CHARPTER III
EXPERIMENTAL SECTION

3.1 Materials

Natural Rubber (NR) latex was provided by Ansell (Thailand) Company. Anionic sodium dodecyl sulfate (SDS) surfactant with a purity of 99 % was obtained from Aldrich. Pyrrole with purity 97% was purchased from Merck and was purified by vacuum distillation and stored in a refrigerator at about 4 °C before using. Reagent grade ammonium persulfate and sodium chloride were received from Merk, which were used without purification. Sodium hydroxide and hydrochloric acid were obtained from Carrlo Eba Reagent Company, which were used for adjusted pH.

3.2 Experimental

3.2.1 Purification of NR Latex

NR latex particles were purified by redispersing in water and centrifugation and several cycles to remove impurity such that the particles were considered to be clean.

3.2.2 Particle Size Measurement

The NR latex after purification was measured by a particle size analyzer The three sizes of lens, 45, 100, and 300 nm, are ranged in sizes of the particles, 0.1-08, 05-180, and 1.2- 600 nm respectively. The lens worked in this experiment was 45 mm for the particle in range 0.1- 80 nm. An active beam length was set to 24 nm. The latex particles were placed in a sample

cell across a laser beam. This machine analyzed the average particle size and standard size distribution from the laser beam obscuration depending on the beam length parameter. Consequently, the specific surface area was calculated from the particle diameter with the assumption of constant volume of spherical particle.

3.2.3 Electrophoretic Mobility Measurement

Electrophoretic mobility was used to determine surface charge of latex particles at various solution pH. NR colloid dispersion was diluted in 250 ml distilled water. The pH in the solution was adjusted by using NaOH or HCl, and was left to equilibrate for 1 day. The electrophoretic mobility of the latex particle was observed at room temperature with a Zeta-Meter 3.0. The electrophoretic mobility is to measure the movement of the colloidal particles dispersed in solution with applied an electric field. Figure 3.1 shows the details of the Zeta Meter which has four parts: a microscope, a temperature bath, an operating unit, and electrophoresis cell. The operating unit is an horizontal quartz cell equipped with the electrophoresis cell. The microscope is used to observe the movement of particles at fixed distance under a given voltage. The electrophoretic mobility is determined by using the microprocessor which counts the time to require the particles to move along distance at a given voltage and analyzed in the units of microns-cm/volt-seconds.

The GT-2 electrophoresis cell consists of a cathode and an anode. The molybdenum cathode is supplied for system with high specific conductance (more than 1000 micro-ohms) and the platinum anode for the low specific conductance (less than 1000 micro-ohms). A platinum cathode is operated with both specific conductances. In this experiment, the latex particles suspended in triply-distilled water varied pH in solution were placed in an horizontal quartz cell and adjusted with a platinum anode and a

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3.2.4 SDS Static Adsorption Isotherms and Pyrrole
Adsolubilization Measurement

SDS solutions (14 ml) of known concentration were adjusted to pH 3.0 with HCl. Then, the solution was added to a screw cap test tube containing latex suspension. The tubes were agitated in a shaker at 30°C while the SDS solution was allowed to equilibrate by adsorption onto latex for 30 hr. After that, a 7 ml solution of pyrrole and NaCl, whose concentration were adjusted to attain the desired final concentrations, was lowered to pH 3.0 and then added to the tube. The combined solutions were agitated at 30°C and left to equilibrate for 6 hr so that pyrrole adsolubilized completely in the SDS bilayers. The latex particles were separated from solution by centrifuged and filtered through a cellulose nitrate membrane at 0.2 μm pore size. The SDS concentrations were determined by Hewlett Packard 1050 High Performance Liquid Chromatography (HPLC). The pyrrole concentrations were analyzed by Hewlett Packard 4950 Gas Chromatography (GC).

The SDS concentrations after the removal of latex particles were determined by a Hewlett Packard 1050 High Performance Liquid Chromatography (HPLC) with an Alltech conductivity detector. The HPLC column was a Nova-Pak phenyl reserve-phase column (Waters Diversion of Millipore). The mobile phase solvents were HPLC grade solvent and triply-distilled water. In order to obtain the SDS concentration accurately, two mobile phase solvents were used sequentially. First, 20%vol methanol in water passed through the chromatography column at 1 ml/min. After 4 min, the solvent mixture was changed to 100%vol methanol at the same flow rate. Salt eluted from the first step and SDS from last step. SDS concentration was determined by using a calibration curve between conductivity versus SDS standard concentration. Samples were run in triplicate and averaged. SDS adsorption after equilibration was calculated by the difference between the

initial concentration before adsorption on latex particles and the final concentration after adsorption.

