CHAPTER III

EXPERIMENTAL PROCEDURE

3.1 Materials

3.1.1 Tapioca Starch

Tapioca Starch was supplied by Siam Modified Starch Co., Ltd.

3.1.2 Hydroxypropylating Agents

Commercial grade of propylene oxide from Siam Modified Starch Co., Ltd. was used as hydroxypropylating agent. Sodium hydroxide, sodium sulfate, hydrochloric acid, ethanol (analytical grade, Merck) and Deuterium oxide (Aldrich) were used as a basic material for modification.

3.2 Instruments

The major instruments used are listed below.

1. Nuclear Magnetic Resonance (NMR) Spectra BRUKER ACF 200 MHz
2. Hewlett Packard General Spectrophotometry.
3. Viskograph Brabender 486044 Brabender OHG Duisburg.
4. Sartorius Moisture Analyzer MA 30
5. JEOL JSM 5800LV Scanning Electron Microscope

3.3 Experimental Procedure

3.3.1 Preparation of Hydroxypropylated Tapioca Starch

The native tapioca starch (100 g, dry basis) was added into water with vigorous agitation. After that, solution of sodium hydroxide (3.5%w/v) was added dropwise. The slurry was mixed and warmed to 40°C in a water bath. Then propylene oxide was slowly added. The reaction mixture was stirred at 40°C for 24 hrs. The reaction mixture was neutralized with 1:1 ratio of HCl:H_2O and filtered. The modified starch was thoroughly washed with water and dried at 50°C for 6 hrs.

3.3.2 Determination Degree of Substitution by ^{1}H-NMR

Dry hydroxypropylated tapioca starch (0.01 g) was weighed accurately into a tube and D_2O (2 ml) was added. Vigorous shaking with heat resulted in a clear solution. The solutions were the transferred into NMR tube. Spectra were obtained from 200 MHz Bruker ACF Spectrometer.

3.4 Examination of Physical Properties

3.4.1 Viscosity Measurement

The Viscograph Brabender Model 486044 was used to determine the pasting properties of starch sample. Hydroxypropylated tapioca starch 6% by dry starch weight and 500 g of distilled water were combined and stirred in the aluminium sample canister. A programmed heating and cooling cycle was used, where the sample was heated to 50°C and heated again to 95°C in 30 minutes, held at 95°C for 30 minutes, cooled to 50°C in 30 minutes and the held at 50°C for 10 minutes. Finally, the viscograph was performed.

3.4.2 Moisture Determination

The moisture content of hydroxypropylated starch was determined by Sartorius moisture analyzer. The sample (2-3 g) was exposed to heat at 100°C until the constant weight was obtained. Moisture of starch was expressed as percent moisture in starch.

3.4.3 Starch Morphology

The SEM at 15 kV accelerating voltage, and detected the electron with backscattered electron emission detector examined the morphology of the hydroxy-propylated tapioca starch.