

CHAPTER III

EXPERIMENTAL



This chapter describes the experimental procedures for RF and RF/alumina preparation. It is divided into four parts: materials used, electrospinning apparatus, experimental procedures and samples characterizations.

3.1 Materials

List of chemicals employed in this work and their structures are listed in Table 3.1 and 3.2, respectively.

Table 3.1 List of chemical agents used in the research.

Chemical agents	Using for	Manufacturer / Grade
Resorcinol ($C_6H_4(OH)_2$)	Synthesis of Resorcinol- Formaldehyde (RF) solution	Fluka, 99%
Formaldehyde (HCOH)	Synthesis of Resorcinol- Formaldehyde (RF) solution	Ajax Fine Chemical, 37% w/v; stabilized by 11-14 wt% methanol
Sodium carbonate(Na_2CO_3)	Synthesis of Resorcinol- Formaldehyde (RF) solution	Ajax Fine Chemical, 99%
Distilled water (H_2O)	Synthesis of Resorcinol- Formaldehyde (RF) solution	
Aluminum acetylacetonate ($C_{15}H_{21}AlO_6$)	Synthesis of Alumina / Resorcinol-Formaldehyde (RF) gel	Fluka, 95%
Acetic acid (CH_3COOH)	Synthesis of Alumina / Resorcinol-Formaldehyde (RF) gel	QReC , 97%

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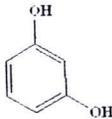
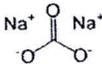
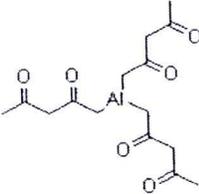
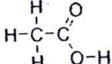
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Table 3.2 Chemical structure of compounds used in this research.

Chemical name	Designation	Chemical structure
Resorcinol	R	
Formaldehyde	F	
Sodium carbonate	C	
Aluminum acetylacetonate	A	
Acetic acid	Ac	

3.2 Electrospinning Apparatus

The schematic of the electrospinning apparatus used in this work is shown in Figure 3.1. The components of the apparatus and their functions are described as follows.

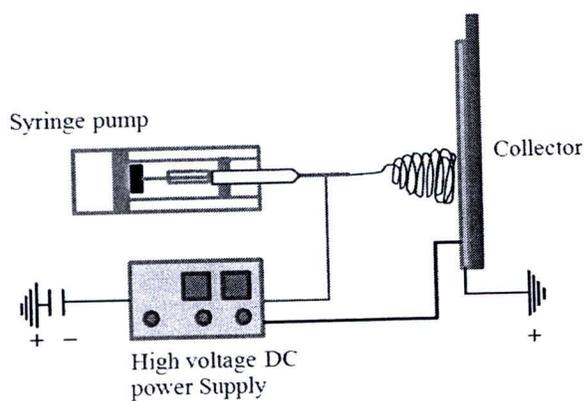


Figure 3.1: Schematic of an electrospinning setup.

The system is consisting of:

- A high-voltage power supply (Protek, DF 1730SB3A, U.S.A) was used to generate either positive or negative DC voltage up to 30 kV, with very low electrical current of 166 microamperes.
- A 5 ml syringe was used as a container for electrospinning solution. The syringe is made of a plastic and set in horizontal orientation.
- A syringe pump (kdScientific, U.S.A) mounted with a syringe to control the flow rate of the spinning solution to be 0.8 ml/h.
- A stainless steel needle (gauge No.21 and the inner diameter of 0.514 mm) was used as a nozzle. The tip of the needle was cut into a flat shape and the length of the needle was 1 cm. The needle is connected to the negative electrode of the DC supply.
- Aluminum foil was used as a ground collector.

3.3 Experimental Procedures

3.3.1 Preparation of RF gel

RF-gel were synthesized by polycondensation of resorcinol (R) and formaldehyde (F) according to the method proposed by Pekala et al [4]. Measured quantities of resorcinol and distilled water (W) were added into a beaker and mixed properly for about 15 min with the help of a magnetic stirrer. Sodium carbonate (C) was dissolved in distilled water in separate beaker. The two solutions were then mixed and stirred continuously for 15 min. Then added formaldehyde into the mixer and stirred continuously for 15 min. The ratio of C/W was kept constant at 10 mol/m³. The molar ratio of R/F, R/W and R/C were varied in this study in the range of 0.1-2.0, 0.03-2.0, and 50-2000, respectively.

3.3.2 Preparation of aluminium-doped RF gel

In the preparation of aluminium-doped RF-gel, aluminium acetylacetonate (A) was used as a precursor for aluminium. RF-gel was firstly prepared according to the method described earlier. The RF-gel was aged for predetermined period of time before being added with a mixture containing F and A in the molar ratio A/F of 1.1, using A/R molar ratio in the range of 0.167-0.236 and F and A mixed properly for about 45 min with the help of a magnetic stirrer. The mixture was stirred for 15 min. In this study, the aging period was varied to investigate the extent of the reaction between Al-precursor to the RF-gel.

3.3.3 Freeze drying of the RF- products

The RF products were pre-frozen by a liquid N₂ for 24 h to freeze the solvent inside the pores, then moved the RF products to a vacuum freeze-drying chamber and dried at -54°C for 2 days.

3.3.4 Pyrolysis of the RF –products

After drying, the RF products were put into a ceramic boat, placed in a horizontal tubular flow reactor and heated up to 250°C under constant flow of nitrogen gas. The flow rate of nitrogen was maintained at 40 ml/min. The heating rate was fixed at 10°C/min. After being held at 250°C for 2 h, the temperature was raised to 750°C using the same heating rate and held at 750°C for 4 h.

3.4 Sample Characterizations

3.4.1 Scanning electron microscopy (SEM)

Morphology of the obtained products was studied using scanning electron microscope (SEM) model JSM-6400 at Scientific and Technological Research Equipment Centre Foundation, Chulalongkorn University. Size of the products was then measured from the micrographs, using image processing software (JEOL Semafore 5.0).

3.4.2 Viscosity measurement

Viscosity of the electrospinning solution was measured using a Brookfield Programmable DV II+ viscometer at the Center of Excellence on Particle Technology, Chulalongkorn University. Using small sample adaptor, spindle No.27, fixed stirring speeds at 40 rpm and sample volume 10 ml.

3.4.3 Fourier transform infrared spectroscopy (FT-IR)

Function groups of spinning solution were identified by using Fourier transform infrared spectrophotometer (FT-IR) Model Spectrum One (Perkin Elmer). Infrared spectra were recorded between wavenumber of 600 and 2000 cm^{-1} .

3.4.4 Surface area measurement

The surface area, pore volume and pore size were measured by an ASAP 2420 adsorption analyzer (Micromeritics) using nitrogen as the adsorbate at the Center of Excellence on Particle Technology, Chulalongkorn University. The operating conditions are as follows:

Sample weight	~ 0.1-0.2 g.
Degas temperature	200 °C
Vacuum pressure	< 10 mmHg

