CHAPTER III
EXPERIMENTAL

3.1 Chemical and Materials

3.1.1 Materials

Woven PET fabric was provided by Thai Negoro Co., Ltd (Thailand).

3.1.2 Chemicals

All chemicals, which were used for coating procedure, are described as follows:

(a) Silver nitrate ($AgNO_3$), assay 99.9%, obtained from Carlo Brea was used as antibacterial agent.

(b) Sodium borohydride solution ($NaBH_4$) was used to reduce the silver compound (Ag^{II}) to metallic silver particle (Ag^0).

3.1.3 Reagent Gases for Reaction

All gases used for plasma treatment were obtained from Thai Industrial Gas Co., Ltd. as follows:

(a) Helium (HP grade)

(b) Oxygen

(c) Nitrogen

(d) Air

3.2 Methodology

The flow chart of the entire experimental procedure is shown in Figure 3.1 below:

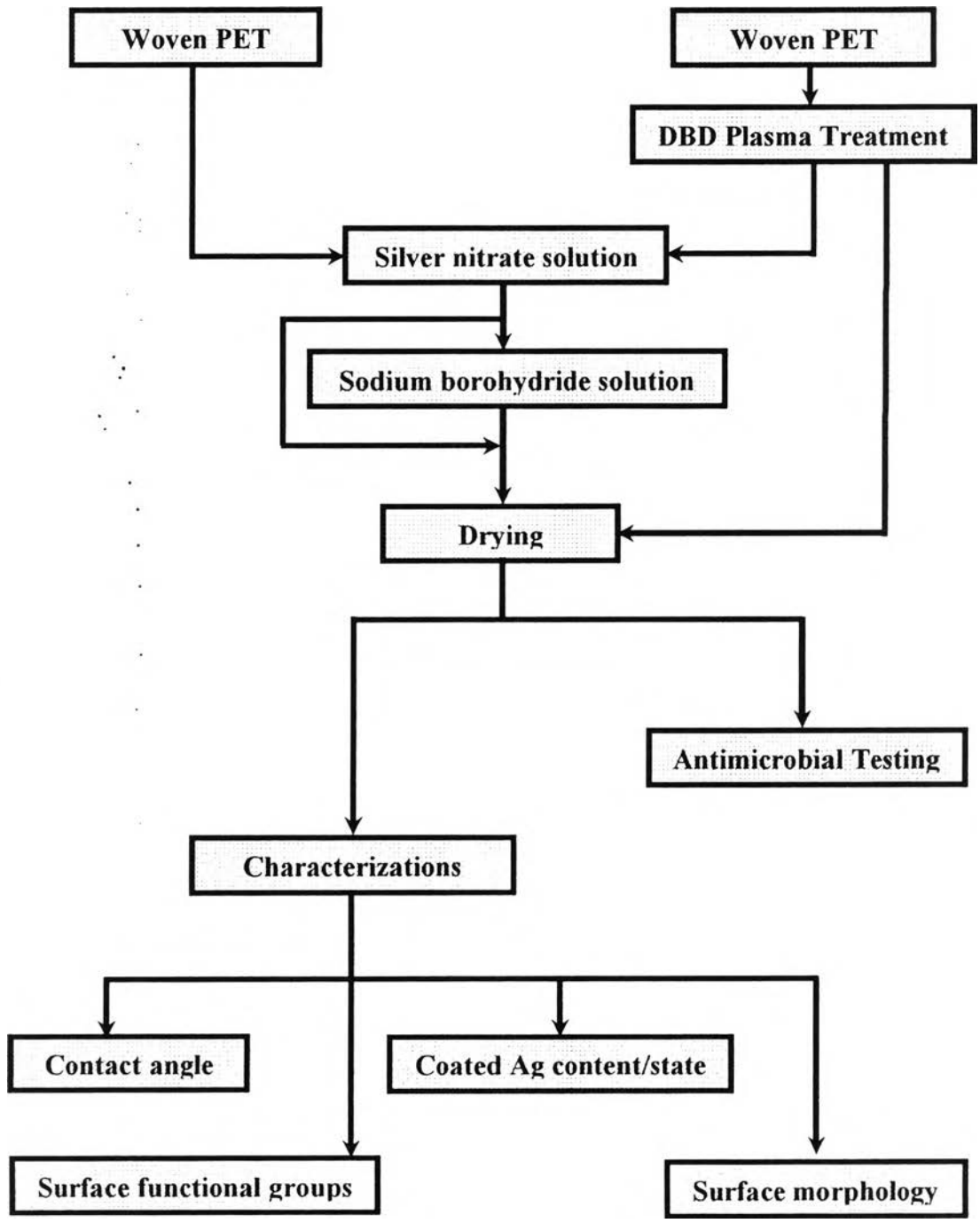

Figure 3.1 Flow chart of the entire experimental procedure.

3.3 Surface Characterizations

3.3.1 Wickability measurement

The wickability test of woven PET was carried out for hydrophilicity/wettability measurement before and after DBD plasma treatment. The wickability was estimated by testing fabric samples of 1 cm×5 cm area, measuring the time required for distilled water to rise 1 cm up the vertical fabric (25°C temperature, 50% relative humidity, 1 mm initial immersion depth). The values presented were averaged over five measurements for each set of treatment conditions. The tests showed that a specific sample had the same wickability in all directions, i.e. there is no preferential capillary flow induced by fiber or yarn orientation of the woven textile material.

3.3.2 Contact angles

Contact angle measurements were carried out by using a Digidrop from GBX. Digidrop apparatus, using a goniometric method, is used to calculate the contact angle between a liquid and a solid. The drop image was stored via a monochrome video camera using PC-based control acquisition and data processing. In this work, distilled water was used as the working liquid. The static contact angle values were the average of 5 measurements. As the drop size was small (20 μL), the gravitational force was neglected.

