CHAPTER III

EXPERIMENTAL

3.1 Materials and Equipment

3.1.1 Materials

(a) Nylon mesh 100% grade from Japan

(b) Shrimp shell by Surapon Food Public Co, Ltd.

3.1.2 Chemicals

(a) Chitin (from shrimp shell)

(b) Calciumchloride dihydrate

(c) 98% Methanol

(d) Nylon 6,6 pellet

(e) Hydrochloric acid 37%

(e) Sodiumhydroxide

(f) Sodium borohydride laboratory grade

3.1.3 Reagent Gases for Reaction

Gas (a), (b) and (c) used for plasma treatment were obtained from Thai Industrial Gas Co., Ltd. As follows:

(a) Air

(b) Argon

(c) Nitrogen

3.2 Instruments for characterization

3.2.1 Contact Angle Measurement

Hydrophilicity of the surface will be evaluated by measuring the contact angle formed between water drops and the surface of the modified nylon-chitin membranes using contact angle measuring system G 10 (KRUSS). For this purpose, the drops of water will be mounted on three different areas of the surface with a microsyringe. The results will be mean values of three measurements on different parts of the nylon mesh.

3.2.2 Thermogravimetric Analyzer (TGA)

The thermal stability and the decomposition temperature of nylon-chitin membranes were analyzed by thermogravimetric analysis (TGA) (Dupont Instrument TGA 5.1, model 2950). The temperature range studied was 50-700 °C. TGA patterns were measured 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 gold-coated samples, which will be obtained using a polaron sputter coater. The SEM operating condition typically at 10 kV will be employed for morphology study. Samples will be mounted onto the sample holder, sputter-coated with gold, and finally used for SEM analysis.

3.2.4 Fourier transformed infrared spectroscopy (FTIR)

The nylon-chitin membranes will be analyzed by a Thermo Nicolet Nexus 670 FTIR spectrometer. Samples will be recorded at a spectral resolution and wave number precision of 0.09 and 0.01 cm^{-1}, respectively. The plastic films (5.0x5.0 cm^2) 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^{-1}. A frequency range of 4,000-400 cm^{-1} was observed

3.2.5 Lloyd Tensile Tester

The mechanical test of the plasma treatment of nylon-chitin membranes with different concentration and untreated can determined for a dried rectangular film (10 × 1 cm^2) by stress-strain technique using Universal Testing Machine (Lloyd, Model LRX) at room temperature. The thickness of polymer films was in the range of 20-25 μm. A strain rate of 25 $mm.min^{-1}$ and gauge length of 50 mm was employed.

3.2.6 Wide Angle X-ray Diffraction (XRD) Analysis

The crystalline structure of nylon-chitin membranes was characterized by an X-ray diffractometer (Bruker AXS, D8 advance) operated with the use of Cu Kα as the X-ray source. The WAXD analysis was done in a continuous mode with a scan speed of 1° min-1 covering the angle (2θ) from 10° to 80°.

3.2.7 Water Absorbency Time

Water absorbency time of the nylon-chitin membranes was measured according to AATCC 79-2007 method. A water droplet of 37 µl was placed on the sheet, which was held horizontally in a frame. The time for the water droplet to get fully absorbed by the sheet was recorded.

3.2.8 Atomic Absorption Spectroscopy (AAS)

The amount of calcium content was determined by standard of calibration curve of calcium concentration that defines from atomic absorption spectroscopy (Varian SpectrAA 300) by convert mean absorbance to calcium content in ppm unit.

3.3 Dielectric Barrier Discharge Plasma Equipment

3.3.1 Power Supply Unit

The block diagram of the power supply unit is shown in Figure 13. For the first step, the alternating current (AC) input of 220 Volt and 50 Hz will be converted to direct current (DC) of about 70-80 Volt by DC power supply converter. For the second step, the DC current will be supplied through a 500-Watt power amplifier, which is connected to the Instek function generator to generate waveform and to amplify voltage and frequency. The signal of alternative current is a sinusoidal waveform. For the third step, the modified current will be passed through the transformer to convert to 230 Volt AC. Thereafter, the variable output will be finally transmitted to a high voltage current by nominal factor 130 times of low side voltage (input). An Extech® series 380801 power analyzer will be used to measure power, power factor, current, frequency, and voltage at the low side of the power supply unit

