CHAPTER III

EXPERIMENT

3.1 The Experimental Scope

In this chapter, the synthetic-, natural- and blend-fiber fabrics were surface-modified by nitrogen plasma. The morphology and properties of untreated and plasma-treated fabrics were then characterized and compared. The flow chart of the entire are experimental procedure is shown below in Figure 3.1

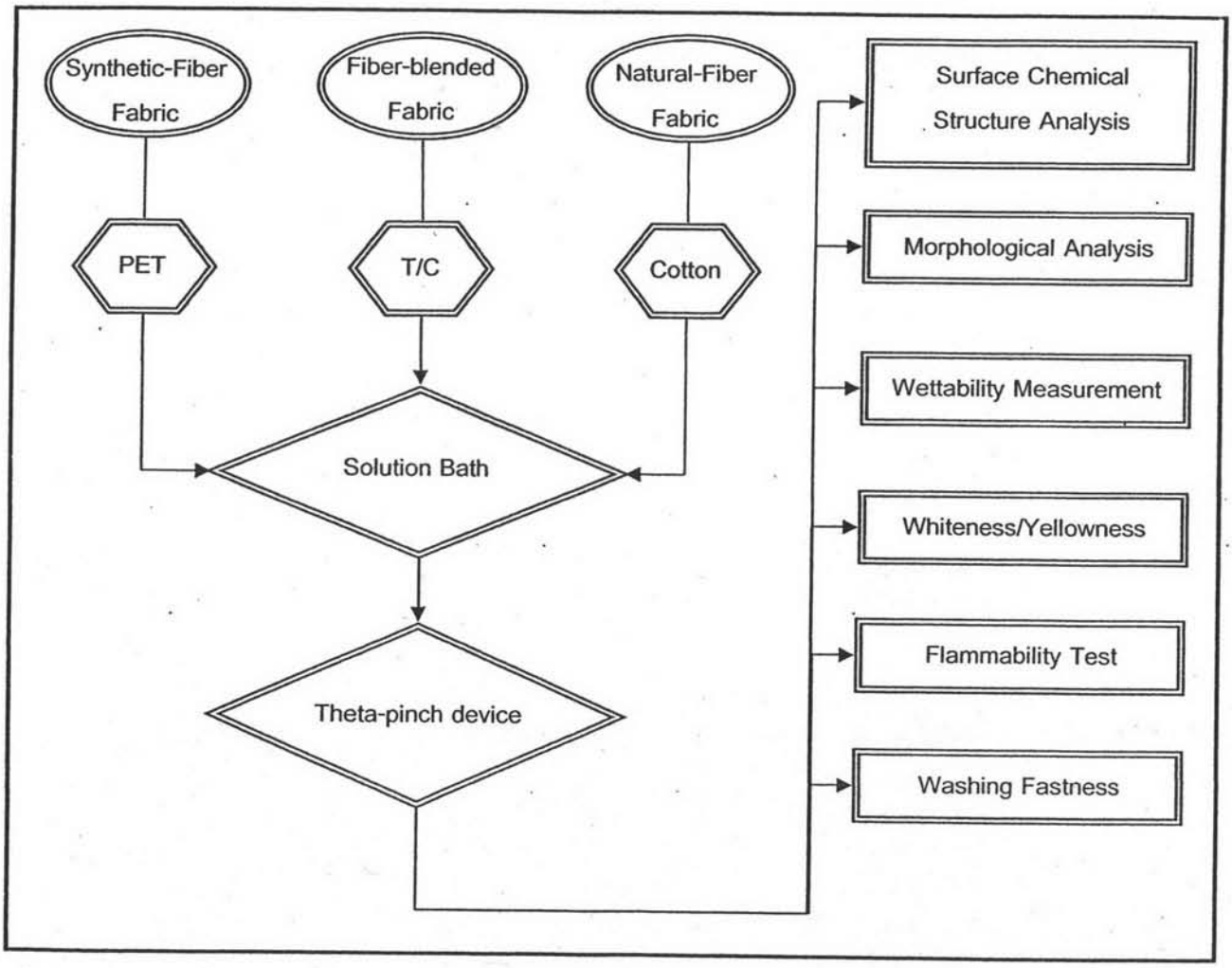

Figure 3.1 The scope of the experiment

3.2 Materials

3.2.1 Samples

3.2.1.1 Polyester (PET) woven fabric (plain); Thai Taffeta Co., Ltd

Yarn count : Warp 70/24 dtex, Weft 160/90 dtex

Density : Warp 118 ends/cm, Weft 80 picks/cm

Weight : 89 g/m^2

3.2.1.2 Cotton woven fabric (plain); Jim Thomson Co., Ltd

Density : Warp 35 ends/cm, Weft 20 picks/cm

Weight : 234 g/m^2

3.2.1.3 Polyester/cotton blended woven fabric (plain) (65/35)

Density : Warp 46 ends/cm, Weft 30 picks/cm

Weight : 101 g/m^2

3.2.2 Chemical

3.2.2.1 Diammonium Hydrogen phosphate from APS Ajax Finechem

3.2.2.2 Standard Soap from SDC Enterprises Limited

3.3 Machines and Equipments

3.3.1 Theta-pinch device supported by Asian African Association for Plasma Training (AAAPT)

3.3.2 Scanning electron microscope (SEM): JSM-6480LV, JEOL Co, Ltd

3.3.3 ATR/FT-IR spectrophotometer, Thermo Nicolet Nexus 670

