APPENDICES

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APPENDIX A

Theory of the Biodegradation Testing (According to ISO14855-1)

ISO14855-1: 2005 (Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions – Method by analysis of evolved carbon dioxide - Part 1: General Method)

Criteria for evaluations

a) Percentage of biodegradation cellulose (positive control) obtained more than 70%

☑ Yes
☑ No

b) Percentage of biodegradation cellulose (positive control) 2 times error ≤ 20%

☑ Yes
☑ No

c) Blank period time 10 fist days obtained CO₂ occurred between 50 to 150 mg/1 g dry solid

☑ Yes
□ No

A. Standard Test Method

ISO 14855-1: 2005

B. ScopeDetermination of the ultimate aerobic biodegradability of PBS and PBS/CAB/Paraplex G40 (70/30/10) and CAB under composting conditions.

C. Theory

Determination aerobic biodegradation of plastic materials under controlled composting conditions in a laboratory-scale test according to ISO 14855-1. Grinded sample into powder pellet were mixed in controlled compost. The biodegradation test was performed in a controlled compost at 58 ± 2 °C for 90 days and the moisture content of the compost was adjusted to about 50-55%. The concentration of oxygen in the reactor was not lower than 6%. The degree of biodegradation of the sample was determined by measure the amount of carbon dioxide evolution.

D. Environmentally Controlled

The reactors were kept in a dark at an average temperature of 58 ±2 °C.

E. Chemicals and Apparatus

- Cellulose powder of thin-layer chromatography grade with particle size of less than 20 μm (cellulose microcrystalline; Merck, Germany).
- 2. LDPE (SCG Plastics Company Ltd.) exhibits a density 0.923 g/cm³, and melt flow index of 1.7 g/10 min at 190 °C and 2.16 kg load.
- 3. Sodium hydroxide (NaOH) GR Analysis grade was purchased from Merck Chemical (Germany)
- 4. As bioreactors, glass flasks of 2 liters internal volume were used.

F. Apparatus

Composting apparatus set-up shown in Figure A.1

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G. Procedure

G.1 Compost Inoculum

Three-month-old compost inoculum was used for biodegradation purpose and its had the following characteristics shown in Table A1.

Table A.1 The characteristics of compost inoculum.

Compost	Dry golide content	Moistura	Volatila solida
Composi	Dry solids content	Moisture	volatile solids
inoculum	(g)	content (g)	content (g)
757.70	360.13	397.57	146.00

G.2 Test Sample

Sample

PBS PBS/CAB/PG40 (70/30/10) CAB







PBS/CAB/PG40 (70/30/10)

Figure A.2 Physical appearance of sample testing: a) sample obtained and b) sample for testing (< 1000 μ m).

Positive Control	:	Cellulose (Cellulose m	icrocrystalline)
Negative Control		LDPE (Low density po	olyethylene)
Age of compost	:	3 month	
Size of Vessel	2115	2 litter	
Test method		determination of carbo	n dioxide occur
Standard method	Chia	ISO14855-1 (2005)	
	Positive Control Negative Control Age of compost Size of Vessel Test method Standard method	Positive Control:Negative Control:Age of compost:Size of Vessel:Test method:Standard method:	Positive Control:Cellulose (Cellulose mNegative Control:LDPE (Low density poAge of compost:3 monthSize of Vessel:2 litterTest method:determination of carboStandard method:ISO14855-1 (2005)