3.3.3 Scanning electron microscopy (SEM)

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

3.3.4 Atomic absorption spectroscopy (AAS)

Atomic absorption spectroscopy (AAS) was used for the quantitative determination of silver coated on the surface of modified woven PET.

3.3.5 X-ray photoelectron spectroscopy (XPS)

XPS analysis was conducted to analyze the surface chemical state and compositions of the plasma-treated woven PET.

3.3.6 Fourier transformed infrared spectroscopy (FTIR)

The woven PET was analyzed by a Thermo Nicolet Nexus 670 FTIR spectrometer. Samples were recorded at a spectral resolution and wave number precision of 0.09 and 0.01 cm^{-1}, respectively. The woven PET (5.0×5.0 cm^2) was placed into the Smart Multi-Bounce HATR sample compartment of the spectrometer and continuously purged with dry air. For each spectrum, 64 scans were acquired at a spectra resolution of 4 cm^{-1}.

3.3.7 UV-visible spectrophotometer

The surface plasmon resonance of the silver nanoparticle-coated woven PET was investigated by using a UV-Visible spectrophotometer (Hitachi U-2010 spectrometer).

3.4 Power Supply Unit

The block diagram of the power supply unit is shown in Figure 3.2. For the first step, the alternating current (AC) input of 220 V and 50 Hz was converted to direct current (DC) of about 70-80 V by DC power supply converter. For the second step, the DC was supplied through a 500-Watt power amplifier, which was connected to the Instek function generator to generate waveform and to amplify voltage and frequency. The signal of alternative current was a sinusoidal waveform. For the third step, the modified current was passed through the transformer to converted to 170 V AC . Thereafter, the variable output was finally transmitted to a high voltage current by nominal factor 250 times of low side voltage (input). An Extech® series 380801 power analyzer was used to measure power, power factor, current, frequency, and voltage at the low side of the power supply unit.