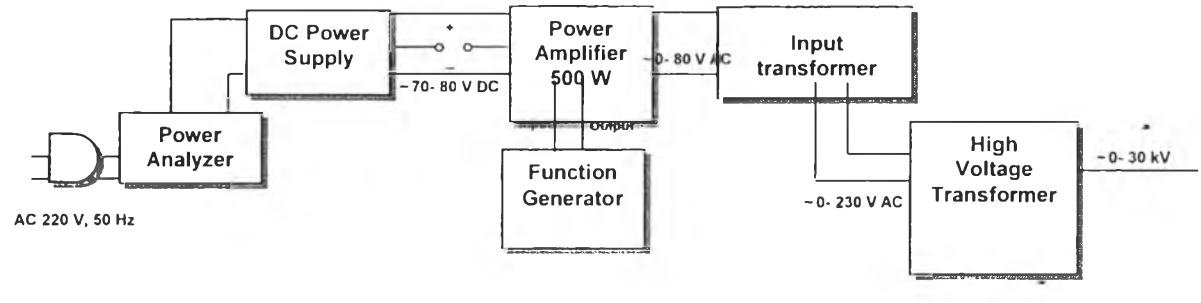


Figure 3.1 Block diagram of the power supply unit.

3.3.2 Experimental Setup

The experimental setup for surface modification of chitin coated nylon mesh and Nylon-chitin membranes films by using dielectric-barrier discharge is shown in Figure 3.2

3.3.3 Operating Condition for Dielectric Barrier Discharge (DBD)

The dielectric discharge has the thickness of 2 mm. The two parallel electrodes are stainless steel. The membranes were put into the parallel-plate dielectric barrier discharge (DBD) reactor for plasma treatment. The experiment was operated with the condition of voltage of 50 kV, frequency of 325 Hz and the electrode gap of 4 mm. The flowing gas was introduced directly through the gab of electrode and then membranes will treated by plasma. The treatment time is 30, 60, 90 and 120 seconds. Finally washing by deionized water and dry, next step is characterization and application this material

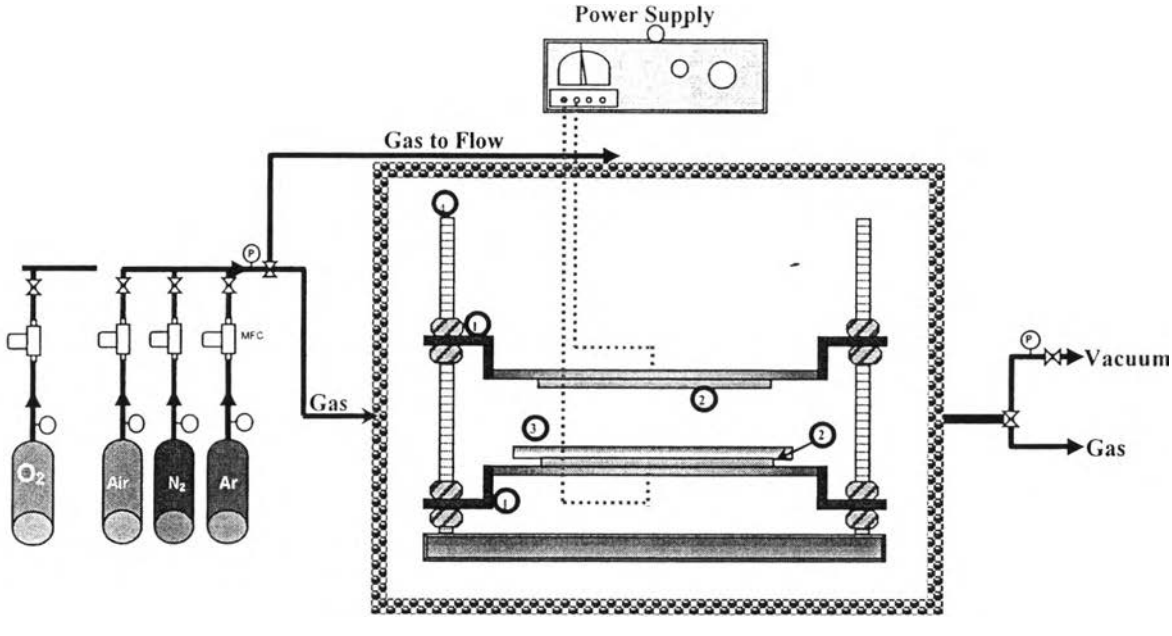


Figure 3.2 Schematic of experimental setup for dielectric-barrier discharge system.

