# CHAPTER I
# INTRODUCTION

According to IUPAC classification, porous materials can be categorized into three groups by their pore size. The first group is microporous materials that have pore diameter less than 2 nm, such as zeolite. The second one is mesoporous materials that have pore diameter between 2 nm to 50 nm. The last one is macroporous materials having pore diameter larger than 50 nm.

Mesoporous materials have some desirable properties, such as high surface area, large pore volume, tunable mesoporous channels with defined pore size distribution, controllable wall composition as well as modifiable surface properties, etc. They are used in many applications, such as adsorbent, catalysts, ion exchanger, energy material, environment material, etc.

SBA-15 (Santa Barbara Amorphous type) is one of highly ordered mesoporous silica materials. It exhibits superb characters, namely, high specific area (600–1000 $m^2/g$), thicker wall (2 to 6 nm), larger pore size (4 to 30 nm), better hydrothermal stability (upto 850 °C) and mechanical properties than M41s. These properties depend on the synthetic conditions. Basically, SBA-15 is synthesized by non-ionic polyethylene oxide-polypropylene oxide-polyethylene oxide triblock copolymer (Pluronic123, P123) as structure directing agent, tetraethoxysilane (TEOS) as a silica source, hydrochloric acid (HCl) as acid catalyst. In this work, home-made silatrane is used as silica precursor due to its better moisture stability than TEOS, thus slowing hydrolysis step and providing more time for mesopore formation (Samran *et al.*, 2011). However, pure SBA-15 is still lack of redox properties and acidity. Thus, incorporation of heteroatom or transition metal is necessary. There are several successful examples of heteroatoms incorporation, such as, Al, Fe, Ti, V, Mo, Pt etc. (Zhang *et al.*, 2012). However, most of these researchers emphasized on incorporation of single metal ion.

Phenol is known as the harmful substance that contaminate in the waste water which is believed to be carcinogenic. Several scientists attempted to convert these toxic into mild product, such as catechol (CAT) and hydroquinone (HQ), used various application. There are a few microporous materials that showed the high

catalytic activity toward the phenol hydroxylation but there are some downsides because of the limitation of pore size and complex preparation method. Furthermore, heteroatom such as titanium and iron incorporated on various supports has been widely used in this area due to their outstanding properties. For example, titanium has shown excellent activities for the selective oxidation of organic compounds when using hydrogen peroxide as oxidant. Moreover, iron-substituted mesoporous materials have received much attention because of its redox properties and unusual activity in alkylation and oxidation reactions when iron was substituted in the framework (Zhang *et al.*, 2008).

Presently, multi-components incorporated SBA-15 has been popular since it could create new redox and active sites, as compared with mono-heteroatom, therefore, displaying different catalytic activies. There are many ways to synthesize these products namely, grafting, post-synthetic impregnation, direct synthesis etc. However, when using post-treatment, there is partial blocking of mesopores. Thus, the aim of this work is to synthesize bimetallic Ti-Fe-SBA-15 via sol-gel process using silatrane, titanium (IV) isoproproxide, iron (III) chloride hexahydrate ($FeCl_36H_2O$), and P123 as silica, titanium, iron sources, and structure-directing agent, respectively. The products are characterized by XRD, $N_2$ adsorption/desorption isotherms, DR-UV, FT-IR, TGA, and FE-SEM. In addition, the optimal condition for catalytic activity in the phenol hydroxylation is investigated using HPLC.