

CHAPTER 3

MATERIALS AND EXPERIMENT

3.1 Materials

Low Density Polyethylene (LDPE) JJ4324 grade
(Thai Petrochemical Industry Co., Ltd)

Tapioca Starch
(Nansurakij Co., Ltd)

Zinc Stearate $\text{Zn}(\text{C}_{17}\text{H}_{35}\text{COO})_2$
(Coin Chemical (Thailand) Co., Ltd)

Glycerol Monostearate ($\text{C}_{21}\text{H}_{42}\text{O}_4$)
(Imperial Industry Chemicals.(Thailand) Co., Ltd)

Epolene E-43p wax
(White Groups Co., Ltd)

3.2 Apparatus

Two-Roll Mill
Labtech Engineering Co., Ltd Model LRM-110

Injection Molding
ENKEL Co., Ltd. Model DTE 26

Instron Universal Testing Machine
LR-10K with RCONTROL software

Scanning Electron Microscope (SEM)
JOEL Model JSM-T220A

Particle Size Analyzer
Marvern Co., Ltd. England Model Master Seizer S

3.3 Sample Preparation

3.3.1 Preparation of pre-mix

Tapioca starch was mixed with low density polyethylene (LDPE) in several proportion as indicated. Epolene or glycerol monostearate or zinc stearate was added to the mixture at 1% by weight of the mixture, when it was needed. Then the mixture was manually mixed for 5 minutes.

3.3.2 Blending materials

A two roll mill compounding machine, Labtech Engineering Co., Ltd., Model LRM110, was used. The temperature of front roll and back roll were set at 145 and 140°C, respectively. After 30 minutes, the setting temperature of roll were reached. The polyethylene resin was pored in between the rolls and allowed to warm for 5 minutes. The rolls were started to turn while the polyethylene pre-mix was melting. Tapioca starch or corn starch with or without the compatibilizer was gradually added into the molten polyethylene. In order to have a good homogeneous mixture, a trowel was used during

mixing . The mixing time was 10 minutes . It was then and let it cool to room temperature. Finally, it was crushed into small pieces. In the case that the compatibilizer, epolene or glycerol monostearate or zinc stearate, was used, it was added at 1% by weight of total mixture of starch and LDPE and mixed manually with starch .

Table 3.1, 3.2 and 3.3 show the composition of each blend. The sample is coded by P/T or P/C which stands for .

3.4 Specimens Preparation

Each blend was injection molded into 3.3 mm. thickness test specimens according to ASTM D 638-93 by ENKEL DTE-26 molding machine . All samples were molded at the melt temperature of 170 °C. The injection speed of 190 , 180 , 175 , 170 and 160 mm/s respectively, a holding pressure of 102 bar , holding time of 5.0 s and a cooling time of 15.0 s . The total cycle time was about 25 s for all blends . The blend containing 5% tapioca starch with epolene or glycerol monostearate or zinc stearate and without compatibilizer was also using none-made film blowing machine at King Mongkut Institute of Technology North Bangkok. The temperature of feed, compression, metering and die zone were 140 , 150 , 160 and 150 °C, respectively .

Table 3.1 The composition of LDPE/tapioca starch blends

| Sample Code | LDPE (g) | Starch (g) |
|-------------|----------|------------|
| P/T00 | 500 | 0 |
| P/T05 | 475 | 25 |
| P/T10 | 450 | 50 |
| P/T15 | 425 | 75 |
| P/T20 | 400 | 100 |

P = Low Density Polyethylene

T = Tapioca starch

00 , 05 , 10 , 15 and 20 = % starch content by weight

Table 3.2 The composition of LDPE/tapioca starch blends with compatibilizers

| Sample Code. | LDPE (g) | Starch (g) | E(g) | Z(g) | G(g) |
|--------------|-----------|------------|------|------|------|
| P/T00E | 495 | 0 | 5 | - | - |
| P/T05E | 470 | 25 | 5 | - | - |
| P/T10E | 445 | 50 | 5 | - | - |
| P/T15E | 420 | 75 | 5 | - | - |
| P/T20E | 395 | 100 | 5 | - | - |
| P/T00Z | 495 | 0 | - | 5 | - |
| P/T05Z | 470 | 25 | - | 5 | - |
| P/T10Z | 445 | 50 | - | 5 | - |
| P/T15Z | 420 | 75 | - | 5 | - |
| P/T20Z | 395 | 100 | - | 5 | - |
| P/T00G | 495 | 0 | - | - | 5 |
| P/T05G | 470 | 25 | - | - | 5 |
| P/T10G | 445 | 50 | - | - | 5 |
| P/T15G | 420 | 75 | - | - | 5 |
| P/t20G | 395 | 100 | - | - | 5 |

E = Epolene E-43 p

Z = Zinc stearate

G = Glycerol monostearate

Table 3.3 The composition of LDPE/corn starch blends with compatibilizers

| Sample Code | LDPE (g) | Starch(g) | Z(g) | G(g) |
|-------------|------------|-----------|------|------|
| P/C00Z | 495 | 0 | 5 | - |
| P/C05Z | 470 | 25 | 5 | - |
| P/C10Z | 445 | 50 | 5 | - |
| P/C15Z | 420 | 75 | 5 | - |
| P/C20Z | 395 | 100 | 5 | - |
| P/C00G | 495 | 0 | - | 5 |
| P/C05G | 470 | 25 | - | 5 |
| P/C10G | 445 | 50 | - | 5 |
| P/C15G | 420 | 75 | - | 5 |
| P/C20G | 395 | 100 | - | 5 |

C = Corn starch

3.5 Measurement

3.5.1 Mechanical Testing

An instron universal testing machine MODEL LR-10K with RCONTROL software was used to evaluate tensile strength and elongation at break in the machine direction. Five specimens of each blend were tested for one measurement by taking the average of five. The thickness of specimens were measured randomly at 5 points with micrometer to nearest at 0.001 mm. The test condition was at 27 ± 2 °C and the cross head speed of testing machine was at 25 mm/min .

3.7.2 Scanning Electron Microscopy (SEM)

JOEL MODEL JSM-6400 (TOKYO , JAPAN) scanning electron microscope was used operating at 15 KV and 80 μ A . The magnification used range from 250X to 1000X . SEM coating unit MODEL JPC-1100 was used to coat the specimens with thin layer of 100% gold (Au) under vacuum for 20 minutes .