

## CHAPTER III EXPERIMENTAL

### 3.1 Chemical and Materials

#### 3.1.1 Banana Trunks

The *Musa sapientum* Linn banana trunks were obtained from local banana farm in Bangkok province, Thailand.

#### 3.1.2 Chemicals

- (a) Aniline monomer
- (b) Ammonium persulfate
- (c) Sodium hydroxide analytical grade
- (d) Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>)
- (e) Hydrochloric acid (HCl)
- (f) Silver nitrate (AgNO<sub>3</sub>)
- (g) Sodium borohydride (NaBH<sub>4</sub>)
- (h) Methanol

### 3.2 Equipment

#### 3.2.1 Fourier Transformed Infrared Spectroscopy (FTIR)

Polyaniline coated on cellulose sheets will be analyzed by a FTIR spectrometer (Thermo Nicolet Nexus 670). The fibers will be placed into the Smart Multi-Bounce HATR sample compartment of the spectrometer and continuously purged with dry air. For each spectrum, 64 scans will be acquired at a spectra resolution of 4 cm<sup>-1</sup>.

#### 3.2.2 Thermogravimetric Analyzer (TGA)

The thermal stability and the decomposition temperature of raw banana fibers, treated banana fiber, polyaniline, polyaniline coated on cellulose sheets will be analyzed by thermogravimetric analysis (TGA) (Dupont Instrument TGA 5.1, model 2950). The temperature range study is 30-800 °C. TGA patterns are measure at a heating rate of 10 °C/min under a nitrogen gas atmosphere.

### 3.2.3 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) will be performed on platinum-coated samples, which will be obtained using a polaron sputter coater. A SEM operating condition typically at 10 kV will be employed for morphology study. Samples will be mounted onto the sample holder, sputter-coated with platinum, and finally use the SEM analysis.

### 3.2.4 X-ray Photoelectron Spectroscopy (XPS)

XPS analysis will be conducted to analyze the surface chemical state and elemental compositions of the polyaniline coated on cellulose sheets.

### 3.2.5 X-ray Diffraction (XRD)

XRD (Rigaku) is an equipment to determine the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident x-rays to diffract into many specific directions.

### 3.2.6 Contact Angle Measurement

Hydrophobicity of the surface will be evaluated by measuring the contact angle formed between water drops and the surface of the modified papers using contact angle measuring system G 10 (KRUSS). For this purpose, the drops of water will be mounted on five different areas of the surface with a microsyringe. The results will be mean values of five measurements on different parts of the cellulose sheets.

### 3.2.7 Bending Test

The stiffness of cellulose sheet without solution plasma and with solution plasma at different time for treatment was determined by bending test.

### 3.2.8 Gel Permeation Chromatography (GPC)

GPC was used to determine the weight-average molecular weight (M<sub>w</sub>) of polyaniline. Polyaniline was dissolve in tetrahydrofuran (THF) and filtrated through nylon 66 membrane with the pore size of 0.45 μm (Millipore, USA) before injection into the GPC instrument.

### 3.2.9 Electrical Conductivity

The electrical conductivity of synthesized polyaniline coated on banana trunk papers will measure at ambient temperature and approximately 50% humidity using a conventional two-probe technique equipped with 6517A

Electrometer/High Resistance Meter (Keithley, model 7517A). The applied voltage and the resultant current were converted to the electrical conductivity by following equation

$$\sigma = \frac{1}{\rho} = \frac{1}{(R_s \times t)} = \frac{I}{(K \times V \times t)}$$

where  $\sigma$  is the specific conductivity (S/cm)

$\rho$  is the specific resistivity ( $\Omega$  cm)

$R_s$  is the resistivity of sample ( $\Omega$ )

$I$  is the resultant current (A)

$K$  is the geometric correction factor

$V$  is the applied voltage (voltage drop, V)

$t$  is the thickness of sample

### 3.3 Methodology

#### 3.3.1 Preparation of Cellulose Fiber

Banana trunks were cut into the length of 10 mm to 30 mm and dry for 48 hours in an oven at 60°C. The dried banana chips were then soaked in different concentrations of NaOH solutions (5, 10, 15 and 20 %w/v) at 100°C for 1 hr. After thoroughly rinsed with distilled water, the banana chips were treated with a 15% (w/v) H<sub>2</sub>O<sub>2</sub> solution at 95°C for 1.5 hr. The purified cellulose fibers were disintegrated to smaller sizes by using a mixer and were partially hydrolyzed at 70°C for 30 minutes using different concentrations of hydrochloric acid solutions (1, 1.5, 2 and 2.5 M). The resulting cellulose fibers were rinsed with distilled water until neutral and then disintegrate with a mixer until free of lumps when suspended in water.

