TECHNICAL UNIVERSITY OF CLUJ-NAPOCA

ACTA TECHNICA NAPOCENSIS

Series: Applied Mathematics and Mechanics
Vol. 56, Issue III, September, 2013

INFLUENCE OF ROTATION SPEED DURING EXTRUSION TO THE PROPERTIES AND MORPHOLOGY OF BIOPOLYMERS BLEND

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Abstract: *Bioplastics, automotive industry and agriculture are closely connected. On one side, renewable resources from agriculture are basic feedstock for bioplastics manufacturing. On the other side, products made from bioplastic materials have many applications in agriculture, in packaging for food industry, in construction and in automotive industry. If bioplastics are biodegradable, the life cycle can be closed by degrading them in industrial or home composters. The purpose of the work was to explore the relationship between the composition of a ternary bioplastic blends and its mechanical properties. Tree polymers were used, PLA, PBAT and PA. PLA (polylactide) is a bio-based polymer produced by Nature Works, designed for injection moulding applications. PBAT (ecoflex F BX 7011) is an oil-based, biodegradable polymer designed for film extrusion and extrusion coating, produced by BASF. PA (platamid HX 2656) is a bio-based polymer produced by ARKEMA, designed to be used textile industry, for adhesives and coatings. Relationship between rotation speed during extrusion, morphology and resistance at impact will be given.*

Key words: *biopolymers, blending, rotation speed, resistance at impact, morphology*

1. INTRODUCTION

Biopolymers represent an area where biodegradability would be a tremendous asset to a variety of casings elements, insulation and packaging products. Starch, it's a key component of these renewable raw materials and is becoming an increasingly important input to activities outside the food industry due to the variety of ways in which it can be modified to find applications in industry.

Biopolymer blending is a convenient and attractive route for obtaining new biopolymeric materials with great proprieties, adapted to different applications. Making a parallel between the developments of a new biopolymer, making blends of currently available biopolymers offers significant savings in time and cost, and the blend properties may be tuned by changing the composition [1, 2]. Therefore, achieving compatible of immiscible polymer blends it was a long-standing academic and technological challenge.

In the last period, in despite of the very large number of studies on the compatible of binary biopolymer blends, some studies have considered ternary or multi-component biopolymer blends [4, 5].

2. METHODS

The materials used in this experiment was PLA polymer 3051D (polylactide) produced by Nature Works [7] and PBAT (ecoflex F BX 7011) produced by BASF [8]. The characteristics of PLA 3051D, supplied by Nature Works are shown in Tab.1.
PLA (polylactide) polymer 3051D is designed for injection moulding applications where the requirements are clarity with heat deflection temperatures lower than 55ºC.

The variety of products made with PLA continues to grow rapidly. Applications include cutlery, cups, plates, saucers and outdoor parts.
The characteristics of PBAT (ecoflex F BX 7011) used, supplied by BASF are shown in table 2.

Table 1

PLA 3051D material proprieties

Property	Value	Test Method
Density (g/cc)	1.25	ASTM D792
Melt Index, g/10min (210^{o}C/2.15Kg)	10 to 25	ASTM D1238
Melting Point, ^{o}C	200^{o}C	DSC
Relative Viscosity (Pa·s)	3.0-3.5	
Tensile Strength, (MPa)	48	ASTM D638
Elongation, %	2.5	ASTM D638
Notched Impact, (J/m)	0.16	ASTM D256

Table 2

PBAT (ecoflex F BX 7011) material proprieties

Property	Value	Test Method
Density (g/cc)	1.25 to 1.27	ASTM D792
Melt Index, g/10min (190^{o}C/2.16Kg)	2.7 to 4.9	ASTM D1238
Melting Point, ^{o}C	110 to 120	DSC
Tensile Strength, (MPa)	34	ASTM D638
Elongation, %	700	ISO 527
Water Permeation Rate, $g/(m^2*d)$	140	DIN 53122

Ecoflex F BX 7011 comes closer than any other biodegradable plastic to the processing properties of a classic polymer. A flexible plastic designed for film extrusion and extrusion coating. Blown film extrusion is a particular area where PBAT shows well-balanced processing properties and the resin can be used in extrusion coating applications.

The density of PA PLATAMID HX 2656 is 1.1 (g/cc), having a melting point around 115-120 ^{o}C.

A laboratory internal mixer type Haake Rheomix R600 (figure 1) with mixing chamber volume of 50 cm^3 was use to conduct the extrusions. Like we can see in the Fig. 1, this extruder has two contra-rotates rotors and allows to impose a tangential kneading type at a shear rate given. This device can also measure the temperature of the material and the engine torque exerted by the fluid on the rotor blades.

