

## CHAPTER IV

### EXPERIMENTAL

This chapter describes the experimental work, including the materials and apparatus as follows.

#### 4.1 SYNTHESIS OF PVA SPONGE

##### 4.1.1 CHEMICALS

The chemicals used in this experiment are of analytical grade, except polyvinyl alcohol of various grades. The specifications are as follows.

1. Polyvinyl alcohol ( $[-\text{CH}_2-\text{CH}(\text{OH})-]_n$ ) of molecular weight of about 115,000 supplied by BDH Laboratory, England.

Minimum degree of hydrolysis 87 %

Viscosity (4%aqueous solution at 20 °C) 40 cP

Ash 0.7 %

2. Polyvinyl alcohol ( $[-\text{CH}_2-\text{CH}(\text{OH})-]_n$ ) of molecular weight of about 72,000 supplied by BDH Laboratory, England.

Minimum degree of hydrolysis 98 %

Loss on drying at 110 °C for 4 hours 3 %

3. Polyvinyl alcohol ( $[-\text{CH}_2-\text{CH}(\text{OH})-]_n$ ) of molecular weight of about 14,000 supplied by Air Products and Chemical. Inc. (commercial grade)

Minimum degree of hydrolysis 99 %

Viscosity (4%aqueous solution at 20 °C) 5 cP

4. Sulfuric acid ( $\text{H}_2\text{SO}_4 = 98.07$ ) supplied by AJAX Chemicals.

Sp.gr.	1.84
Minimum assay	98.0 % w/w

5. Sodium Lauryl Sulfate / Sodium Dodecyl Sulfate ( $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$ ) supplied by AJAX Chemicals.

Minimum assay	96 % w/w
Chloride	0.01 % w/w
Heavy Metal(Pb)	0.002 % w/w

6. Formaldehyde ( $\text{CH}_2\text{O} = 30.03$ ) supplied by AJAX Chemicals.

Assay	Min 36.5 % w/w
	Max 38.0 % w/w
Colour (APHA)	Max 10

7. Polyethylene glycol average molecular weight about 200 supplied by AJAX Chemicals.

Hydroxyl number	535-590
Wt. per ml at 20° C	about 1.12 g

8. n-Hexane ( $\text{CH}_3(\text{CH}_2)_4\text{CH}_3$ ) supplied by CARLO ERBA reagenti r.p.l. MonTedison group.

Minimum assay	99% w/w
Boiling point	68.7%
Density 15° C/4° C	0.662
Refraction index at 20° C/D	1.3749±0.0030

#### **4.1.2 SYNTHESIS UNIT**

A synthesis unit for sponge consists of the following components:

1. Reaction kettle of 1000 ml capacity
2. Variable high speed stirrer required 240-2000 rpm supplied by RW20 JANKE & KUNKEL JKA LABORTECHNIK.
3. Heating mantles, 220W, 450 °C Maximum, SS2 5PS Electrothermal.
4. Thermometer

Figure 4.1 shows the assembling of the synthesis unit.

#### **4.1.3 PVA SOLUTION PREPARATION**

The hot water-soluble grade of PVA was slurried in cold water and then heated to complete solution. The following procedures were followed.

1. Required amount of water was placed in the reaction kettle at room temperature. Agitation was then started.
2. PVA resin was added into the vortex so that it rapidly wetted out and dispersed. Addition was done slowly to avoid agglomeration
3. Temperature was raised to 90°C or above and agitation was continued until the resin was completely dissolved. The heating was turned off and the kettle content was allowed to cool. Stirring was continued as PVA aqueous solution came to room temperature.