1. Acrylic Structure
2. Electrode Plate (Stainless steel plate)
3. Glass Plate (Dielectric insulator)
4. Acrylic Screw

3.4 Methodology

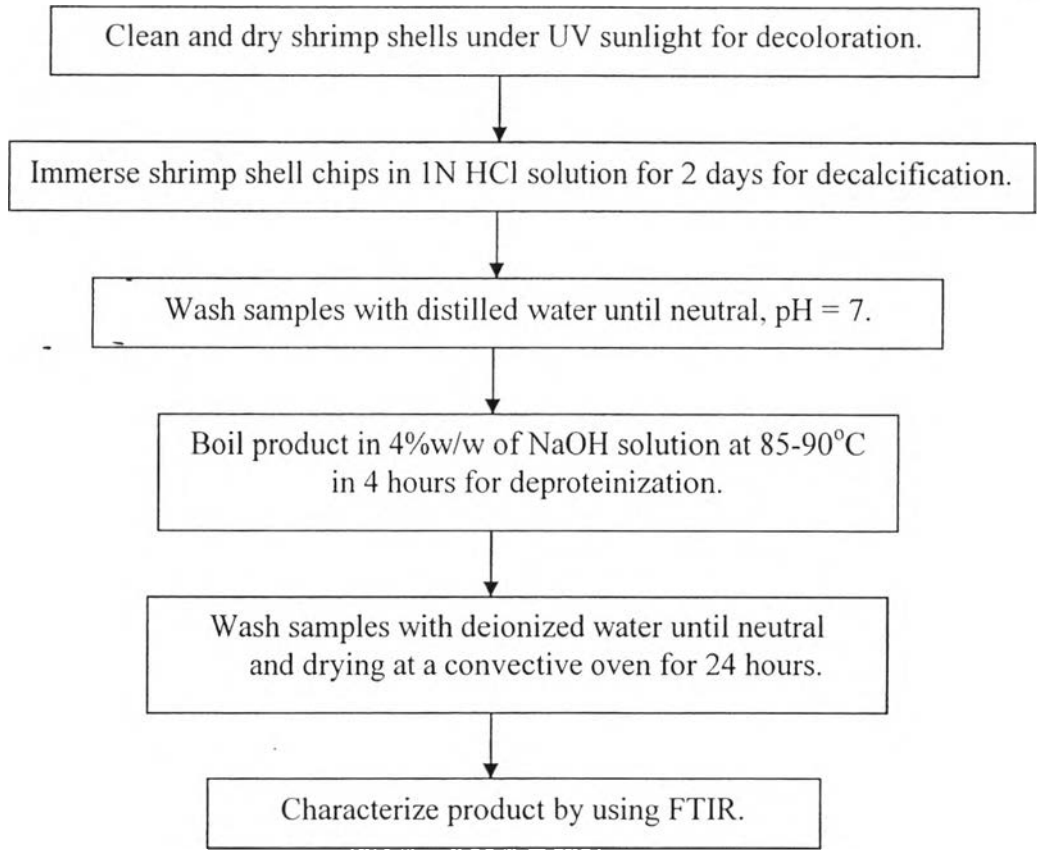
3.4.1 Preparation of Chitin

Chitin was prepared from shrimp shell by decalcification and deproteinization to remove calcium carbonate and protein, respectively.

1.) The shrimp shells were cleaned and dried under sunlight in one week
2.) Crush down into small powder.
3.) Shrimp shell powder was treated by immersion in 1N HCl solution for

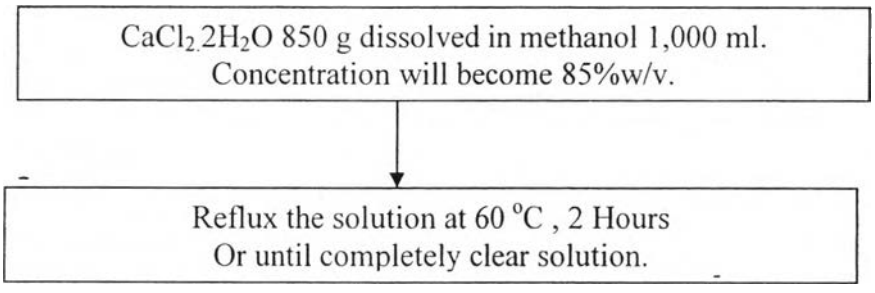
    2 days with occasional stirring. The decalcified product was washed with distilled water until neutral.
4.) Deproteinization was followed by boiling in 4 % w/w of NaOH solution at 85-90°C for 4 h. After NaOH solution was decanted, the powder was washed with deionized water until neutral.
5.) The product obtained was dried at 60°C in a convective oven for 24 h