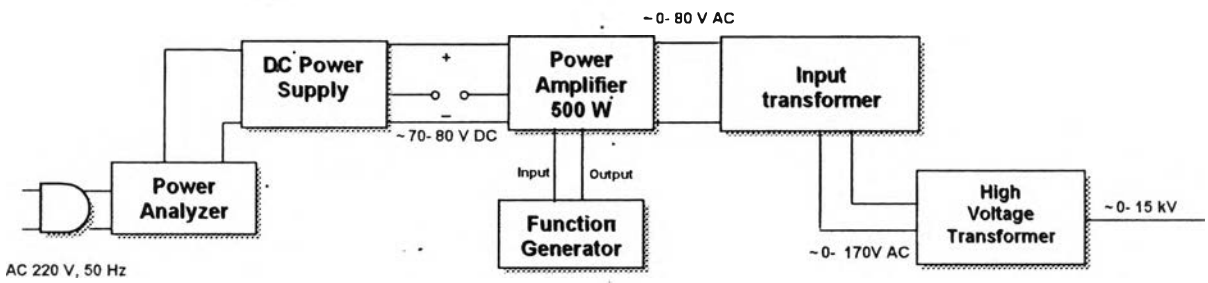

Figure 3.2 Block diagram of the power supply unit.

3.5 Experimental Setup

The experimental setup for surface modification of woven PET fabric by using dielectric barrier discharge is shown in Figure 3.3.

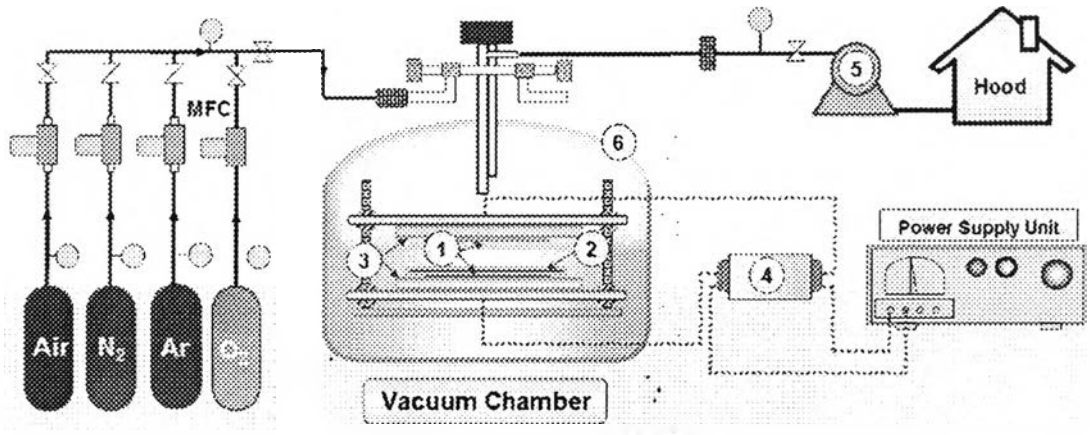

Figure 3.3 Schematic of experimental setup for dielectric barrier discharge system.

1. Electrode plate (Stainless steal plate)
2. Dielectric plate (Glass plate)
3. Acrylic plate
4. High voltage transformer
5. Vacuum pump
6. Vacuum chamber

3.6 Studied Conditions

The experiments were carried out in the atmosphere of 4 different gas types: air, nitrogen, oxygen, and argon. The conditions for all studied parameters, including electrode gap distance, applied voltage, input frequency, pressure in the reactor, and time for plasma treatment, are shown in Table 3.1.

