CHAPTER I



INTRODUCTION

1.1 Background

There are interesting applications of interconnected macropore monolith materials in many fields, for example, column in HPLC, electrode for electric double layer capacitor (EDLC) and catalyst supports. Previously, column of HPLC is packed with fine particles. The reducing in particle sizes leading to better column efficiency is normally used but also causes an increasing in pressure drop. Therefore the monolithic column with flow – through pores is a potential candidate for column. These columns provide better stability and high performance than conventional columns packed [1]. Another application is electrode in EDLC which requires porosity materials. The high specific surface area and electric conductivity are required for double layer formation [2].

There are many types of interconnected macropore monolith materials which are made of polymer, silica and carbon. Polymer gel columns are prepared by in situ polymerization. This method is direct polymerization of monomers in column. Another material is silica which prepared by the sol – gel method. The metal alkoxide is used as the precursor and cause the hydrolysis and polycondensation reaction to form gel network. Normally, macroporosity carbon can be prepared by using templates. In preparing this carbon, silica, zeolite, stable emulsion and polymer latex are usually used for the templates. The carbon precursors are polymeric materials or the precursor of polymeric materials such as sucrose, some thermoplastics and phenolic resin.

Recently, interconnected macroscopic carbon without templates using is reported in literature [3]. The new method for preparing 3D interconnected macroporous carbon monolith is sol – gel method which use Resorcinol(R) – Formaldehyde (F) as the precursors and apply ultrasonic irradiation during gelation state. The character of this material is flow – through pores, the macropore size are narrow distributions with size around $1 - 3 \mu m$ moreover the mesopore cannot observe on the structure of 3D interconnected macroporous carbon monolith and there is a little micropore on its. The structure of 3D interconnected macroporous carbon monolith has been shown in Figure 1.1. Therefore creating of porosity on the skeleton structure is necessary because both general and specific applications require nanopores on the surface of carbon skeleton.

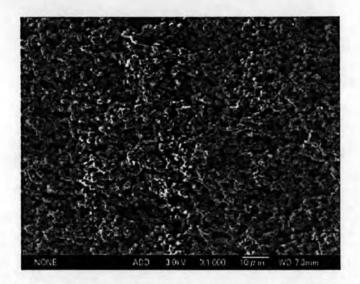


Figure 1.1 The SEM micrographs of 3D interconnected macroporous carbon monolith (cross section plane) [3]

The activation method is a well known process used to generate and increase porosity in carbon material. There are two types of activation methods, physical and chemical activation. In physical activation steam, carbon dioxide and air are used as activation agents [4, 5]. In chemical activation, there are many activation agents such as ZnCl₂, H₃PO₄, H₂SO₄, Ca(NO₃)₂, alkali hydroxide and carbonate etc. In gasification of coals, chars and carbons are widely used catalyst for enhancement reaction rate and reduction reaction temperature [6-9].

Therefore an increasing in porosity of 3D interconnected macroporous carbon monolith, the use of this material will be promoted especially in the fields of chromatography, catalyst supports and electrode in electrochemistry applications in the near future.

1.2 Objectives of study

To generate and increase micropore or mesopore on the 3D interconnected macroporous carbon monoliths (3D – IMM) by physical and chemical activation.

1.3 Scopes of research

1. Investigation effect of gas activation of 3D interconnected macroporous carbon monoliths (3D - IMM)

1.1 Effect of activation agents (CO₂ and steam)

1.2 Effect of activation patterns (one step and two step activation)

2. Investigation effect of gas activation with metals loading on 3D interconnected macroporous carbon monoliths (3D – IMM)

2.1 Effect of chemical agents in activation process (sodium, potassium, cesium and calcium)

2.2 Effect of carbon dioxide couple with calcium in activation process

2.3 Effect of activation temperature with calcium in activation process

1.4 Expected benefits

Generation of carbon structure with bi-modal (micropore – macropore or mesopore – macropore) or tri-modal (micropore – mesopore – macropore) structure will be conducted. Increasing the porosity of 3D interconnected macroporous carbon monoliths to apply in applications such as column for chromatography, catalyst support and porous electrode in electrochemistry.