G.3 Experimentally

Biodegradation test on polymeric plastics and their starting materials were carried out in a laboratory scale-compost experiment. The compost was sieved through a 10 mm and inert materials such as glass, stone and metal were removed. It's had the following characteristics: total solids, volatile solids; pH 7.55. Cellulose power of thin-layer chromatography grade with a particle size less than 20 µm (cellulose microcrystalline) was used as a reference material (i.e., positive control). The sample were grinded into powder particle size of less than 1000 µm The compost (360 g, dry weight) and sample/reference (60 g, dry weight) were mixed throughly and place inside a glass reactor. The ratio of dry mass of compost to dry mass of sample or reference was about 6:1. Each blank consisted of the compost only, containing about 360 g (dry weight) to determine the respiration activity of this compost. The experimental replicates of the tested materials or blank were two (diplicate). The set of bioreactors was exposed to humidified air free of CO₂ gas for a test period of 90 days at a constant temperature of 58 ± 2 °C. The moisture content of the compost was adjusted to about 50-55% by the deionized water and the C/N ratio range between 10 and 40. The concentration of oxygen in the reactor was not lower than 6% at all times. Check CO_2 and O_2 concentrations in the outgoing air at least daily with a minimum time interval of 6 hour after the first week for the reminder of the test. Check air flow daily before the composting vessels and at the outlets, ensuring that no leaks are present in the complete system. Adjust air flow to maintain a CO₂ concentration of at least 2% volume over volume to allow accurate determination of CO₂ level in exhaust air. The degree of biodegradation were calculated resporation CO₂ amount determined from a blank, and theorectical produced CO_2 amount of the added sample. The amount of CO_2 gas evolved was measured using cumulative (tritration method). The CO₂ evolved from the bioreactor was absorbed in 1000 ml of 0.5 N NaOH solution and determined by titration with 0.1 N hydrochloric acid.

L J L I V

Bioreactor	Com	ipost	Sample		
Bioreactor	Wet weight (g)	Dry weight (g)	Wet weight (g)	Dry weight (g)	
Blank	757.70	360.13		-	
Cellulose (Positive Control)	757.51	360.07	63.00	60.00	
LDPE (Negative Control)	772.29	420.05	70.23	70.14	
САВ	772.69	420.27	70.02	69.21	
PBS	757.39	360.01	61.61	60.00	
PBS/CAB/Paraplex G40 (70/30/10)	757.66	360.14	61.00	60.00	

Table A.2 The composition of compost and sample/reference in the bioreactors.

G.4 Test Period

The period of the composting studies was 3 months (90 days).

H. Calculation

Determine volatile solid, organic carbon and the theoretical carbon dioxide (ThCO₂) of all test substances are shows data in Table A.4. The determination total carbon content of the test material by elemental analysis.

Determination of the theoretical quantity of carbon dioxide $(ThCO_2)$ evaluation of polymer samples (include positive control and negative control) to be calculation as follow equation (1):

 $ThCO_2 = M_{TOT} \times C_{TOT} \times 44/12$ (1) where M_{TOT} is the total dry solids in grams of test material, introduced into composting vessels at start of the test; C_{TOT} is the proportion of total organic carbon in the dry solids in the test material in grams.

Determination of the percentage biodegradability was calculated by comparing the amount of car dioxide in the sample (include positive control and negative control) with the blank by following equation (2):

%Biodegradation = $[(CO_2)_T - (CO_2)_B] \times 100 / ThCO_2 \dots (2)$

where $(CO_2)_T$ is the cumulative amount of CO_2 evolved in each composting vessel containing the test material in grams; $(CO_2)_B$ is the cumulative amount of CO_2 evolved in blank (inoculums) vessel in grams; ThCO₂ is the theoretical carbon dioxide evolved, based on total carbon in sample.

ThCO₂ Volatile Total organic Dry solids Moisture Sample solids carbon content (CO₂ g/100 g) (%) (%) (%) (%) sample) Cellulose 95.24 4.76 100.00 43.52 159.57 (Positive control) LDPE 99.87 99.98 0.13 86.48 317.09 (Negative control) CAB 98.84 99.94 56.86 208.47 1.16 PBS 97.39 2.61 99.99 57.02 209.07 PBS/CAB/PG40 98.36 1.64 99.95 58.28 213.69 (70/30/10)

Table A.3 Evaluation volatile solid, organic carbon and theoretical carbon dioxide(ThCO2) of all the test substances.

