)-block-poly(ethylene glycol), self assemble into cylindrical micelles, which are encapsulated by an inorganic material. Calcination, a thermal processing technique is then used to remove the organic surfactant, leaving a hexagonal arrangement of mesopores (Goltner *et al.*, 1997; Wanka *et al.*, 1994; Chu *et al.*, 1996). Many researchers almost used tetraethoxysilane (TEOS) as a silica source, however, for this research work, we introduced another source of silica from metal alkoxides precursor.

During the last 9 years, Wongkasemjit and coworkers (2001–2009) synthesized moisture stable metal alkoxides, namely silatrane, alumatrane, cerium glycolate, zirconium glycolate, titanium glycolate, tin glycolate and molybdenum glycolate, directly from inexpensively corresponding metal oxide using ethylene

glycol solvent via the "Oxide One Pot Synthesis (OOPS)" process (Phiriyawirut *et al.*, 2003; Charoenpinijkarn *et al.*, 2001). The reaction gives highly pure metal alkoxides. Both synthesized silatrane and alumatrane have been successfully used as precursors for synthesis of microporous and mesoporous zeolites via sol-gel process such as, LTA (Sathupanya *et al.*, 2002), ANA, GIS (Sathupanya *et al.*, 2003), MFI (Phiriyawirut *et al.*, 2003), TS-1 (Phonthammachai *et al.*, 2003) and MCM-41, Ti-MCM-41, Mo-MCM-41 (Thanabodeekij *et al.*, 2005-2006).

In addition, the inclusion of guest molecules in mesoporous materials is one of focused subjects. Some efforts have been made to incorporate metal atoms, such as V, Mo, Cu, Fe, and Ti, into mesoporous silica materials by using silatrane as a precursor. In terms of catalytic activity, metal loaded-SBA-15 tends to have higher activity than pure silica SBA-15. For example, Cu-SBA-15 showed higher activity and selectivity for hydroxylation of phenol than the pure SBA-15 (Wang *et al.*, 2005), Ti-substituted SBA-15 (Chen *et al.*, 2004), Fe-SBA-15 (Zhang *et al.*, 2007), also showed better catalytic activity for styrene oxidation than pure SBA-15. Therefore, many scientists have developed techniques to incorporate metal into the frameworks, such as a physical-vapor-infiltration, a wet-impregnation, and a sol-gel process. Among them, direct synthesis via sol-gel method has been effectively used by many groups. Wongkasemjit and coworkers have successfully synthesized various types of mesoporous materials using this method, for example, Fe-SBA-1, Ti-SBA-1, cubic mesoporous silica (Tanglumleart *et al.*, 2008-2009), Fe-MCM-41 hexagonal mesoporous silica (Thitsartarn *et al.*, 2008), titanium loaded TS-1 zeolite (Phonthammachai *et al.*, 2006).

Thus, the major aims of this work are to study a novel synthesis route to produce the remarkably high quality SBA-15, and metal loaded SBA-15 viz. Ti-SBA-15, Fe-SBA-15 and Mo-SBA-15 by silatrane precursor and using poly(ethylene glycol)-block-poly(propylene glycol)-block-poly(ethylene glycol) as a structure directing agent. The catalytic activities of the synthesized SBA-15 and metal loaded SBA-15 are also focused in this research.