CHAPTER III
EXPERIMENTAL

3.1 Materials

Nata de coco was purchased from local food market. Polyamide11 (PA11) was purchased from Sigma-Aldrich Co., Ltd. Formic acid (AR grade, 99%) (AnalaR NORMARPUR) and sodium hydroxide (NaOH) (AR grade, 98%) were purchased from S.M. Chemical Co., Ltd.

3.2 Experimental Procedures

3.2.1 Bacterial cellulose preparation (Thi Thi Nge *et al.*, 2010)

Bacterial cellulose was extracted from nata de coco. The nata de coco was first washed with water to remove some excess sugar and blended using laboratory blender. Then treated with 0.1 M NaOH at 80°C for 1 hour to reduce the size to be BC pellicles and remove any remaining microorganisms, medium component and soluble polysaccharides. The BC pellicles were then washed with distilled water until reach neutral pH. The BC pellicles were dispersed in D.I. water and kept in 2 liter bottle.

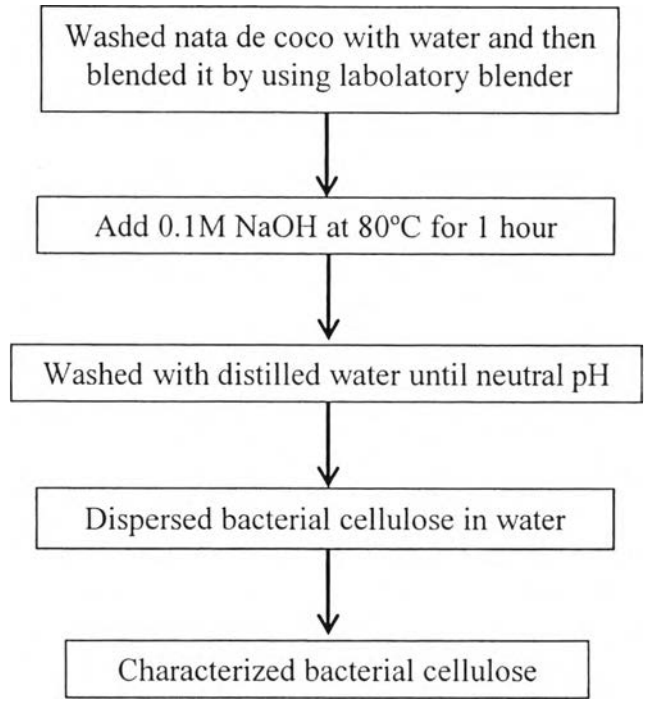

Figure 3.1 Bacterial cellulose preparation

3.2.2 PA/BC Preparation

Polyamide pellets were dried in oven at 70°C for 24 hours, and then the pellets were dissolved in formic acid. The concentration of the obtained solution was in the range of 4-25wt% (Yanhuai *et al.*, 2009). The polyamide solution is stirred at 80 °C for PA11 and did not apply heat for PA6 and stirred it until polyamide fully dissolved in formic acid. The various percent weight ratio of BC (0.2, 0.4, 0.6, 0.8, 1wt% for PA11 and 1wt% for PA6) in formic acid was mixed with PA solution and stirred it. After that, the solution will be dropped into cool water and waited for 10 minutes. After that, they are heated in vacuum oven at 70 °C for 2 days, the blend will be obtained. Scanning electron microscopy will be used to determine the morphology of pure films. Fourier transform infrared spectroscopy is used to determine the residual solvent and miscibility between polyamide and BC. Differential scanning calorimetry is used to determine the transition temperature and amount of crystalline. X-ray diffraction spectroscopy is used to determine the presence of amount crystallinity of polyamide films. Thermal gravimetric analysis is used to determine the thermal properties of PA films.

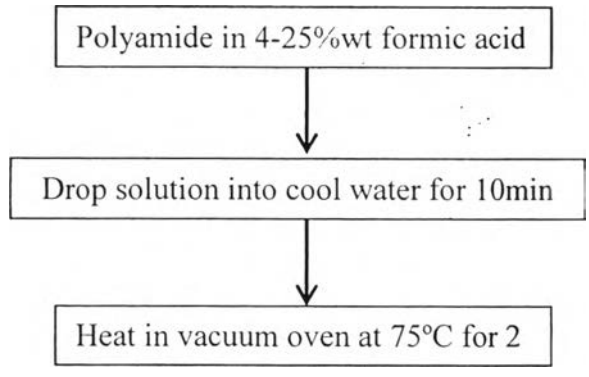

Figure 3.2 Polyamide preparation.