Flow Process Chart Preparation of Chitin



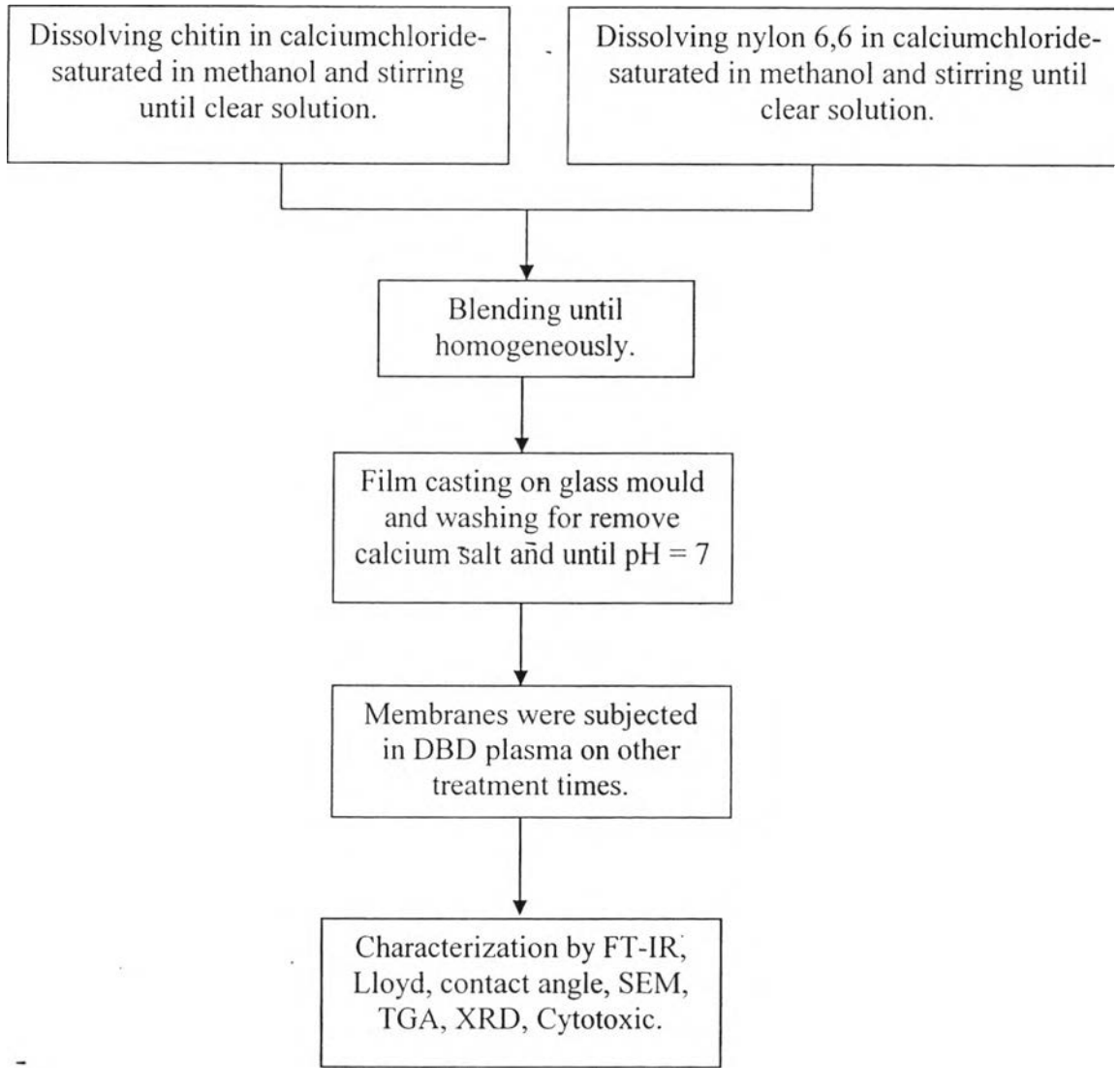
3.4.2 Preparation Calciumchloride-Saturated in Methanol