The chamber of extruder was feed at 80 % from full capacity. The experiments characteristics are:

1) *Experiment 1* - 60 % PLA / 20 % PBAT / 20 % PA – temperature 180 °C, mixing time 12 min., rotation speed 30 rot/min.;
2) *Experiment 2* - 60 % PLA / 20 % PBAT / 20 % PA – temperature 180 °C, mixing time 12 min., rotation speed 50 rot/min.;

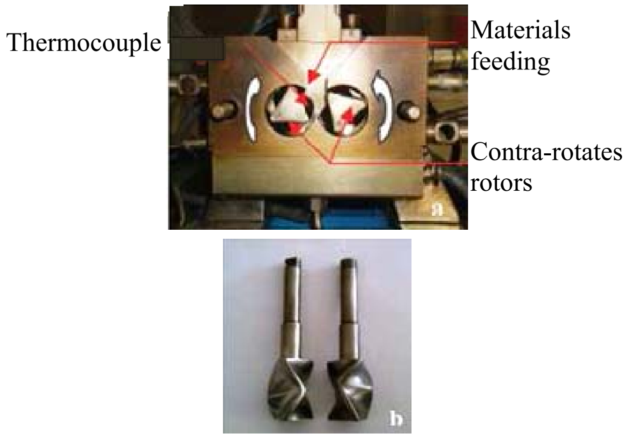

Fig. 1 Internal mixer Haake Rheomix 600 (a) mixing chamber, (b) Contra rotates rotors, profiles.

3) *Experiment 1* - 60 % PLA / 20 % PBAT / 20 % PA – temperature 180 °C, mixing time 12 min., rotation speed 30 rot/min.;
4) *Experiment 2* - 60 % PLA / 20 % PBAT / 20 % PA – temperature 180 °C, mixing time 12 min., rotation speed 50 rot/min.;
5) *Experiment 3* - 60 % PLA / 20 % PBAT / 20 % PA – temperature 180 °C, mixing time 12 min., rotation speed 80 rot/min.;
6) *Experiment 4* - 60 % PLA / 20 % PBAT / 20 % PA – temperature 180 °C, mixing time 12 min., rotation speed 100 rot/min.

Before blending all feeding material was dried in the oven at 80 ^{o}C, minimum 4 hours. All blends was mixed 12 min. at 180 ^{o}C having a rotors speed of 80 rpm. After blending, the material it was collected in one use bags, crushed in small pieces and formed in bars by compression moulding. The compression moulding process was made at 180 ^{o}C for all bars.

From each blend it was formed 3÷4 bars. After that each bar was tested separately at resistance at impact. The dimensions of the bars were 80 x 10 x 3 mm. The depth under the notch of the specimen is 8 mm.

3. APPLICATION AND RESULTS

The determined parameter from resistance at impact tests is impact strength that conform ISO is expressed in kJ/m^2. Impact strength is calculated by dividing impact energy in J by the area under the notch. The machine used for determination of impact strength is CESAT

9050 Impact Pendulum. This machine is designed for determining the resilience of thermoplastic materials to impact. Tests conform to internationally recognized standards belonging to Charpy, Izod, Pipe and Tensile Impact methods, ranging in energy from 0.5 - 50J.

A graphic interpretation of experimental data is presented in figure 2.

From graphic representation from Figure 4 we can observe that an increasing of rotation speed (up to 100 rpm) during the blending is benefic for obtaining a higher resistance at impact proprieties.

In parallel it was made a morphology study of the mixed blends. The obtained morphology is presented in the figures 3 up to 6.

The samples for microscope observations was prepared by cryo fracture by cooling in liquid azote, after that deposit a thin layer of platinum on the studied surface.

For microscope observation it was used ESEM laboratory equipment ZEISS SUPRA 40 with GEMINI column.

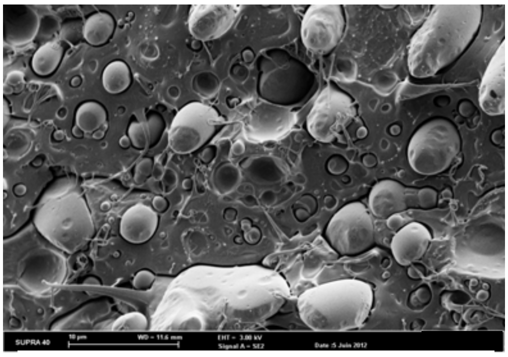

Fig. 3 Photos with obtained morphology for *Experiment 1*.

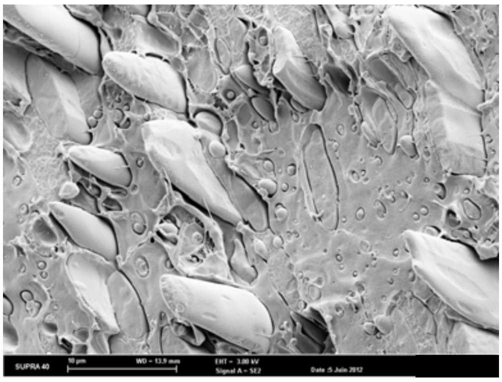

Fig. 4 Photos with obtained morphology for *Experiment 2*.

Fig. 5 Photos with obtained morphology for *Experiment 3*.

