Studies on Compatibility of Polymer Blends between Poly (trimethylene terephthalate) and Polyamide 4 Prepared by Melt Blend Technique

P. Sirisangsawang and S. Pivsa-art

Abstract— Preparation of polymer blends between Poly (trimethylene terephthalate) (PTT) /Polyamide 4 (PA 4) ratio at 50:50, 60:40, 70:30 and 80:20 %wt with Haake rheomix. The results indicated that the polymer blends are less viscosity and rapid flow. Mixing of polymer blends are difficult and can't be compressed by compression mold machine. Another melt blend technique, twins screw extruder was used replace to Haake rheomix. Twins screw extruder was used to prepare PTT /PA 4 at the ratio of 95/5, 90/10, 85/15 and 80/20 % wt. The effect of additive blend (bond fast) 1, 2, 3, 4 and 5 %wt was studied. The thermal properties of PTT and PA4 by using DSC showed the melting temperatures were 229 and 214.1 ℃, respectively. The polymer blends at different ratios with the melting temperature in the range of 214-229 ℃. The morphological were test with scanning electron microscopy found that the ratio of PTT/ PA 4 at 95/5 is the most compatible. The effect of additive will increased the compatibility of polymer blends. The quantity of bond fast was not effect on physical properties. However, all of polymer blends were still brittle and could not forming part to test the mechanical properties.

Keywords— Poly (trimethylene terephthalate), Polyamide 4, Polymer blends, Biodegradable Polymer

1. INTRODUCTION

The conventional plastics from petroleum have been widely used, which has impact on environment problem. Therefore, biodegradable polymers have received more attention and used to replace conventional plastics due to it is friendly. The biodegradable polymers can be produced from natural polymers, synthesis polymer or microbial fermentation [1]

Poly (trimethylene terephthalate) (PTT), a new aromatic polyester, it is a biodegrable polymer and was commercialized. PTT consists of 1,3-propanediol chain and aromatic ring of terepthalic acid . T_m and T_g of PTT were 228 and 50 °C, respectively [2]. Moreover PTT is also strong, tolerance to UV, and flexibility in molding.

Polyamide 4 was synthesis from 2-pyrrolidone by ring-opening polymerization. It becomes attractive because it is susceptible to biodegradable in composted oil [3]. Furthermore, polyamide 4 has resistance thermal, high melting temperature and high tensile strength (100 MPa). Kawasaki et.al studied to improvement properties polyamide 4 with branched initiator it was degraded 41% in an activated sludge [4].

Blending polymer has emerged to manufacturing new materials with specific properties. Most commercial polymer blends are designed to improvement of the parent components, including better processability, higher impact strength, better chemical resistance. PTT can join with the other polymer such as PBT, polycarbonate, poly (ethylene naphthalate) [5,6,7]. Blend PTT with polyamide 12 were investigated and found to be miscible showing a phase-separated morphology [2]

In this research, PA4 which biodegradable polymer but it is difficult to forming was blending with PTT, a high strength polyester and biodegradable as review. The melt blend technique was investigated for blending PTT/PA4 and the compatibilizer was added to improve the compatibility. This paper purposed to prepare new biodegradable materials and use this research for basic data for preparation of biodegradable polymer.

2. MATERIALS AND METHOD

Materials

All the materials of this study were analytical grade without any treatment. Polyamide 4 was synthesis by the method of Kawasaki et. al. as follow. PTT and PA4 were dried at 60 °C for 4 hours before prepared polymer blend.

Synthesis of Polyamide 4

2-Pyrrolidone (50 mmol) and sodium (0.6 mol) were put in round bottom flask with magnetic stirrer. The reaction was controlled at 50 °C under reduce pressure. After the reaction is completely, the mixture of benzoyl chloride (3 mmol) and tetrahydrofuran (0.8 ml) were added into flask and keep at 50 °C and reduce pressure for 390 minutes. The mixture was dissolved in formic acid and precipitated in acetone. The precipitate was filtrated and washed with water and methanol.

Blend Preparation

The ratio of PTT and PA4 was prepared at 50/50, 60/40, 70/30 and 80/20 %wt. The polymer blend was added in Haake rheomix at 235-240 °C, 30 rpm for 10 minutes. The polymer was extruded and cut at 50 millimeters per minutes. The mixture was compressed by compression molding.

The twin screw extruder was used for prepared polymer blend at the ratio 95/5, 90/10, 85/15 and 80/20 %wt. The condition of extruder varied temperature at 235-240 °C, screw speed of 230 rpm for 10 minutes. The polymer was extruded and cut at 50 millimeters per minutes.

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The effect of compatibilizer was investigated. The bond fast was added to increase the compatibility of polymer blend at 1, 2, 3, 4 and 5%wt for PTT/PA4 at 95/5. The twin screw extruder was used for studying this effect. The condition of twin screw same as above.

Characterizations

Thermal Properties of polymer blends was tested by Differential Scanning Calorimeter (DSC) with Perkin-Elmer DSC 8000. The sample about 5-10 mg was weighed and put in pan. The nitrogen gas was purged during the scan. The temperature was heated from 30 °C to 270 °C at heating rate were 10 °C/min.

