

CHAPTER III EXPERIMENTAL

3.1 Materials

Montmorillonite(Na-MMT) with cation exchange capacity of 119 meq/100 g was supplied by Kunimine Industrial Co., Ltd., Japan. Di(hydrogenated tallow)dimethylammonium chloride was kindly supplied by Unilever Thai Holdings Co., Ltd. Poly(ethylene oxide) $M_v \sim 100,000$ was purchased from Aldrich Chemical Company. The chemicals for modification of Na-MMT and organic solvent adsorption test were purchased from either Fluka or Aldrich Chemical Company. Poly(amidoamine), PAMAM dendrimer with second generation in methyl alcohol (20 wt%) was purchased from Aldrich Chemical Company. All of the chemicals used in this work are used as received without further purification.

The chemical structure of di(hydrogenated tallow) dimethylammonium chloride and octadecylamine were shown in Figure 3.1.

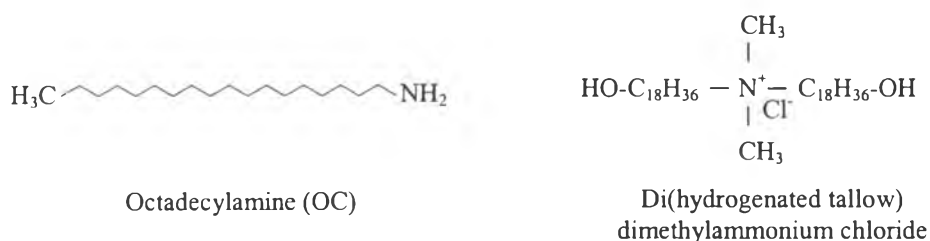


Figure 3.1 Chemical structure of modifying agents.

3.2 Equipment

3.2.1 Atomic Adsorption Spectrometer (AAS)

Atomic adsorption spectrometer is a particular instrument used to determine sodium ion exchange percentage of Na-MMT. A Varian SpectrAA-300 was applied using acetylene as fuel and air as supported gas. The sodium-hollow cathode lamp with 5 mA was chosen to produce the wavelength at 589.0 nm. All of standard solutions were prepared by dilution technique from the 1000 ppm-

concentrated NaCl solution, and the standard curve was created for Na⁺ determination (See Appendix A).

3.2.2 Fourier-Transform Infrared Spectrometer (FT-IR)

FT-IR was used to determine functional groups of organic substances. The spectra were obtained from a BRUKER Equinox 55 spectrometer with 16 scans at a resolution of 4 cm⁻¹. A deuterated triglycinesulfate detector (DTGS) with a specific detectivity, D*, of 1×10⁹ cm.Hz^{1/2}.W⁻¹ was used to measure intensities within the frequency range of 4000-400 cm⁻¹. KBr pellet technique was applied in the preparation of samples.

3.2.3 Thermogravimetric Analyzer (TGA)

A DuPont TGA was employed to observe degradation temperature of materials used. The chamber was continuously flushed with nitrogen gas at the flow rate of 20 ml/min. The temperature range was between 30-800°C and the heating rate was set at 10°C/min.

3.2.4 Wide-angle X-ray Diffractometer (WAXS)

XRD spectra were recorded by using a D/MAX-2000 series of Rigaku/X-ray Diffractometer that provides X-ray of Cu K-alpha at 40 kV/30 mA. The glass sample holders were applied to both ground samples and composite sheets. The experiment was operated in the 2θ range of 2-30 degree at the scan speed of 5 degree/min and 0.02 degree of scan step.

3.2.5 Capillary Viscometer

The viscosity of PEO/MMTs and PAMAM/MMT nanocomposites in aqueous solution were determined by using Cannon Ubbelohde-type number 50 of capillary viscometer. The viscosity of the nanocomposites solution were measured by the time taken to flow between fixed marks in a capillary tube under the draining effect of gravity. The relative viscosity is actually measured first, by dividing the viscosity of solution by that of the solvent as following.

$$\eta_{\text{rel}} = \frac{\eta}{\eta_0} \approx \frac{t}{t_0} \quad (3.1)$$

where:

η = viscosity of the solution

η_0 = viscosity of the pure solvent

t = time taken to flow of the solution

t_0 = time taken to flow of the pure solvent

and the specific viscosity is defined as

$$\eta_{\text{rel}} - 1 = \left(\frac{\eta - \eta_0}{\eta_0} \right) = \eta_{\text{sp}} \quad (3.2)$$

3.2.6 Dynamic Light Scattering

The particle sizes of nanocomposites in aqueous solution were measured by using Photon Correlation Spectroscopy (PCS) of MALVERN instrument. The measurement was carried out at 30°C with pin hole 100 and a single angle (90°).

3.2.7 UV/VIS Spectrometer

The amount of remaining organic solvent in wastewater after treated with the nanocomposite was determined by using Lambda 10 UV/VIS spectrophotometer of Perkin Elmer.

3.2.8 Transmission Electron Microscope (TEM)

The structure of layer silicate clay/polymer nanocomposite was observed by using TEM from the Center Laboratory Instrument of Chulalongkorn University. Microtomed thin section with polymer resin was the method used to prepare the specimens.

3.3 Methodology

3.3.1 Preparation of Organically Modified Montmorillonite

Na-MMT (10 g) was stirred for 2 hours in 300 ml of distilled water and heated up at 80°C for half an hour. The solution of 1.5 equivalent alkylamine and 3 equivalent of HCl was heated at 80°C at the same time as Na-MMT suspension for half an hour to give an alkylammonium solution. Then, it was gradually added into the Na-MMT suspension (at 80°C) for another 2 hours with vigorous stirring. The sediment of organically modified MMT was collected by filtering and washing with 2 L of hot distilled water. The product was dried overnight at 80°C, ground with mortar and was kept in a bottle.

3.3.2 Preparation of PEO/MMT Nanocomposites

PEO/MMT nanocomposites were accomplished by heating PEO with MMTs at 80°C. Both components were thoroughly mixed in an agate mortar, formed into a pellet using hydraulic press and a pressure of 70 MPa, and heated at 80°C in ambient atmosphere for 2 hours. The amount of MMTs was varied between 1-60%.

3.3.3 Preparation of PAMAM/MMT Nanocomposites

PAMAM/MMT nanocomposites were prepared by introducing 10 wt% of MMTs to PAMAM dispersed in methyl alcohol for MMT and 1-butanol for OC-MMT and OH-MMT. The mixture was stirred in air at 50°C with a magnetic stirrer for two hours. After dried in air for two days at 80°C, the resulting viscous gel was transferred to a vacuum oven at 110°C for another one day.

3.3.4 Adsorption Test

The nanocomposites were applied to 100 ml of wastewater with 10 v% of several organic solvents (toluene and xylene) varied in amount of the nanocomposites. The mixture was stirred for 1 day to allow equilibrium to be achieved and the aggregate was isolated from the slurry by centrifuging at 10,000 rpm

for 10 minutes. The organic fraction was extracted by using n-hexane for toluene and cyclohexane for xylene and the adsorbate concentration was analysed by using UV\VIS spectrometer. The amount of organic adsorbate uptake was then calculated by using mass balance equation.

Table 3.1 UV maximum wavelength (λ_{\max}) of adsorbate studied

| Adsorbate | λ_{\max} (nm) |
|-----------|-----------------------|
| Toluene | 207 |
| Xylene | 218 |