Pyrrole concentrations after the removal of the latex particles were analyzed by Hewlett Packard 4950 Gas Chromatography (GC) with a Flame ionization detector (FID). The GC column was Hewlett-Packard fused silica capillary column (HP-1 cross-linked methyl silicone) which has diameter 0.32 mm, film thickness 0.52 μm, length 25.0 m, and phase ratio 150. This column was considered to analyze the amine peak from pyrrole. The carrier gas was a helium gas. The flow rate of helium was 1 ml/min at velocity 21 cm/sec. The operating temperature of oven was 80 °C and of injection was 183 °C. The injection volume was 1 μl at split ratio 20:1. Each sample was reinjected five times to get the absolute pyrrole concentration. Pyrrole concentration after the removing of latex particles was determined by comparing the sample peak to standard solutions peak. The equilibrium pyrrole concentration was calculated by the difference between the initial concentration before adsorbed on latex particles and the final concentration after adsorbed on latex particles.

The determination of SDS static adsorption isotherms and pyrrole adsolubilization is calculated from the equation below

$$\Gamma = \frac{(C_i - C_f) \cdot V}{A} \qquad (3.1)$$

where Γ is the amount of surfactant adsorbed to the surface particles or the amount of pyrrole adsolubilization inside the SDS bilayer $(moles/m^2)$

C_i is the initial concentration of surfactant before adsorption or pyrrole before adsolubilization inside SDS bilaver (μM)

C_f is the final concentration of surfactant after adsorption or pyrrole after adsolubilization inside SDS bilaver (μM)

A is the total particle surface area of latex (m^2/ grams)

V is the volume of solution (ml)

3.2.5 Polymerization of Pyrrole onto Latex Particles

6.5 ml aqueous solution of NR latex (3.9 g of dry weight) was prepared into in three tubes followed by 6.5 ml of one of the following three solutions (i) 10 mM pyrrole; (ii) 10 mM pyrrole and 16 mM SDS; (iii) 10 mM pyrrole, 16 mM SDS and 0.6 M NaCl. These solutions were controlled to be at 30°C. After 30 min, an equimolar amount of ammonium persulfate (based on pyrrole) was added to the concentrated solution. After 4 hr. the latex was rinsed with distilled water and dried at 60°C under vacuum oven. The morphology of the films was observed by optical microscope.

3.2.6 Conductivity Measurement

The films were placed between two electrodes which had titanium attached with platinum. The voltage from power supply was applied to the electrodes. The current was measured by a Picoampmeter. The composition of this measurement is shown in Figure 3.2. Resistance (R) of the films was calculated using equation 3.2 by the relationship between current and voltage. The dimension of the film sample was measured, so the conductivity can be known using equation 3.3.
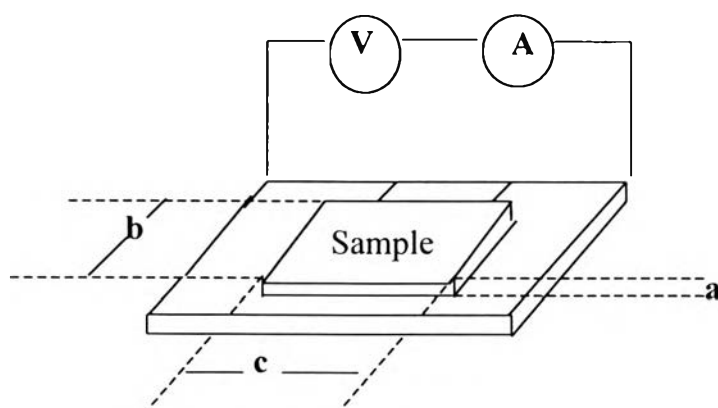
Figure 3.2 Geometry of conductivity measurement.

$$R = \frac{V}{I} \tag{3.2}$$

$$\rho = \frac{1}{\sigma} = \frac{Rab}{c} \tag{3.3}$$

where

R	is	the resistance (W)
V	is	the voltage (volt)
I	is	the current (A)
ρ	is	the resistivity (Ω.cm)
σ	is	the conductivity (Scm^{-1})
a	is	the film thickness (cm)
b	is	the film widht (cm)
c	is	the film length (cm)