Table 3.1 Experimental conditions used in this study

Type of feed gas	Gap distance (mm)	Input voltage (V)	Input frequency (Hz)	Pressure in chamber (bar)	Time of plasma treatment (s)
Air	4-15	15-80	50-500	1 and 0.15	10-120
O_2	4-15	15-80	50-500	1 and 0.15	10-120
N_2	4-15	15-80	50-500	1 and 0.15	10-120
Ar	4-15	15-80	50-500	1 and 0.15	10-120

3.7 Antimicrobial Activity Testing

Antimicrobial activities of silver nanoparticle-coated woven PET were investigated against *Escherichia coli* as the model gram-negative bacteria and *Staphylococcus aureus* as the model gram-positive bacteria. The antimicrobial activities of silver nanoparticle-coated woven PET were carried out by two methods.

3.7.1 The Disk Diffusion Method

This method was performed in Luria–Bertani (LB) medium solid agar Petri dish. The silver nanoparticle-coated woven PET was cut into a disc shape of 2.0×2.0 cm^2, sterilized by autoclaving 15 min at 120°C, and placed on *Escherichia coli*-cultured and *Staphylococcus aureus*-cultured agar plates, which were then incubated at 37°C for 24 h. Finally, inhibition zone was monitored.

3.7.2 The Colony Forming Count Method

Silver nanoparticle-coated woven PET was cut into a disc shape of 1.0×1.0 cm^2. Before inoculation of the bacteria, the pieces of sample were sterilized by autoclaving at 120°C for 15 min. The experimental design is shown in Figure 3.4. Sample was divided into two groups; each group consisted of four pieces. The first group was seeded with 1 cm^3 sterile nutrient broth as sterility control. The second group was seeded with fresh *Escherichia coli* or *Staphylococcus aureus* culture at a concentration of 10^7 colony forming units per cm^3 (cfu/cm^3), and then incubated in shaking incubator at 37°C for 24 h. After incubation, 500 cm^3 saline was added to each group and then all tubes were vortexed. The 0.05 cm^3 of bacterial suspension was drawn from each tube, spread on to a nutrient agar plate, and incubated at 37°C for 48 h for colony forming counts. The same procedure was performed on pure bacterial cellulose. The reduction percentage in bacterial count was calculated by the formula (Li *et al*, 2006):

$$\text{The percentage of reduction / increase in bacterial count} = \frac{(\text{Viable count at 0 h} - \text{Viable count at 24 h})}{\text{Viable count at 0 h}} \times 100\%$$

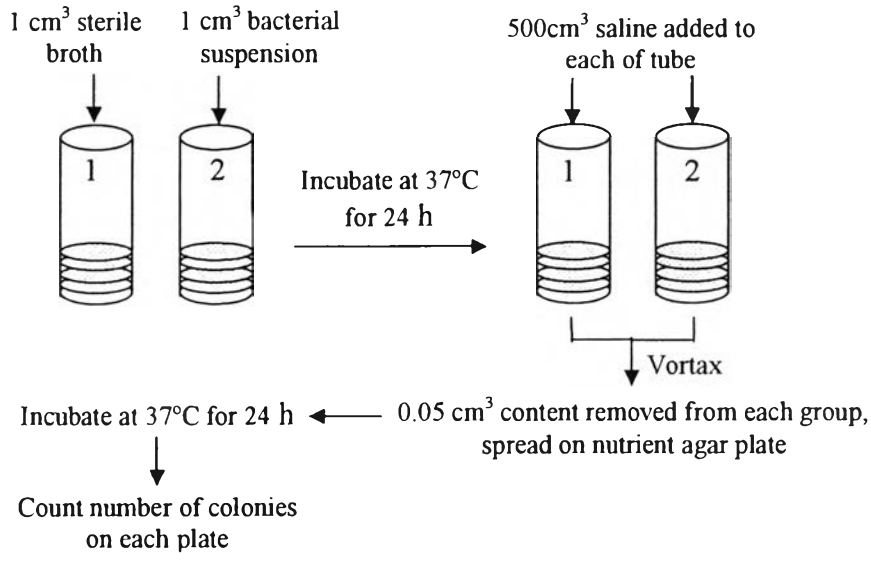

Figure 3.4 Flow chart showing the experimental procedure for antimicrobial activity study.