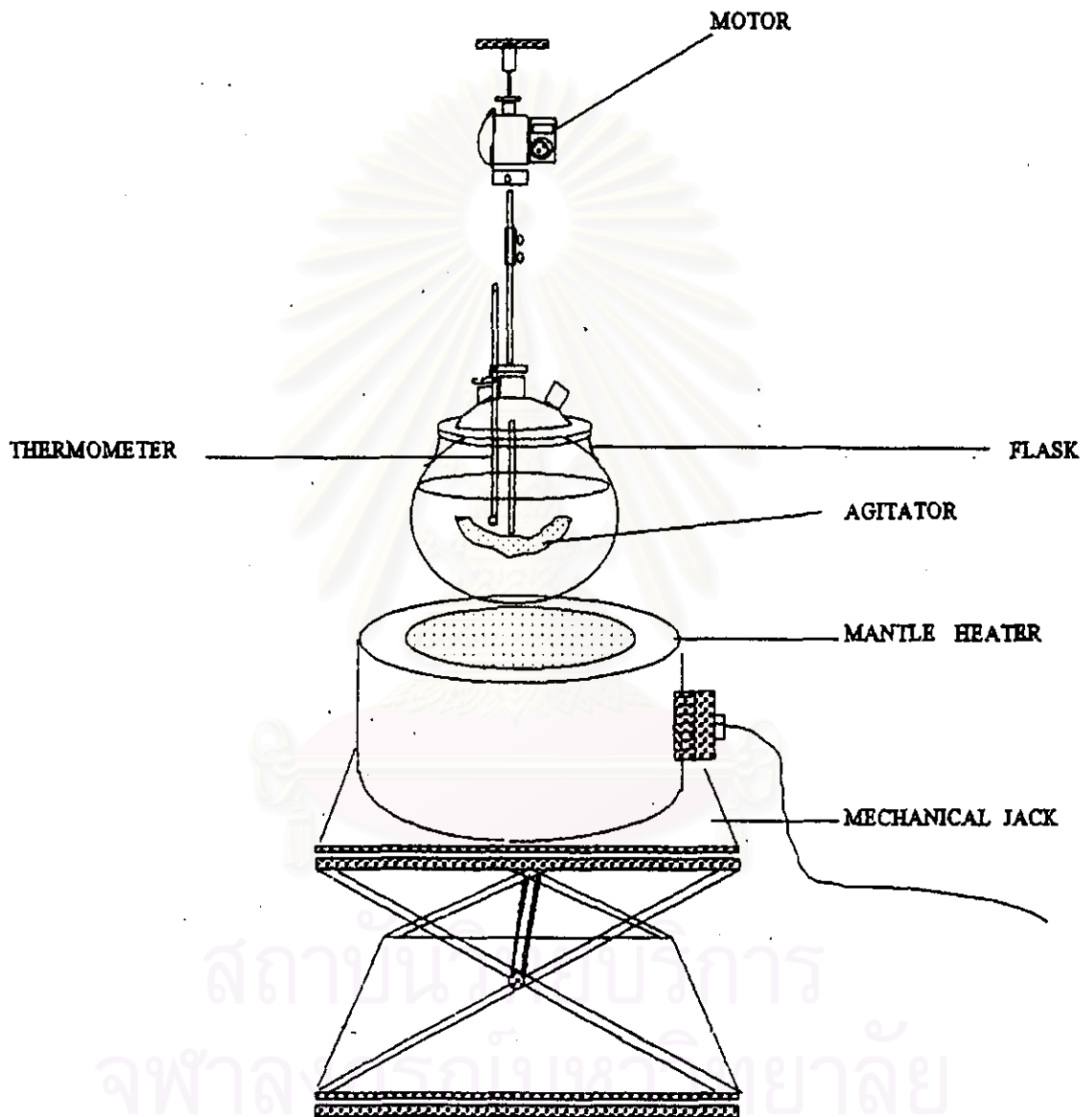


Figure 4.1 Sponge-synthesis unit

#### **4.1.4 METHOD OF MAKING PVA SPONGE**

The polyvinyl alcohol solution of 10 % concentration combined with the surfactant. And the resultant mixture was agitated for 5 minutes and agitation rate about 1800rpm. The sulfuric acid was added to the mixture, the temperature was then raised to 50°C and agitation was continued until the temperature approached room temperature. Formaldehyde, at 30°C, was slowly added to the froth and again beaten to disperse the formaldehyde and acid until it has attained maximum volume. The resulting froth is poured into a plastic mold and allowed to cure at room temperature for 20 hours. After the sponge had cured, it was washed to remove the unreacted material. The alternate steps of washing and squeezing are repeated until substantially all of the elutable surfactant, formaldehyde and acid are removed from the sponge. The alternate washing and drying is continued until a pH of 5.5-6.5 occurs in the wash water as measured by pH paper. The wash water upon vigorous shaking did not produce a foam which maintained itself longer than 10 seconds.