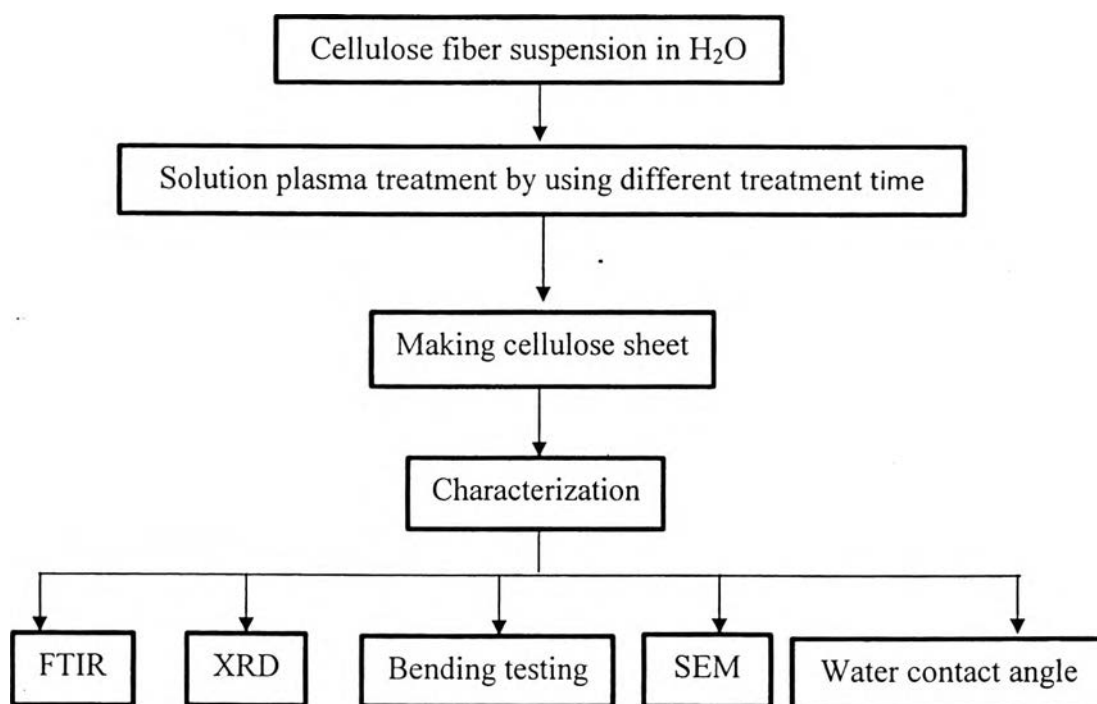
#### 3.3.2 Preparation of Cellulose Sheet

Cellulose fiber suspension was poured onto a filter paper placed in a Buchner funnel. Water is drawn through the funnel into the flask below by vacuum.

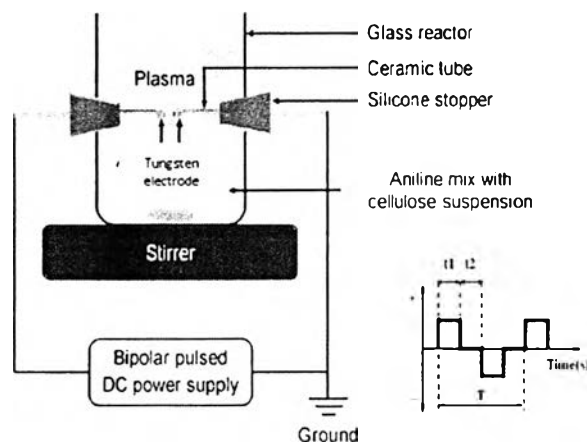
The fibers were trapped by the filter and form sheet after remove of water. The cellulose sheet was put between two filter papers to absorb moisture and a weight will applied on the top to provide pressing force during drying at room temperature for 24 hr. The cellulose sheet was then dried in an oven at 60°C for 24 hr.

### 3.3.3 Treatment of Cellulose Fibers by Solution Plasma

As showed in figure 3.1 cellulose fiber 1 g. was suspended in distilled water 100 ml. and then poured into the reactor equipped with a pair of tungsten electrodes. Plasma was generated at frequency of 23 kHz, voltage of 1.6 Kv, current of 1 A, pulse width of 2  $\mu$ s and electrode gap of 0.5 mm as shown in figure 3.2. Plasma treatment time was varied at 0, 30, 60, 90, 120, 180 and 240 minutes.



