CHAPTER III
EXPERIMENTAL

3.1 Materials and Chemicals

A commercial raw Thai silk fiber of 55/60 D was obtained from Chul Thai Silk Co., Ltd. Standard soap without optical brightening agent (SDC reference detergent type 1, SDC enterprises limited, UK) was purchased from Sumeth Labtest Co., Ltd. Sodium carbonate (Analytical grade) was purchased from Carlo Erba Reagent. Hirus Supra Red 3BL 140 % (C.I Direct Red 80) was supplied by Phisit Intergroup Co; Ltd.

A pure oxygen gas (purity 99.999 %) was obtained from Praxair. Low pressure plasma with glow discharge type, model AH-2H was supplied by Asia Pacific Plasma Ltd., Taipei, Taiwan.

3.2 Degumming Process

3.2.1 Silk Degumming with Soap and Sodium Carbonate

Silk samples were treated with alkaline solution containing 10 g/L standard soap without optical brightening agent and 2 g/L sodium carbonate in a liquor ratio of 1:30 (silk(g):alkaline solution(mL)) at 90-95 □ C for 45 min. After that the degummed silks were washed with hot and cold distilled water and dried at room temperature (Nakpathom *et al.*, 2009).

3.2.2 Silk Degumming with Plasma

Low pressure plasma with glow discharge type, model AH-2H was used in this study (Asia Pacific Plasma Ltd., Taipei, Taiwan). Glow discharge was generated by a 13.56 MHz power source 3000 W (RF discharge). Oxygen gas (purity = 99.999 %) was used as an ionizing gas. In the plasma treatment, raw silk fiber was place in a plasma chamber horizontally on the sample plate between the two parallel electrodes. Then vacuum pump generated vacuum condition in the chamber. Once

the gas was introduced into the reactor and pressure in the chamber reached 0.20 Torr, the glow discharge was initiated, and plasma was generated. In this study the plasma treatment was operated at discharge power of 60, 250, 1000 and 2000 W, exposure time was varied at 5, 10 and 15 min with oxygen flow rate at 250, 500 and 1000 cc/min and temperature chamber was varied at 25, 50 and 75°C. Electrode distance was fixed at 3.5 cm. After finishing the treatment and releasing the chamber pressure, the sample were then taken out and kept in a zip lock bag to protect the moisture from air and avoid possible surface contamination. Degumming of silk will be performed using plasma generator at Bestrade Precision Limited, Bangkok, Thailand.

3.3 Characterizations and Testing

The degummed silk was characterized with regarding to the physical properties, mechanical and surface morphology, which were weight loss, staining with direct dyes, tensile testing and scanning electron microscopy (SEM) and X-ray diffraction (XRD).

3.3.1 Determination of Weight Loss

Degumming loss indicates the weight loss of the silk fiber after conventional or plasma treatment. The percent weight loss is calculated by Equation (1):

$$\%\ Weight\ loss = \frac{W_0 - W_1}{W_0} \qquad (1)$$

Where

W_0 =Fiber weight before conventional or plasma degumming.

W_1 = Fiber weight after conventional or plasma degumming.

3.3.2 Determination of Tensile Strength

Tensile strength of degummed silks fibers was performed according to ISO 2062 on a universal testing machine (Lloyd, model SMT2-50N) for

characterization of the mechanical properties of degummed silk. The gauge length is 250 mm ± 2 mm. All tests were conducted at a strain rate of 250 mm/min. The strength and elongation at break were obtained.

3.3.3 Fiber Surface Morphology Analysis

The quality of degumming can be qualitatively assessed by observation of the degummed fibers under a scanning electron microscope (SEM). Scanning electron microscope (SEM, Phenom FEI) operated at an accelerated voltage of 10 kV and all samples were sputtered with gold (7 nanometres in thickness) were employed to observe the surface morphology of a degummed silk samples to identify the change of surface after plasma treatment.

3.3.4 Plasma Etching Effect on Raw Silk Fiber

The X-ray diffraction (XRD) (Rigaku, SmartLab) was used to determined plasma etching effect on silk fiber. A silk sample were first pressed into a hollow of glass holder and held in place by a glass window. After that, it will be scanned in the 2θ range from 5 to 40° in the continuous mode with the rate of 5°/min. The 20 characteristic diffraction peak of raw silk fiber is about 20 □ (Long *et al.*, 2008).

3.3.5 Staining method with direct dyes

Hirus Supra Red 3BL 140 % (C.I. Direct Red 80) was used to determine the remaining of sericin after silk degumming (Silk containing serisin is dyed dark red and silk without serisin is un-dyed). The silk samples were immersed into a 1 % dye solution for 1 minute and then rinsed with distilled water. The color strength of dyed samples (K/S values) was measured by a spectrophotometer (Hunter Lab, model Color Quest XE) under illuminant D65 with a 10 □ standard observer and wavelength range 400-700 nm. The color strength of silk samples was measured at the wavelength 540 nm.