The finished sponge can be soaked in the plasticizer. The plasticizer of this work is a polyethylene glycol. When a solution is used an aqueous solution is preferred in which the sponge is soaked so as to absorb the maximum amount of solution and then the excess solution is removed.

## **4.2 SYNTHESIS OF ACTIVATED CARBON-FILLED SPONGE**

### **4.2.1 CHEMICALS**

The chemicals used in this experiment are described in 4.1.1. The activated carbon was provided by CABO KARN Co., Ltd. Its specification is given in Table 4.1.

Table 4.1 Specification of Activated Carbon for Air Purification.

Table 4.1 Specification of Activated Carbon for Air Purification.

Physical Properties	Specification
particle size distribution :under 325 mesh (ASTM mesh/mm)	max. 60%
apparent density (g/cc)	min. 0.53
moisture (%w/w)	max. 5
ash (%w/w)	max. 5
pH	8-10
surface area (BET)	min. 1000
Iodine Number (mg/g) (AWWA B604)	min. 950
Carbon Tetrachloride Adsorption (%w/w)	min. 40
Methylene Blue (cc/g) (JIS K1407-67)	min.130

#### **4.2.2 METHOD OF MAKING ACTIVATED CARBON-FILLED SPONGE**

PVA sponge can be modified to the activated carbon-filled sponge. Activated carbon can be introduced into the initial solutions before the frothing stage then the procedure is the same as that described in 4.1.4.

In this work, quantity of activated carbon powder used was varied.

#### **4.3 VARIABLES STUDIED**

The variables studied in this work are as follows:

##### **4.3.1 DETERMINE OPTIMUM COMPOSITION OF PVA SPONGE**

The experimental program is shown in Table 4.2 and Figure 4.2

Table 4.2. The experimental program

Formulation No.	Sodium lauryl sulfate (g)	Sulfuric acid (cm <sup>3</sup> )	Formaldehyde (cm <sup>3</sup> )	Curing time (hrs)
1	Variable	6.0	5.0	20
2	*	Variable	5.0	20
3	*	*	Variable	20
4	*	*	*	Variable

\* Optimum composition/condition of preceding stage will be selected for using in the next stage

#### **4.3.1.1 Molecular Weight of Pva Used**

Four different molecular weight were used in this work. They were 115000, 72000, 645000, and 43000. PVA of molecular weight 64500 was a mixture of equal amount of PVA of molecular weight 115000 and PVA of molecular weight 14000. And, PVA of molecular weight 43000 was a mixture of equal amount of PVA of molecular weight 72000 and PVA of molecular weight 14000. The experimental results are presented in 5.1.1.1

#### **4.3.1.2 Determining Optimum Quantity of Surfactant**

The amount of sodium lauryl sulfate was varied from 0.5 to 2.5 percent by weight of 40 gram/batch of polyvinyl alcohol solution, which represents the recommended value[8] for yielding the maximum froth volume, froth stability and giving a uniformity of pore size. The experimental results are presented in 5.1.1.2

#### **4.3.1.3 Determining Optimum Formaldehydeconcentration**

Optimum concentration of sodium lauryl sulfate from the previous result was used for variation the formaldehyde concentration. The concentrations of