**Figure 3.1** Flow chart of treatment of cellulose fibers by solution plasma.



**Figure 3.2** Solution plasma set up.

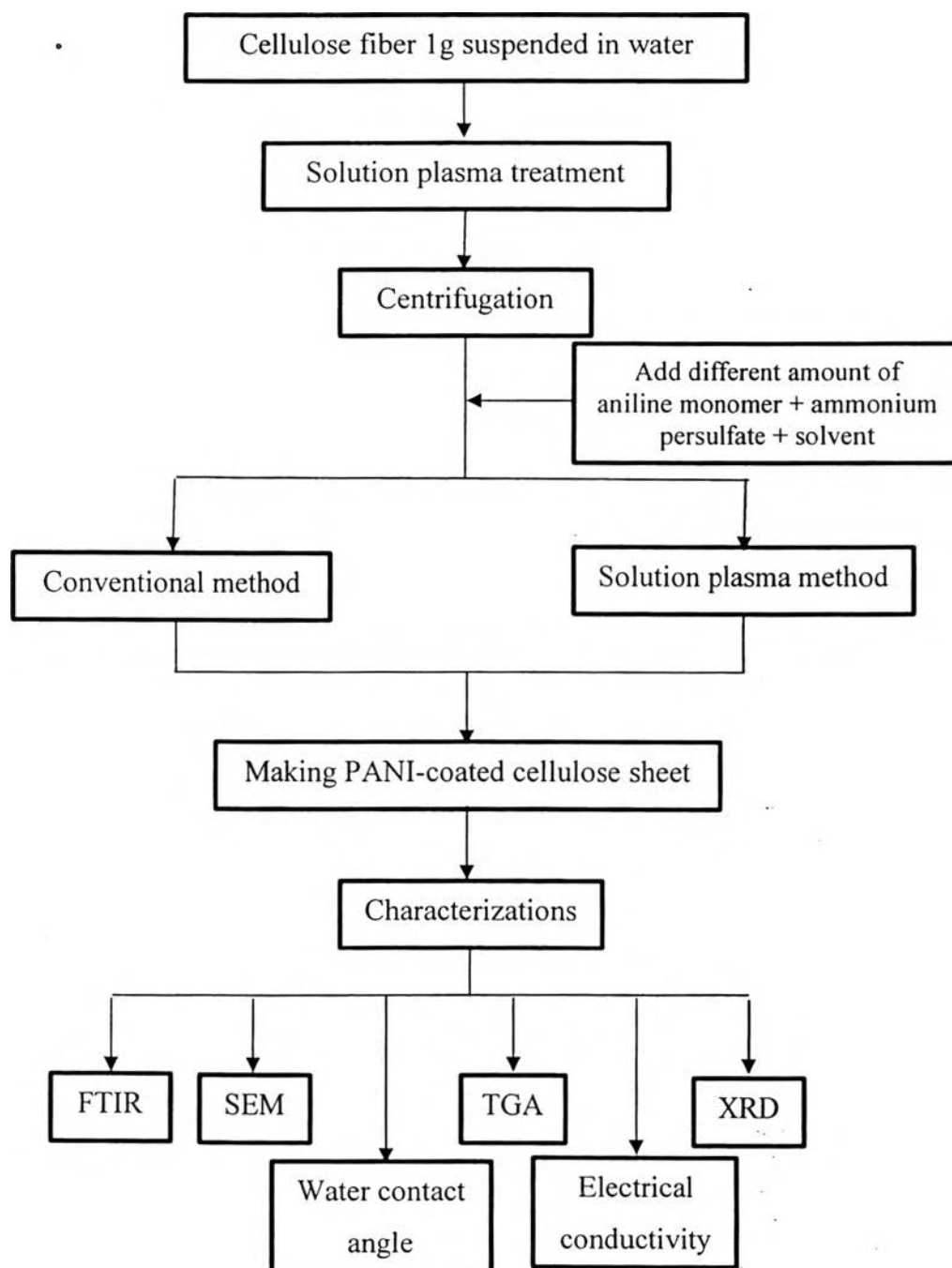
### 3.3.4 Polymerization of Polyaniline Coated on Cellulose Sheet by Solution Plasma Process.

The plasma-treated cellulose fiber was mixed with aniline monomer at difference ratios in 0.4 M HCl solution. Ammonium persulfate dissolved in 0.4 M HCl was slowly dropped into the as-prepared cellulose fiber mixed with aniline monomer as showed in figure 3.3. All of this was done in a solution plasma reactor. The plasma was generated between two tungsten electrodes using frequency of 15 kHz, voltage of 1.6 KV, current of 1 A. and pulse width of 2  $\mu$ s. The two electrodes having the gap distance of 0.5 mm was equipped inside the plasma reactor where the polymerization reaction occurred. Once the power is applied, the plasma will be generated between the two electrodes.

### 3.3.5 Preparation of Polyaniline and Silver Particles Co-Coated on Cellulose Sheets.

The cellulose fiber suspension 1 g, silver nitrate having different concentrations and  $\text{NaBH}_4$  were added into the plasma reactor and plasma was applied for 1 hour. After that the cellulose fiber was washed with distilled water to remove the unbound silver particles that did not attach on cellulose fiber. After that cellulose fiber containing silver particles was mixed with aniline monomer and

stirred for 30 minutes. Ammonium persulfate was slowly added into the cellulose fiber suspension and the plasma was provided for 20 minutes.



**Figure 3.3** Flow chart of polymerization polyaniline coated on cellulose sheet.