CHAPTER 2

Experiment

2.1 Preparation of Cotton Cloth

The cotton cloth was purchased from Warorod market, Chiang Mai, Thailand. The cotton cloth was washed with liquid detergent and rinsed with exceed clean water for a two time period, respectively. Then, the washed cotton cloth was dried at room temperature for overnight. The clean cotton cloth was cut to a size 2 x 6 inch square of 10 pieces for surface modification of cotton cloth supported with $SiCl_4$.

2.2 Preparation of $SiCl_4$-Treated Cotton Cloth

Parameters for preparation of $SiCl_4$-treated cotton cloth including deposition time and repeating number of reaction were investigated to form $SiCl_4$ thin film on cotton surface.

2.2.1 Determination of Deposition Time

The experimental setup for preparation of $SiCl_4$-treated cotton cloth is shown in Figure 2.1. Firstly, the cotton cloth samples were put into flask B and set within a closed system. The cotton cloth samples were dried in the flask under N_2 for 30 min by open the gas valve on the nitrogen tank and nitrogen gas flows through the pipe A, flask B, and flask C within the system and then out through the tip of the needle D. After that, the cotton cloth samples were separately exposed to $SiCl_4$ vapor via a cannula connecting flask under a steady N_2 stream at room temperature for 5, 10, 15, 20, and 30 min to determine the optimal deposition time. 10 mL of $SiCl_4$ was injected into the flask C in a closed system at various times. After reaction with $SiCl_4$, the cotton cloth samples were purged with nitrogen gas for 5 min and were then left under air.

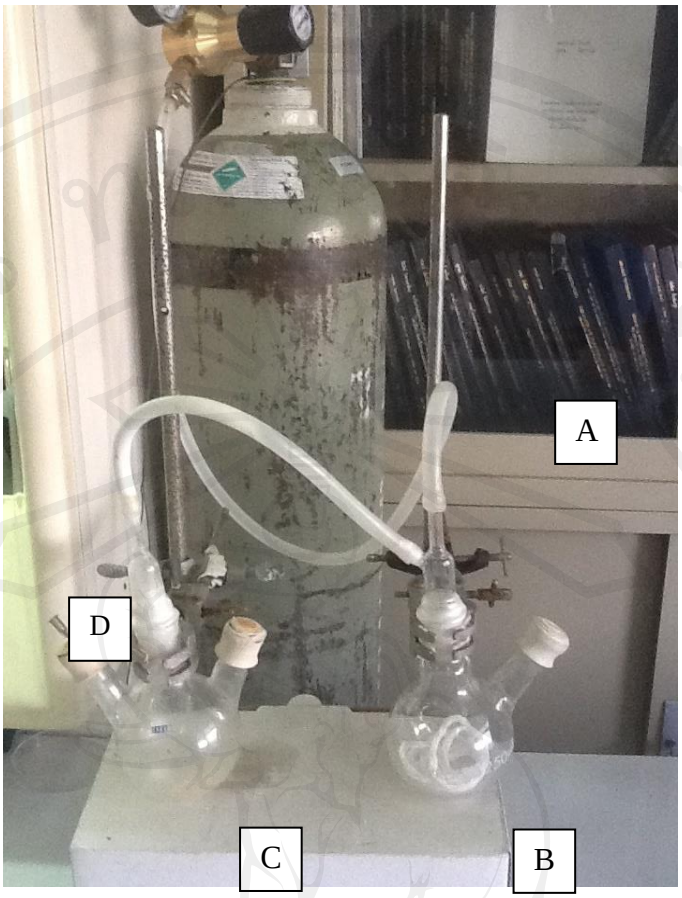

Figure 2.1 Experimental setup for preparation of $SiCl_4$-treated cotton cloth.

2.2.2 Repeating time of $SiCl_4$ Deposition

Repeating time of $SiCl_4$ deposition was performed for 0-10 times to investigate optimal amount of $SiCl_4$ on cotton surface. Similar to section 2.1, a sample was dried in a flask under N_2 for 30 min. It was separately exposed to $SiCl_4$ vapor at the optimum deposition time via a pipe connecting flask in a steady stream of N_2 at room temperature. At the end of the reaction, residual $SiCl_4$ in the flask was purged with N_2. The sample was then exposed to air for 5 min to allow hydrolysis occur. Before the repeating time of $SiCl_4$ deposition, the sample flask was pumped down under a vacuum and filled with N_2 again for 5 min prior repeating of $SiCl_4$ deposition.

2.2.3 Characterizations

The optimal repeating time of $SiCl_4$ deposition was investigated by SEM and EDS. SEM was employed to observe morphology of $SiCl_4$-treated cotton cloth and EDS was employed to determine amount of deposited $SiCl_4$ on the cotton surface with various deposition times.