3.3.4 Macbeth reflectance spectrophotometer, COLOR-EYE® 7000

3.3.5 Washing machine: Whirlpool, United States

3.3.6 Electrical analytical balance: Mettler Toledo, Japan

3.3.7 45 degrees flammability tester: Atlas, United States

3.4 Glassware

3.4.1 Beaker

3.4.2 Buret

3.4.3 Cylinder

3.4.4 Petri dish

3.4.5 Watch glass

3.4.6 Stirring rod

3.5 Procedure

3.5.1 Surface Modification of Fabrics Using Theta-pinch Device

Each type of fabric was cut into the required dimension, 48×26 cm. Fabric sample was then dipped in diammonium hydrogen phosphate for 5 minutes and dried in the air. After that, the fabric was wound around a sample holder. In the middle of reaction chamber of theta-pinch device, a

sample fabric was placed as shown in Figure 3.2. Before starting the process, air and old gases had to be pumped out by the vacuum pump, thus almost a vacuum level was created in the reaction chamber. Afterward, nitrogen gas was introduced into this chamber. All the treatments were performed using the following conditions shown in Table 3.1

Table 3.1 The operating conditions of theta-pinch device

Operating Conditions of Theta-pinch Device	
Pressure	2 pascal
Maximum Input	125 kA
Charging Voltage	20 kV

When the treatment completed, the fabric was taken out of the chamber and was further tested and characterized.