Flow process chart preparation of calcium chloride-saturated methanol



3.4.3 Preparation Nylon/Chitin Membranes (Solution Casting Method)

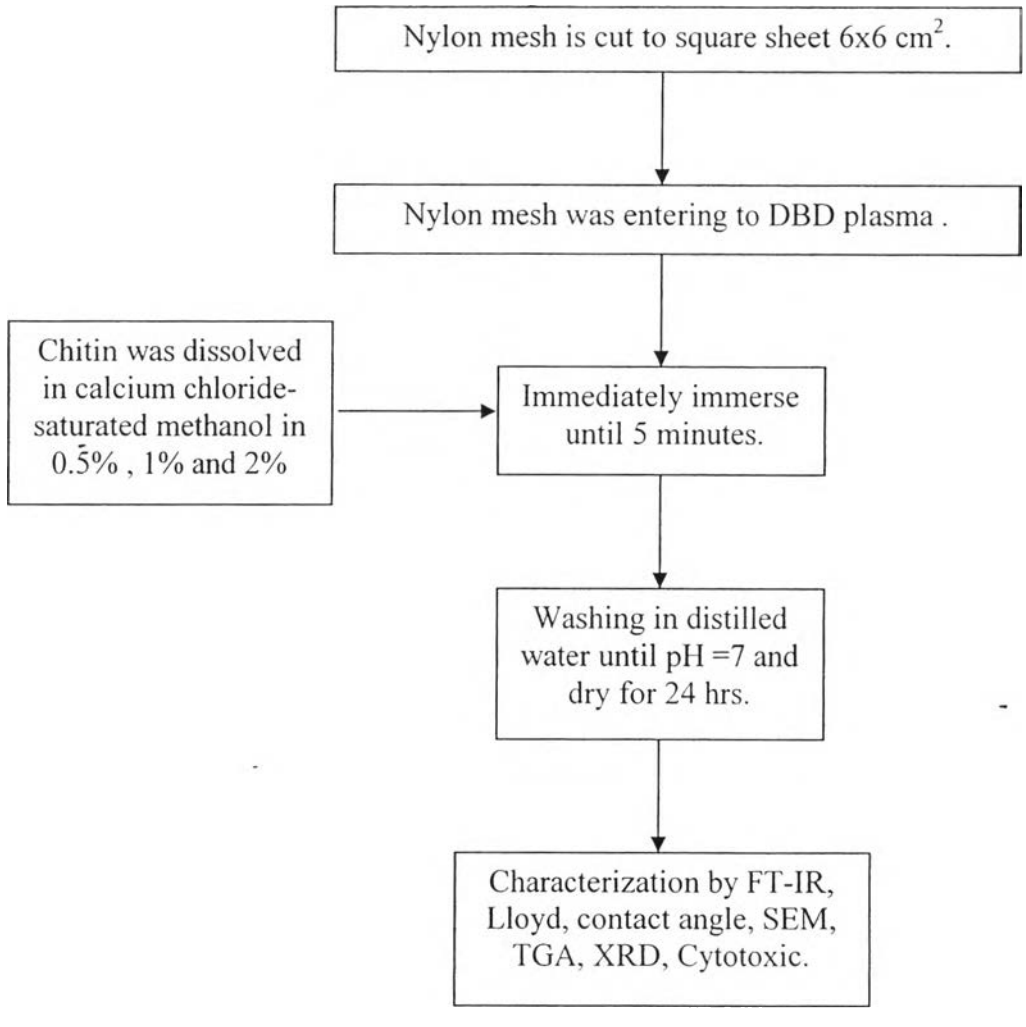
Flow Process Chart Preparation of Nylon/Chitin Membranes



Nylon and chitin were separately dissolved in calcium chloride-saturated methanol solvent before mixing together.

3.4.4 Preparation Chitin Coated on Nylon Surface via DBD Plasma(Surface Coating Method)

Flow Process Chart Preparation of Chitin Coated on Nylon Surface via DBD Plasma



3.4.5 Preparation the Standard Calibration Curve of Calcium Content

Flow Process Chart of Standard Preparation and Calibration Curve