CHAPTER III

CHEMICALS, EQUIPMENTS AND CHARACTERIZATIONS

An experiment in this research is carried out in a laboratory-scale for the preparation of the hierarchical porous carbon monolith without using any templates. The experimental procedures are divided into three sections. The first is the preparation of the RF monolith gel with an interconnected macroporous structure by combinations of ultrasonic irradiation and sol-gel polycondensation at the same time. The second is the formation of hierarchical porous structure in the carbon monolith by carbonization, direct thermal and direct chemical activation. And the last is the preparation of a hierarchical porous carbon monolith by inverse-phase suspension during gel formation and followed by both the direct thermal activation and the direct chemical activation. The chemical agents, equipments and characterization technique for using in this research are described in this chapter.

3.1 Chemical agents

The specifications of chemical agents for using in this research are shown in Table3.1.

Table3.1: Lists of chemical agents for using in the research

Chemical agents	Using for	Manufacturer/grade
1. Resorcinol ($C_6H_4(OH)_2$)	Synthesis of Resorcinol-formaldehyde (RF) gel	Fluka/99.8%
2. Formaldehyde (HCOH)	Synthesis of Resorcinol-formaldehyde (RF) gel	BDH/38 % w/w
3. Sodium carbonate (Na_2CO_3)	Synthesis of Resorcinol-formaldehyde (RF) gel and Impregnation into RF monolith for CO_2-activation	Ajax/analytical grade

Table3.1 (Next): Chemical agents for using in the research

Chemical agents	Using for	Manufacturer/grade
4. Deionized water	Synthesis of Resorcinol-formaldehyde (RF) gel	Production from MilliQ apparatus (Millipore, Bedford, MA).
5. Calcium nitrate tetra hydrate ($Ca(NO_3)_2.4H_2O$)	Impregnation into RF monolith for CO_2-activation	Ajax/analytical grade
6. Nitrogen (N_2)	Carbonization	TIG/purity 99.999%
7. Carbondioxide (CO_2)	Activation	TIG/purity 99.8%

3.2 Equipments

3.2.1 Ultrasonic reactor apparatus

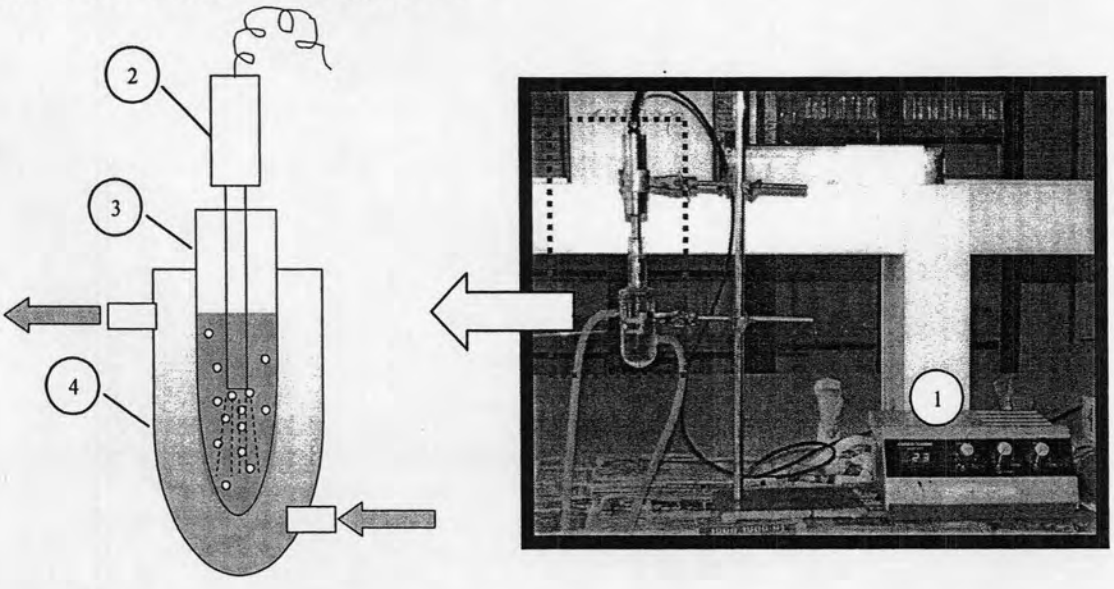

Figure 3.1: Illustration of ultrasonic reactor set for preparation of RF monolithic gel, the reactor consists with (1) ultrasonic generator, (2) Ultrasonic probe, (3) tested tube for containing RF solution and (4) water cooling jacket.