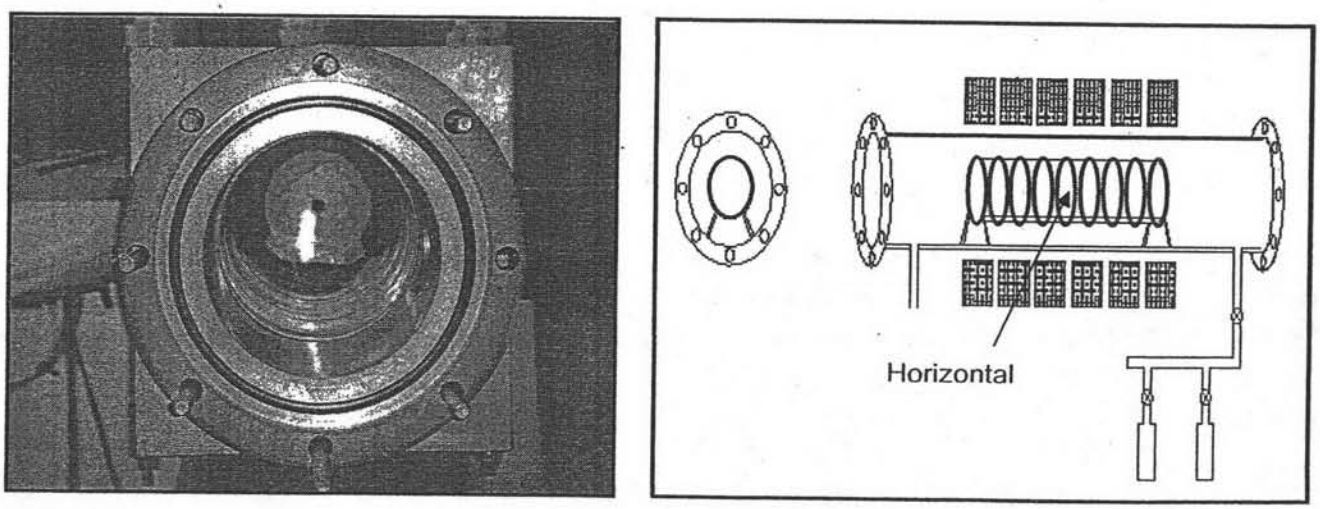

Figure 3.2 Configuration of theta-pinch device and Position of the fabric sample in chamber

In order to investigate the effect of high temperature pulse-plasma on the binding properties between diammonium hydrogen phosphate and fabrics, the parameters; especially, the concentration of diammonium hydrogen phosphate and the number of plasma shots were varied. Table 3.3 shows the conditions used for surface modification of each fabric

Table 3.3 Modification conditions used in surface modification of each fabric.

Concentration of flame retardant	Number of Plasma Shots			
5 %	1	2	5	10
10 %	1	2	5	10
15 %	1	2	5	10
20 %	1	2	5	10

3.5.2 Characterization and Testing

3.5.2.1 Wettability Measurement

In order to investigate the wettability (or hydrophilicity) of untreated and treated fabrics, a water droplet absorption time measurement was applied according to AATCC standard test method 79 (Absorbency of Bleached Textiles).

A distilled droplet was allowed to fall from a burette held 10-mm height from the stretched fabric surface. The time for the disappearance of water droplet on the surface was measured as the wetting time. The averages of wetting time at different positions on the sample surface were recorded. The studied area of the fabric was divided into 9 parts horizontally to the chamber as shown in Figure 3.3 and the wetting time of each area was determined.

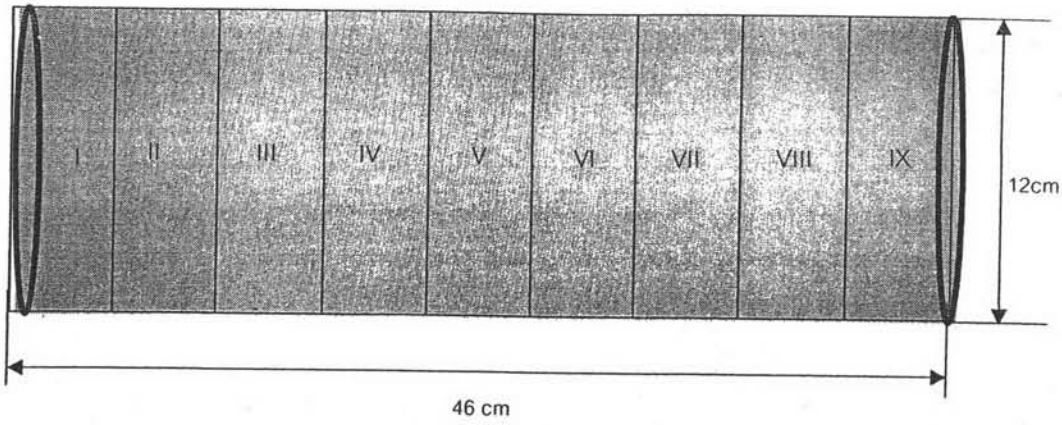

Figure 3.3 Horizontally divided areas of the fabric

3.5.2.2 Morphological Analysis

In order to observe the influence of the concentration of diammonium hydrogen phosphate and the number of plasma shots on surface morphology of plasma-treated fabrics, scanning electron microscope (SEM) (JSM-6480LV) as shown in Figure 3.4 was used to characterize the sample morphology.

All of the fabric samples were coated with thin evaporated layer of gold in order to improve conductivity and prevent electron charging on the surface before SEM analysis. The operation was at 5 keV acceleration voltages. SEM photographs were taken at different angles of view with magnification of 500X.