2.3 Modification of Untreated Cotton Cloth and $SiCl_4$-Treated Cotton Cloth

2.3.1 1H, 1H, 2H, 2H-Perfluorooctyltriethoxysilane (POTS) Modification

Effect of POTS deposition time on degree of hydrophobicity of the modified untreated cotton cloth and $SiCl_4$-treated cotton cloth were investigated. The cotton cloth sample was placed into a sealed vessel containing 1 μL of POTS. Then, the sample in the sealed vessel was heated in an oven at 125 ºC for 10, 30, 60, 120 and 180 min. After that, the sample was exposed in the air and heated in an oven at 150 ºC. Finally, water contact angle measurements carried out to determine degree of hydrophobicity of the treated cotton cloth compared to the untreated cotton cloth.

2.3.2 Dimethyldichlorosilane (DMDCS) Modification

Effect of DMDCS deposition time on degree of hydrophobicity of the modified untreated cotton cloth and $SiCl_4$-treated cotton cloth were investigated. The cotton cloth sample was placed into a sealed vessel containing 0.5 mL of DMDCS. Then, the sample in the sealed vessel was heated in an oven at 70 ºC for 10, 20, 30, 60 and 120 min. Finally, water contact angle was measured to compare the hydrophobicity with untreated cotton cloth.

2.3.3 Stearic acid (SA) Modification

0.20 mg/L ethanolic solution containing stearic acid stock solution was prepared by dissolving 14.2 g of stearic acid in absolute ethanol adjusted the volume to 250 mL in a volumetric flask. 0.2 mg/L stock solution of SA was used for preparing 0.005, 0.01, 0.05 and 0.1 mg/L SA solution. The volume of

the stock SA solution used to prepare various concentrations is shown in Table 2.1.

Table 2.1 Volume of the stock SA solution prior adjusted to 50.00 mL in a volumetric flask with absolute ethanol.

SA concentration (mg/L)	Volume of SA 0.2 mg/L stock solution (mL)
0.005	1.25
0.01	2.50
0.05	12.5
0.1	25.0

The $SiCl_4$-treated cotton cloth was immersed in the 0.005, 0.01, 0.05, 0.1 and 0.2 mg/L ethanolic solution containing stearic scid at room temperature for 30 min and dried in the air. Finally, the water contact angle was measured to compare hydrophobicity with the other one.

2.4 Characterizations of Untreated Cotton Cloth and Treated Cotton Cloth

Many techniques were used to investigate properties of the untreated cotton cloth and $SiCl_4$-treated cotton cloth modified by POTS, DMDCS, and SA compared to untreated cotton cloth. Morphology, chemical composition, cross-linking, crystallization, and wettability were characterized by SEM and EDS, FTIR, XRD, and water contact angle measurements, respectively. All procedures are described as follows:

2.4.1 Morphology and Chemical Composition Investigation

Scanning electron microscope (SEM) is a type of electron microscope that creates various images by focusing a high energy beam of electrons onto the surface of a sample and detecting signals from the interaction of the incident electrons with the sample's surface. The instrument used in this work was a Jeol JSM-5910LV Scanning Electron Microscope.

Energy dispersive spectrometer (EDS) makes use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis. Quantitative analysis (determination of the concentrations of the elements present) entails measuring line intensities for each element in the sample and for the same elements in calibration standards of known composition.

SEM and EDS were used to investigate the morphological structure and chemical composition of $SiCl_4$-treated cotton cloth surface, compared to the untreated cotton cloth.

2.4.2 Cross-Linking Investigation

Fourier Transform Infrared Spectroscopy (FTIR) is probably the most extensively used method for the investigation of chemical structure and the analysis of functional groups. An infrared spectrum represents a fingerprint of a sample with absorption peaks which correspond to the frequencies of vibrations between the bonds of the atoms making up the material.

In this study, Bruker Model FTIR Tenser 27 Infrared Spectrometer was used to characterize the chemical functional groups of the $SiCl_4$-treated cotton cloth compared to the untreated cotton cloth.

2.4.3 Crystallization

X-ray diffractrometer (XRD) was used to investigate crystal structure of $SiCl_4$-treated cotton cloth and $SiCl_4$-treated cotton cloth modified with POTS, DMDCS, and SA compared to the untreated cotton cloth. The crystallinity of the cotton cloth samples were examined by using an XRD analyzer with CuKα as the X-ray source. The 2×2 cm^2 cotton cloth sample was put on a sample holder, and then fixed to the sample handler by masking tape. The angle degree (2θ) was varied from 20 to 80 in continuous scan mode with scan rate of 12.000 deg./min and width of the sample of 0.015 degree.

2.4.4 Wettability

The most widely used technique of contact angle measurements is a direct measurement of the tangent angle at the three-phase contact point on a sessile drop profile. The equipment consists of a horizontal stage to mount a solid or liquid sample, a micrometer pipette to form a liquid drop, an illumination source, and a telescope equipped with a protractor eyepiece. The measurement was achieved by simply aligning the tangent of the sessile drop profile at the contact point with the surface and reading the protractor through the eyepiece. This direct optical method is advantageous because of its simplicity and the fact that only small amounts of liquid (a few microliters) and small surface substrates (a few square millimeters) are required.

The cotton cloth that has been surface modified by the conditions of POTS, DMDCS, and SA were tested by contact angle of water droplets on the surface of the cotton cloth. The self-made contact angle meter captured image of 1μl of deionized water and analyzed by using image analysis software.