

CHAPTER III

EXPERIMENTAL

3.1 Materials

All chemicals were used without further purification. Polypropylene (PP) and Maleic anhydride grafted polypropylene (MAPP) were purchased from Global Chemical Trading Co., Ltd., Wood flour (WF) was purchased from V.P. Wood Co., Ltd., Hexadecyl-trimethyl-ammonium bromide (CTAB) was purchased from S.M. Chemical Supplies Co., Ltd., and bentonite was supported from Thai Nippon Chemical Industry Co., Ltd., Thailand.

3.2 Equipment

1. Scanning Electron Microscope or SEM (Hitachi S-4800)
2. Universal Testing Machine (Shimadzu DSS-10T)
3. Pendulum Impact Tester (Zwick 5113)
4. X-ray Diffractometer or XRD (Rigaku TTRAX III)
5. Twin Screw Extruder (Collin T-20)
6. Thermogravimetric Analyzer or TGA (Perkin-Elmer TGA7)
7. Compression Molding Machine (Wabash)
8. Injection Molding Machine (Battenfield BA250)

3.3 Methodology

3.3.1. Preparation of nanoclay, wood flour and PP/MAPP blends

The bentonite clay received was used without further purification and the ions contained in the clay are shown in table 3.1. The clay (300 g.) was dispersed in 1 liter solution of water and ethanol at ratio 4:1. The mixture is stirred overnight at room temperature. The 0.5%w – 3.0%w of CTAB was slowly added in the 50 ml solution of water and ethanol at ratio of 1:1 and stirred vigorously until the CTAB is completely dissolved. The solution is slowly poured into the clay mixture and stirred for 12 hour at 80° C to retrieve the organoclay. Then, the organoclay was filtered and washed several times with distilled water and dried in oven overnight at 100° C. The retrieved organoclay series were label as Na-Ba for untreated bentonite clay and Ba-

CTAB 0.5, Ba-CTAB 1.0, Ba-CTAB 1.5, Ba-CTAB 2.0, Ba-CTAB 2.5 and Ba-CTAB 3.0 for 0.5%w – 3.0%w of CTAB treated organoclay respectively. X-ray diffraction was used to measure the effectiveness of organic treatment.

Wood flour was used without further modification and dried in oven overnight at 80° C before used.

Maleic anhydride grafted polypropylene was used without further purification and dried in oven overnight at 100° C before used. The blends of PP and MAPP were prepared by twin screw extruder. The mixing ratios of MAPP: PP was 0%, 3%, 6% and 9% by weight, respectively. All of the extruded compounds were pelletized by rotating blade.

3.3.2 Compounding of the organoclay and wood flour

Polypropylene, WF and nanoclay were weighed and bagged. The content of WF with PP/MAPP blends were fixed at 30: 70 % by weight. All the components were finely mixed and processed in a COLLIN twin screw extruder (Model T-20, Germany) with the temperature profile as follows; 160-170-170-175-175-180-180-180-185-185°C and screw speed of 35 rpm. The obtained organoclay-plastic composites were pelletized and stored in dried containers.

3.3.3 Preparation of specimens

The specimens were prepared by BATTENFIELD injection molding machine (Model BA 250, Austria). The temperature profile was set at 190-200-210-200° C and the maximum injection pressure was 65 bars. The mechanical properties were studied at room temperature and 50% of relative humidity.

Table 3.1 Chemical Compositions of bentonite clay

Ions	% by weight	Ions	% by weight
SiO ₂	70%	MgO	3%
Al ₂ O ₃	17%	CaO	2.5%
Fe ₂ O ₃	2%	K ₂ O	0.8%
Na ₂ O	2.5%	TiO ₂	0.3%

3.4 Characterizations and Testings

3.4.1 Characterizations

3.4.1.1 X-ray Diffractometer

Small angle X-ray Scattering (SAXS) analysis was carried out with a Rikaku DMAX 2200 HV/X-Ray with CuK α radiation ($\lambda=1.54$ nm, 18 kW) at room temperature. Scanning rate was 1°/min. The obtained modified bentonites at various ratios were dried and grounded into fine powder, later on, they were put on glass holders and placed in the instrument. The angle used for measurement started from 0.5° to 10°.

The composite pellet obtained from compounding was pressed into a film by Wabash compression molding machine. Compress pressure was 10 bar. The film thickness was approximately 0.1-0.25 mm. The films were cut in the size of 1 cm in height and width, then, put on the glass holders and placed in the instrument. Scanning rate was 1°/min and the angle used for measurement started from 0.5° to 10°.

3.4.1.2 Tensile Properties

Tensile testing was carried out according to the ASTM 3500 standard with Shimadzu DSS-10T universal testing machine. The specimens were prepared in dumbbell shape with approximate thickness and width of 3.2 and 12.7 mm respectively.

3.4.1.3 Flexural Properties

Flexural testing was carried out according to the ASTM D790 standard with Shimadzu DSS-10T universal testing machine. The specimens were prepared in approximate thickness and width of 3.2 and 12.3 mm respectively.

3.4.1.4 Impact Strength

Impact strength measurement was carried out according to ASTM D256 standard. The specimens were prepared in approximate thickness and width of 3.2 and 12.3 mm respectively. All the samples were notched on the center of one longitudinal side and each sample, five replications were tested.

3.4.1.5 Thermal Properties

The thermal behavior was investigated by Thermogravimetric Analyzer (TGA), Perkin Elmer TGA7. The sample were prepared by weighting the sample of 5-10 mg and put into the platinum pan. The measurement was carried out by heating the sample under nitrogen flow with the heating rate of 20° C/min from 30° C to 600° C.

3.4.2 Water Absorption

The experiment was carried out by preparing the specimens approximate dimension of 6.35×1.27×0.27 cm³. The specimens were measured the constant weight and then immersed into a static deionized water bath at 70°C and held in water for 45 days.

Water absorption (WA) was calculated according to the following fprmular:

$$WA (\%) = [(W_e \times W_o) / MW_o] \times 100$$

Where W_e is the weight of the sample after immersion (g); W_o is the weight of the sample before immersion (g).