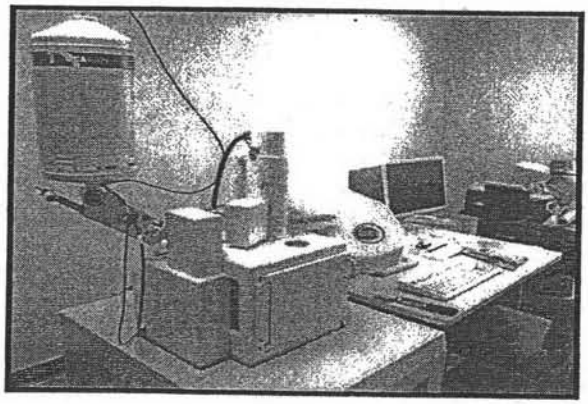

Figure 3.4. Scanning electron microscope (JSM-6480LV)

3.5.2.3 Surface Chemical Analysis

The changes in surface chemical structure of the fabric samples were characterized by using attenuated total reflection/fourier transform infrared spectroscopy (ATR/FT-IR) (Thermo Nicolet Nexus 670 spectrophotometer) as shown in Figure 3.5. The samples were scanned at the frequency range of 4000-600 cm^{-1} with 300 consecutive scans and 4 cm^{-1} resolution.

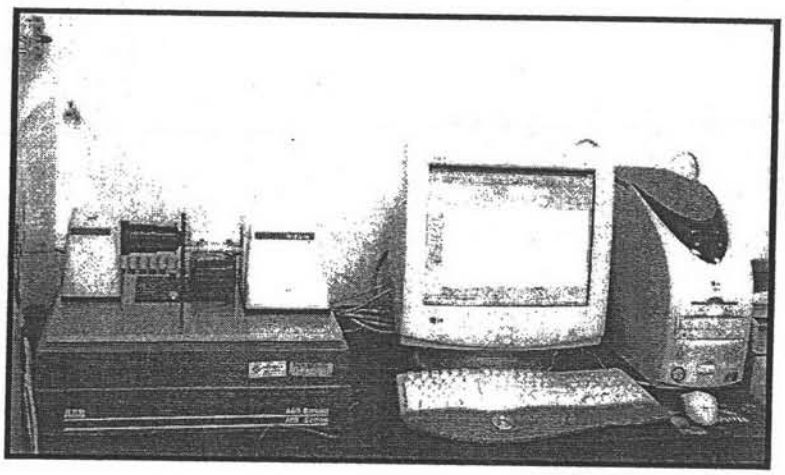

Figure 3.5 ATR-FTIR Spectrophotometer (Thermo Nicolet Nexus 670 spectrophotometer)

3.5.2.4 Whiteness/Yellowness

Whiteness and Yellowness of each fabric sample were investigated by using spectrophotometer (Macbeth reflectance spectrophotometer, COLOR-EYE® 7000) as show in Figure 3.6. Each sample was measured at five times and then the average value was calculated.

Figure 3.6 spectrophotometer (Macbeth reflectance spectrophotometer, COLOR-EYE® 7000)

3.5.2.5 Flammability Test

Flammability of untreated and treated fabrics was determined following ASTM D 1230-94. The specimens cut form untreated and treated fabrics were held in a flammability tester as shown in Figure 3.7 at an angle 45 degrees. The time required for the flame to proceed up the fabric a distance of 127 millimeters was recorded.

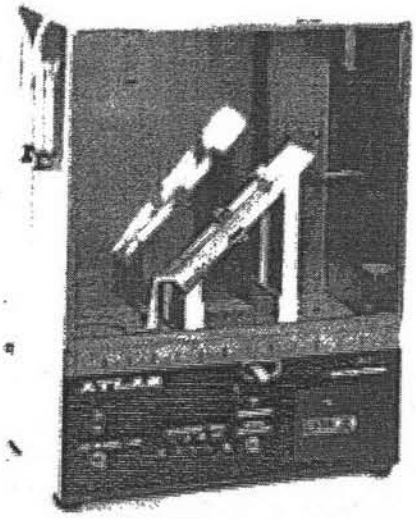

Figure 3.7 45 Degrees Flammability Tester