Melt Flow Index (MFI) was test by ASTM D 1238-98 with XRL 400. The instrument was warm at 235 °C for 15 minutes. The 6 g of polymer was added in piston. Bring the weight on piston, polymer will melt and flow pass dyne for 10 seconds. The melted polymer was weighed for calculated MFI.

Scanning Electron Microscope (SEM) was performed to investigate the morphology of the materials with JEOL Jsm-6510. The samples were coated with gold. The SEM was operated under vacuum using 15 kV.

3. RESULTS AND DISCUSSION

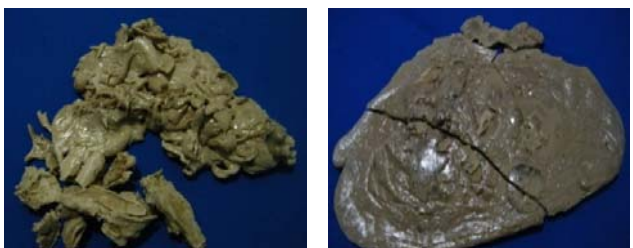
The effect of synthesis polyamide 4

The effect of initiator was studied. This experiment used the mixture of benzoyl chloride and tetrahydrofuran, terephthaloyl dichloride and tetrahydrofuran were initiator. The synthesis that using the mixture of terephthaloyl dichloride and tetrahydrofuran as initiator, the quantity of PA4 is low. While the mixture of benzoyl chloride and tetrahydrofuran as initiator gets higher PA4, 47.30 g PA4. The polyamide 4 is white shown in Fig.1.

Fig.1 Polyamide 4

Preparation polymer blend

Fig.2 (a)-(d) show the polymer blends were prepared by Haake rheomix at the raito of PTT/PA4 50/50, 60/40, 70/30 and 80/20. The all of polymer blends from Haake rheomix were brittle because the polyamide 4 is brittle, so the high quantity of PA4 in polymer blend show brittle too. The decreasing of the PA4 may increase compatibility of polymer blend. The ratio of PTT/PA4 at 95/5, 90/10, 85/15 and 80/20 %wt was studied and the twin screw extruder was used replaced Haake rheomix.



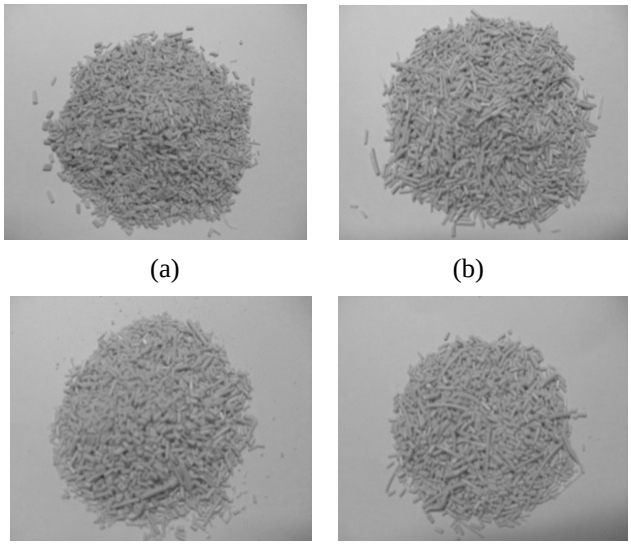
(a) (b)

(c) (d)

Fig.2. Polymer blend between PTT/PA 4 from Haake rheomix

(a) 50/50 (b) 60/40 (c) 70/30 and (d) 80/20 % wt

The PTT/PA4 from twin screw extruder showed heterogeneous, the characteristic of polymer blends was line of PTT and the roughness of PA4. Therefore, 1 wt% bond fast was added as compatibilizer. The additive can improve the compatibility of polymer blend. The blends can cut to small rod but it still brittle. The PTT/PA4 at 1 % wt bond fast was given in Fig.3.



(a) (b)

(c) (d)

Fig.3. PTT/PA4 at 1 %wt bond fast with twin screw extruder

(a) 95/5 (b) 90/10 (c) 85/15 and 80/20 %wt

DSC thermograms of neat PTT and neat PA4 show the melting temperature (T_m) were 229 and 214 °C respectively as shown in Fig.4. From this result the condition of blending was set at 235-240 °C. At 1 % wt bond fast were analyzed. T_m of polymer blends is 214-229 °C. The T_m of blends at 95/5, 90/10, 85/15 and 80/20 were 229.8, 227.9, 226.8, 224.9 °C respectively. The T_m vary with PA4, when PA4 decrease when T_m of blends decrease too. The polymer blends at 80/20 and 85/15 showed 2 peaks of thermograms, they were incompatibility on that blends. While the polymer blend at 90/10 and 95/5 show only 1 peak of themogram that is

compatibility on these blends.