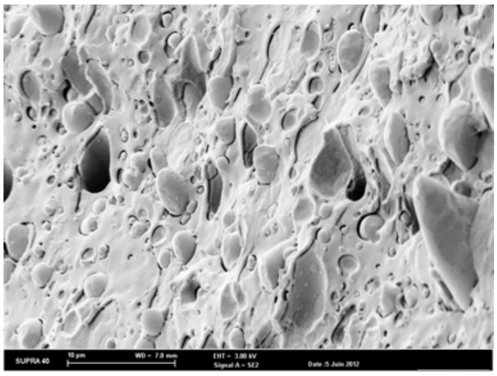

Fig. 6 Photos with obtained morphology for *Experiment 4*.

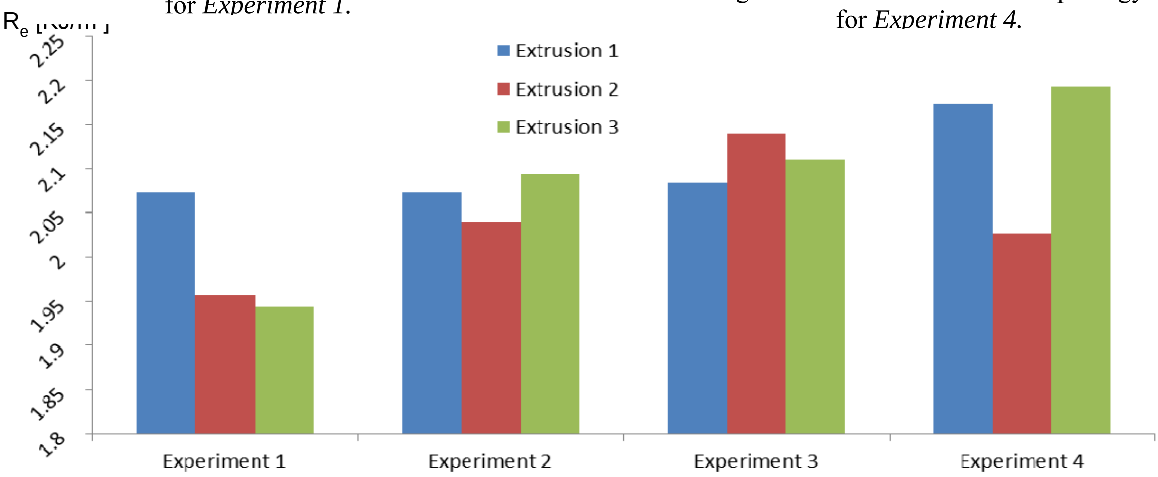

Fig. 2 Influence of rotation speed to the impact strength for studied blends.

After all this experimental studies we can say that the best volume ratio for obtaining the best resistance at impact, between PLA/PBAT/PA ternary blends is 60/20/20. The best resistance at impact is obtained in case of Experiment 4 with 100 rpm, 180 °C during 12 min.

4. CONCLUSIONS

By combining more biopolymers, we can obtain a biopolymer blend with new properties. By changing the matrix volume ratio in the blend, we can obtain different materials with different mechanical properties, adapted to each application. In the present study, we used two biodegradable polymers.

If the goal is to design a material with good mechanical properties, an option is to use PLA as the matrix and PBAT as the inclusion and PA the rest. This leads to good impact properties.

In general, to increase mechanical properties will require adding compatibilizing agents.

Acknowledgments. This research was performed with a grant from Q-DOC project of Technical University of Cluj-Napoca, Romania.

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Influenţa rotaţiei practicate pe parcursul extrudarii asupra proprietăţilor şi morfologiei amestecurilor de biopolimeri

Rezumat : Masele plastice biodegradabile, industria autovehiculelor şi agricultura sunt strâns legate. Pe de o parte, resursele regenerabile din agricultură sunt o materie primă de bază pentru fabricarea maselor plastice biodegradabile. Pe de altă parte, produsele fabricate din materiale bioplastice au multe aplicaţii în agricultură, în industria ambalajelor (pentru industria alimentara), in constructii si in industria auto. În cazul în care materialele plastice biodegradabile sunt regenerabile, ciclul de viaţă pote fi închis prin degradare în conditii naturale de mediu. Scopul lucrării a fost de a explora relaţia dintre compoziţia unui amestec biopolimeric, conditiile de extrudare, proprietăţile mecanice şi morfologia obţinuta. Trei polimeri au fost utilizati, PLA si PBAT (polilactidă) şi PA. PLA este un polimer pe bază de bio-produs fabricat de Natura Works, dezvoltat pentru aplicaţii de injecţie sau turnare. PBAT (ECOFLEX F BX 7011) este un polimer biodegradabil conceput pentru procese de extrudare si formare a folilor pentru ambalare, produs de BASF. PA (platamid HX 2656) este un biopolimer produs de ARKEMA, destinat utilizării în industria textilă,pentru adezivi sau în procese de acoperire (înfoliere). În aceasta lucrare s-a analizat relatia dintre turaţia practicată pe parcursul extrudării, morfologia obţinută şi rezistanţa la impact a mostrelor de material obtinute.

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