The simultaneous combination processes of sol-gel reaction and ultrasonic irradiation are carried out with the ultrasonic reactor apparatus as shown in Figure 3.1. The apparatus is assembled from three main parts marked with the numbers in Figure 3.1 as followed;

- Number (1) is the ultrasonic generator built from Sonics and Materials Inc (Model VB120). The ultrasonic generator can produce ultrasonic wave with constant frequency at 20 kHz, and can be tuned the ultrasonic power up to 30 W.
- Number (2) is the ultrasonic probe in diameter of 0.6 mm made from titanium alloy which is vertically immersed into the vessel contained with the RF solution.
- Number (3) is the tested tube in diameter of 2.5 cm as a container for the RF solution.
- Number (4) is the water cooling jacket which is made from glass container (with inlet and outlet for cooling water at the bottom and top respectively).

3.2.2 Horizontal furnace reactor

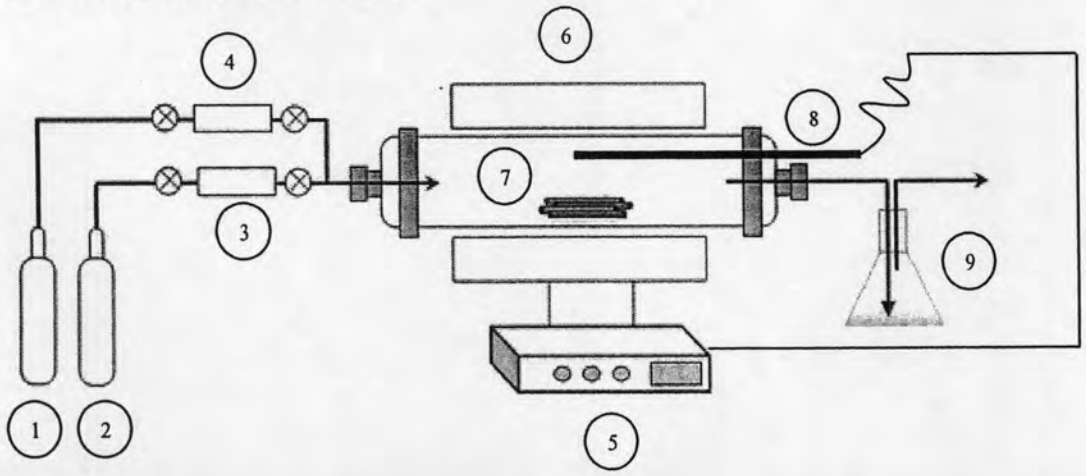

Figure 3.2: Schematic diagram of the quartz tube furnace used in this work, it is composed with (1) N_2 gas container, (2) CO_2 gas container, (3) CO_2 gas rotameter, (4) N_2 gas rotameter, (5) furnace controller, (6) furnace, (7) cylindrical quartz tube, (8) Thermocouple and (9) flask containing alcohol for residual trap.

Both carbonization and activation processes in this research are carried out with horizontal furnace reactor as shown the schematic diagram in Figure 3.2. The furnace is constructed with the main parts showing with the numbers in the Figure 3.1 as followed;

- Number (1) and (2) are the cylindrical gas containers of nitrogen 99.999% and carbon dioxide 99.8%, respectively.
- Number (3) and (4) are the gas rotameters of CO_2 and N_2 for measurement gas flow rate, respectively.
- Number (5) is the temperature controller box.
- Number (6) is the electrical furnace. It can be heated with the power output up to 2000 W.
- Number (7) is the cylindrical quartz tube in a length and diameter of 60 cm and 10 cm, respectively. It is used for holding the samples while being kept in the electrical furnace.
- Number (8) is the thermocouple K-type (maximum temperature 1200 °C).
- Number (9) is the flask contained with ethanol to trap the residue gas.

3.3 Characterizations

1. Porosity is characterized by nitrogen adsorption – desorption at – 196 °C (BEL; BELSORP – mini)

1.1 BET surface (S_{BET}) is determined by BET equation

1.2 micropore volume (V_{mic}) is calculated by t – method

1.3 mesopore volume (V_{mes}) is estimated by DH – method

2. Microstructure was characterized by SEM (Scanning Electron Microscope) (JEOL; JSM – 5800LV)

3. Interconnected macropores size distribution and volume are measured by mercury intrusion technique by Micromeritics, Pore-Sizer-9320.

4. Fourier Transform Infrared (FTIR) spectra were recorded using spectrometer (Perkin Elmer, 1615), and the samples for analysis is formed as pellets made by crushing and mixing the samples with spectroscopy grade of KBr.

5. A decrease in weight of the obtained carbon monoliths, % burn-off, was measured after carbonization and both physical and chemical activations.