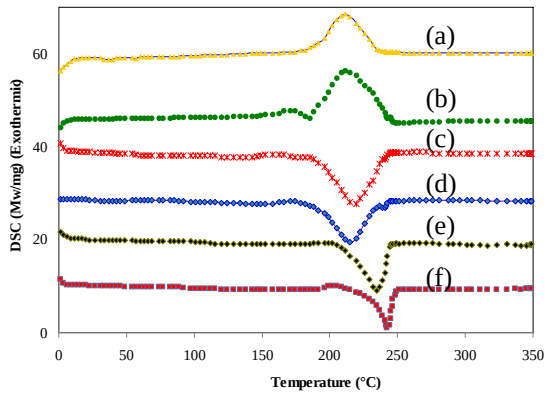


Fig.4. Thermogram of polymer blends between PTT/PA4 95:5 at 1 % wt bond fast

(a) neat PTT, (b) neat PA4, (c) 80/20, (d) 85/15, (e) 90/10 and (f) 95/5

The physical properties of polymer were tested by MFI. The MFI of PTT is 20.22 g/10 min. The polymer blend was vary with the quantity of PTT, if PTT was increase the MFI will decrease that is low viscosity. In contrast to the MFI at 80/20, it decreases as only 38.49 g/10 minutes due to the ratio at 80:20 as incompatibility polymer blends. However, MFI in all of blends still high. The low MFI shows high molecular weight so the 95/5 is lowest MFI in this experiment, 35.46 g/10 minutes, it was selected to test the quantity of bond fast.

Table 1. MFI of polymer blends at vary ratios

quantity of bond fast (%wt)	MFI (g/10 minutes)
95:5	35.46
90:10	45.84
85:15	53.57
80:20	38.49

The neat PTT and PA4 were shown in Fig. 5 (a) and (b) and PTT/PA4 at difference ratio as shown in (c)-(f). In SEM micrograph of samples show a two-phase morphology where PA4 is disperse phase. When the amount of PA4 is increased as observed PA4 scattered in PTT. All of blends show immiscibility.

The effect of compatibility

Table 2. shows thermograms of polymer blends between PTT/PA4 at 95/5 with twin screw extruder by adding 1, 2, 3, 4 and 5 wt% bond fast. The thermal properties of all polymer blends are not difference. From the results show 1 peak that is the compatibility of polymer blends in all conditions.

While the MFI of blends are rather difference. That is the MFI is not increase when the compatibility increase. Maybe the polymer blends can compatible only some part of blends. When the bond fast was added the compatibility will decrease. The bond fast and polymer blends separated phase. The 2 wt% bond fast get lowest MFI was 18.96 g/10 min.

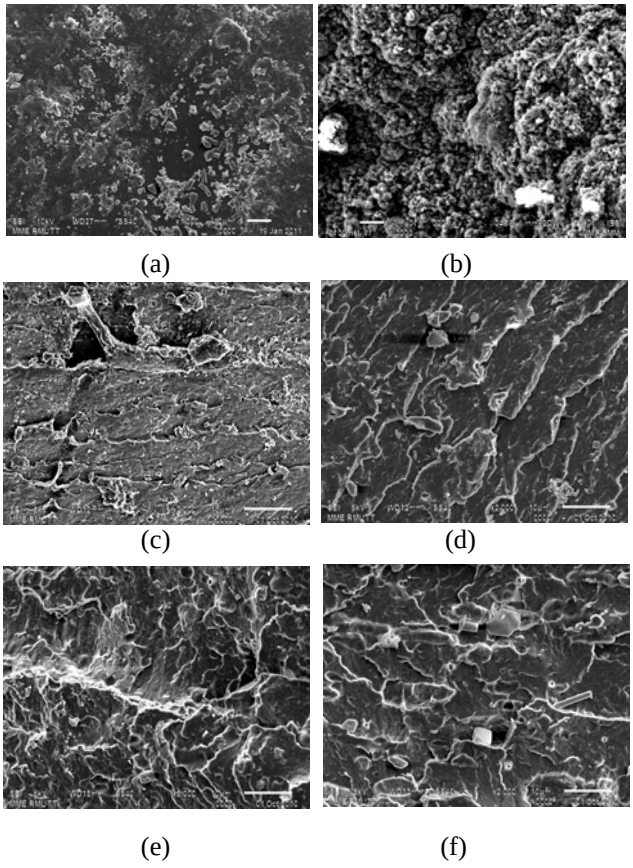


Fig. 5. SEM micrographs of PTT/PA4 at (a) PTT, (b) PA4, (c) 95/5, (d) 90/10, (e) 85/15 and (f) 80/20 at 1wt% bond fast

Table 2. T_m of PTT/PA4 at 1, 2, 3, 4 and 5 wt% bond fast

quantity of bond fast (%wt)	T_m (°C)	MFI (g/10 minutes)
1	229.8	35.46
2	226.1	18.96
3	227.1	24.72
4	225.6	19.68
5	227.1	23.58

4. CONCLUSION

In summary, DSC thermograms show T_m of PTT and PA4 were 229 and 214 °C respectively. And T_m of PTT/PA4 was 214-229 °C.

Polymer blends observed from DSC and SEM micrograph between PTT and PA4 were found to be immiscible. And the MFI still high.

The additive blend increase compatibility of polymer blends. But it is no effect on T_m and MFI.

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