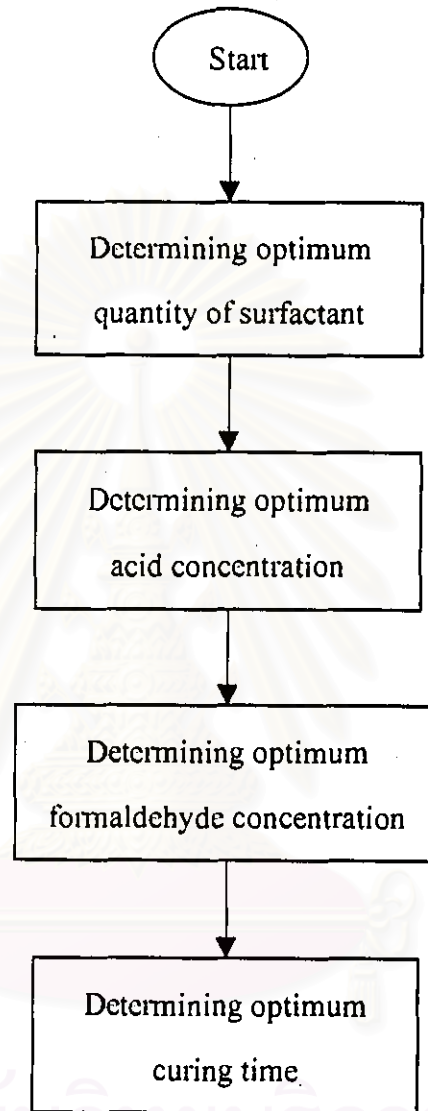


Figure 4.2 Experimental program for determining the optimum composition for each PVA molecular weight



formaldehyde was varied from 10 up to 30 percent by weight of polyvinyl alcohol solution were chosen for study. The experimental result is presented in 5.1.2.1

#### **4.3.1.4 Determining Optimum Acid Concentration**

Optimum quantity of sodium lauryl sulfate and formaldehyde concentration were used when the acid concentration was determined. Sulfuric acid concentration was varied from 10 to 30 percent by weight of polyvinyl alcohol solution. The experimental result is presented in 5.1.2.2

#### **4.3.1.5 Determining Required Curing Time**

After the frothing stage had been completed, the sponges were cured by placing in the plastic mold at the room temperature (27-30°C). An observation concerning curing time range from eight hours up to twenty hours was investigated. The experimental result is presented in 5.1.2.3.

### **4.3.2 DETERMINING OPTIMUM QUANTITY OF ACTIVATED CARBON**

The results of observation in 4.3.1 were used as the basis for foaming process of activated carbon-filled sponge. Only the optimum factors were used. In this study, the temperature and other conditions were controlled as same as in 4.1.4.

## **4.4 SPONGE CHARACTERISATION**

### **4.4.1 DESIGN AND CONSTRUCTION OF A TESTING UNIT FOR DETERMINING ADSORPTION ABILITY OF SPONGE CHARACTERISTICS**

The testing unit of which the detail is shown in Figure 4.3, is a steady-state flow type to provide the following data:

- 1) resistance of sponge to air flow; this is determined by measuring the pressure drop across a sponge.
- 2) gaseous pollutant adsorption ability of activated carbon-filled sponge. Samples of air and pollutants mixtures before and after passing through the sponge are analyzed by Gas Chromatography.

A sponge slice of 3 mm. in thickness and 5 cm. in diameter was used as a testing specimen. It was placed in the sponge housing of the testing unit.

#### **4.4.2 PORE SIZE AND PORE DISTRIBUTION**

Sample of sponge for pore size observation was a slice of 1 millimeters in thickness with flat surface. The surface of sponge layer was slightly coated with ink to shade the pore for better observation with a microscope and photography. The experimental result is presented in 5.3.1.