3.3 Characterization and Testing

3.3.1 Compression Molding Machine (Labtech, model LP20)

Film samples are preformed by a compression press (Labtech, model LP20) with preheating 5 min, follows by compressing 10 min at the force of 5 Tons. The operating temperatures of mold are maintained at 240 °C and 220 °C for PA6 and PA11, respectively.

3.3.2 Universal Testing Machine

The tensile properties of the blend films were investigated by universal testing machine (Lloyd) with 500 N of load cell. The tensile testing was followed the ASTM D882. Gauge length was fixed at 5 cm with the speed test at 5 mm/min. Samples were prepared into 10 cm x 1 cm with >300 μm of thickness.

3.3.3 Corona Discharger

Film samples are cut into square shape(1.5 x 1.5 cm^2). Then corona poling at the highest voltage input to achieve the highest dielectric and piezoelectric properties at 120 °C for 20 minutes.

3.3.4 Differential Scanning Calorimeter, DSC7

Heating profiles of PA films, PA/BC films were performed by a differential scanning calorimeter 7, DSC7 (Perkin Elmer) with a heating rate of 10 °C/min. Ten milligrams of samples were heated in aluminium pan from 0 °C to 300

°C. Differential scanning calorimetry, DSC is used to determine the crystallization temperature (T_c) and crystalline melting temperature (T_m) for the dynamic phase diagram. The degree of crystallinity will be measured as the ratio between ΔH_m and ΔH_m^0, as below equation.

$$X_c = \frac{\Delta H_m}{\Delta H_o(1-\alpha)} \quad (3.1)$$

Where α is fiber weight content, ΔH_o is the melt enthalpy for 100% crystalline, when ΔH_o of PA6 is 230.1 J/g and ΔH_o of PA11 is 206 J/g.

3.3.5 Thermal Gravimetric Analyzer, TGA

Thermal degradation is performed by a high resolution TG-DTA Pyris Diamond (Perkin Elmer). Sample are loaded on the alumina pan heated from 50 °C to 900 °C with a heating rate of 10 °C/min under N_2 flow.

3.3.6 Fourier Transform Infrared Spectrometer, FT-IR

FTIR was performed on using a Thermo Nicolet, model NEXUS 670. The absorption peak was recorded over wavenumbers region of 4000-400 cm^{-1} by taking 64 scans with a resolution of 4 cm^{-1} using standard KBr tablet samples. To prevent humidity effect, samples and KBr were dried in an oven at 60°C.

3.3.7 UV/Visible Spectrophotometer (UV-2500)

The transmission of film will be observed by UV/visible spectrophotometer, UV/vis. The films samples are cut into 1.5x3 cm^3 and analyzed by using UV/Visible spectrophotometer (Shimadzu Scientific Instrument, model 2550)

3.3.8 Scanning Electron Microscope, FE-SEM

The structure and morphology on surface, cross-sections of film will be observed by scanning electron microscopy, FE-SEM. It operated at 2 kV. Cross-section samples were prepared. The fracture films were obtained by soaking the samples in liquid nitrogen for 5 min, then break the samples immediately at room temperature. The prepare samples were sputtered with platinum for 150 s. The films samples are platinum sputtered and analyzed by using a scanning electron microscope.

3.3.9 X-ray Diffraction Microscope, XRD

Crystal phase and structure of PA films were analyzed by using a Rigaku, SmartLab XRD with Ni-filtered CuKα radiation operated at 40 kV and 30 mA. The samples were scanned at 2θ ranging from 10° to 70° with a scanning speed of 5 °C/min.

For PA6, there were 2 types of stable crystalline forms, monoclinic α-crystalline form and monoclinic γ-crystalline form. The peak appeared at around 2θ = 20° and 24.8 ° were α-crystalline form which was (200) and (002) plane respectively. The peak at around 2θ = 21.4 ° was γ-crystalline form.

For PA11, there are 3 peaks, the left peak is alpha form, and It can be seen the two strong reflections (100) and (010,110) at the diffraction angle 2 theta of 20.02 and 23.018, which were characteristic of the triclinic α -form.

3.3.10 Dielectric Measurement

Dielectric properties of PA film and PA/BC blend films will be measured by impedance/gain-phase analyzer (Hewlett Packard., model 4194A and Agilent., model E4980A) in parallel capacitance (Cp) mode, with frequency from 1 kHz to 10 MHz at room temperature and measured by impedance/gain-phase analyzer (Agilent., model E4991A) with 10 MHz to 1 GHz and -20 °C to 150 °C. The dielectric constant of materials is calculated from the capacitance by using below equation:

$$\varepsilon = \frac{Cd}{\varepsilon_0 A} \tag{3.2}$$

Where C is the capacitance (F), ε_0 is the free space dielectric constant value (8.85×10^{-12} F/m), A is the capacitor area (m^2) and d is the thickness of specimens.