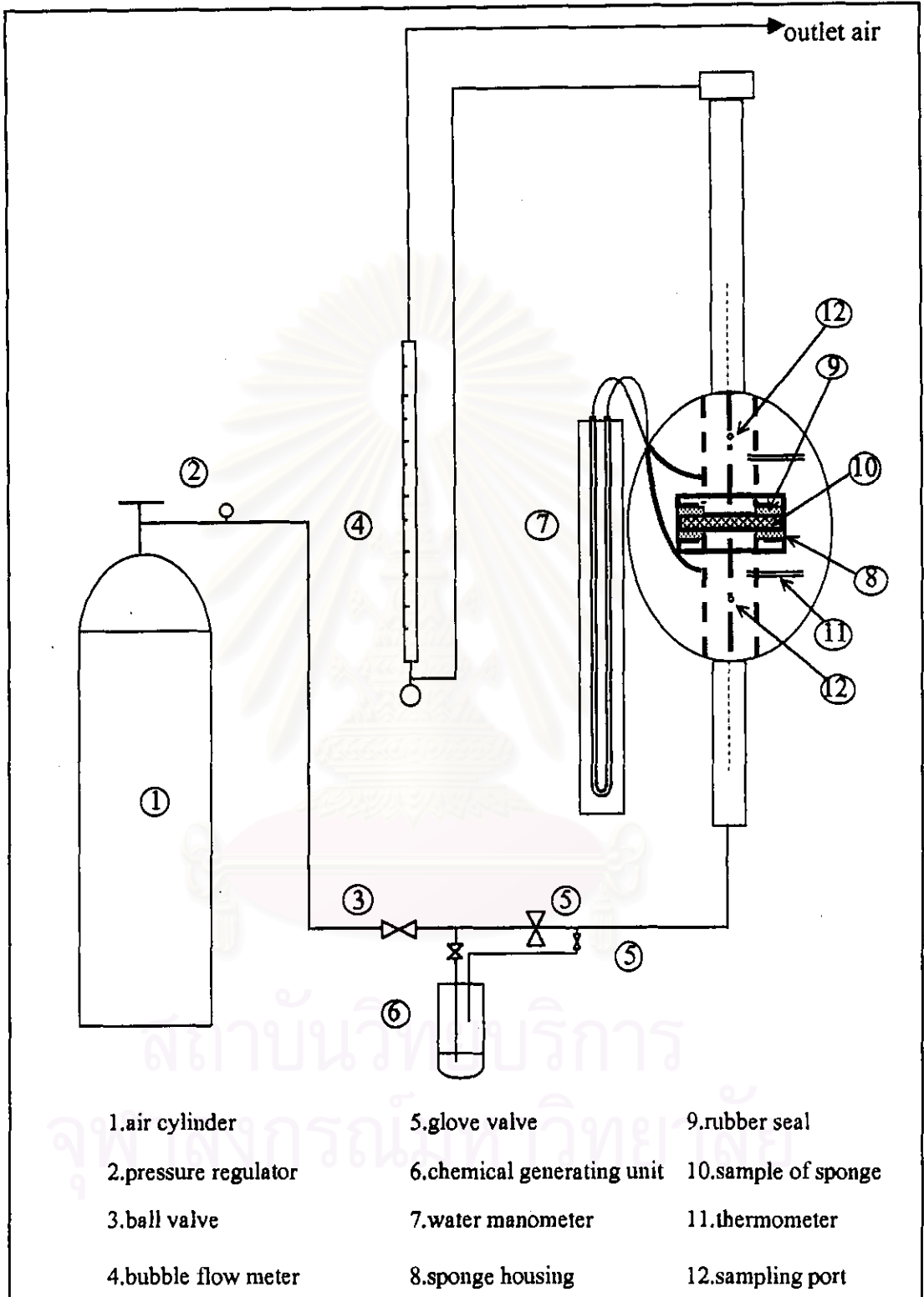


Figure 4.3 Schematic diagram of a testing unit

#### 4.5 GAS CHROMATOGRAPHY EXPERIMENT

Hexane was used as a gaseous pollutant. Its quantity in the air was measured with the aid of a gas chromatography. The operating condition of the gas chromatography is presented in Table 4.3.

Table 4.3 Operating condition of gas chromatograph.

Gas Chromatography	Model : HP 6890
Oven	
Initial temperature	80 °C
Inlet	
Initial temperature	200 °C
Flow rate	5.6 ml/min
Gas type	Helium
Column	Capillary column : Wasson KC21
Boiling point column	300 °C
Pressure	20.9 psi
Initial flow	2.1 ml/min
Average velocity	43cm <sup>3</sup> /sec
Detector	Flame Ionization Detector (FID)
Temperature	250 °C
Hydrogen flow	30 ml/min
Air flow	400 ml/min
Make up flow	30 ml/min
Make up gas type	Helium

**Experimental procedure:**

1. Install the chromatographic column, and establish stable instrument operation at the proper operating conditions as shown in Table 4.3. Adjust column temperature and flow rate to achieve sufficient resolution. A retention time of 5-6 min for hexane has been found to yield sufficient resolution with the recommended column.

2. Inject a repeatable volume of sample into the chromatograph. Start the recorder and obtain the chromatogram.

3. Process and analyse the data given out from the gas chromatograph.



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