I. Results

The cumulative CO_2 production and %biodegradation of all test substances are shows in Table A.4 and A.5. Graph cumulative production of ${}^{14}CO_2$ and %biodegradation of sample and reference as shown in Figure A3., A.4 and A.5, respectively.

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		Cumu		%Biodegradation (%)			
Days	Blank	Cellulose	LDPE	Net Cellulose	Net LDPE	Cellulose	LDPE
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1	2.19	2.97	2.93	0.78	1.82	0.81	0.82
2	3.21	6.71	3.96	3.50	2.16	3.66	0.97
3	4.52	20.63	5.39	16.11	2.98	16.82	1.34
4	5.55	30.78	6.13	25.23	3.20	26.35	1.44
5	6.52	37.78	6.80	31.26	3.36	32.65	1.51
6	7.34	42.37		35.03	-	36.59	-
7	8.23	46.51	7.89	38.28	3.38	39.98	1.52
8	9.13	50.57	-	41.44		43.28	
10	10.60	56.35	9.20	45.75	3.27	47.78	1.47
12	12.11	62.01	9.82	49.90	3.09	52.11	1.39
14	13.57	67.55	10.50	53.98	3.05	56.38	1.37
17	15.72	74.89		59.17	/ - /	61.80	-
20	17.74	80.94	-	63.20	/ -/	66.00	
23	20.47	87.82	-	67.35		70.34	
26	22.00	90.54	- 1	68.54	- 0	71.58	<u> </u>
30	24.35	95.43	14.92	71.08	2.42	74.24	1.09
35	27.81	102.12	15.84	74.31	2.07	77.61	0.93
40	29.86	106.04	16.52	76.18	1.51	79.56	0.68
45	32.27	110.39	17.73	78.12	1.20	81.59	0.54
50	34.63	114.41	18.73	79.78	1.22	83.32	0.55
55	36.81	118.03	19.77	81.22	0.76	84.83	0.34
59	38.50	120.70	· • •	82.20		85.85	-
65	40.53	124.04	21.03	83.51	-0.02	87.22	-0.01
70	42.18	126.50	21.72	84.32	-0.44	88.06	-0.20
75	43.95	129.15	22.41	85.20	-0.85	88.98	-0.38
80	45.68	131.51	23.25	85.83	-1.13	89.64	-0.51
85	47.69	134.35	23.90	86.66	-1.47	90.51	-0.66
90	49.31	136.88	24.82	87.57	-1.60	91.46	-0.72

Table A.4 Cumulative CO₂ production and %biodegradation of blank, cellulose and LDPE.

Cumulative CO ₂ production (g)								%Biodegradation (%)		
Days	Blank	PBS	sample	CAB	Net PBS	Net Sample	Net CAB	PBS	Sample	CAB
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1	2.19	3.06	4.47	1.43	0.87	2.28	0.32	0.69	1.78	0.22
2	3.21	4.34	6.63	2.22	1.13	3.42	0.42	0.90	2.67	0.29
3	4.52	6.79	8.93	2.96	2.27	4.41	0.55	1.81	3.44	0.38
4	5.55	9.11	10.56	3.59	3.56	5.01	0.66	2.84	3.91	0.46
5	6.52	11.54	12.02	4.25	5.02	5.50	0.81	4.00	4.29	0.56
6	7.34	13.56	13.23	-	6.22	5.89	-	4.96	4.59	-
7	8.23	15.89	14.46	5.48	7.66	6.23	0.97	6.11	4.86	0.67
8	9.13	18.34	15.71	-	9.21	6.58	-	7.34	5.13	10t
10	10.60	22.32	17.66	7.16	11.72	7.06	1.23	9.34	5.51	0.85
12	12.11	26.66	19.59	8.06	14.55	7.48	1.33	11.60	5.83	0.92
14	13.57	31.18	21.46	8.72	17.61	7.89	1.27	14.04	6.15	0.88
17	15.72	37.81	24.13	-	22.09	8.41	-	17.61	6.56	-
20	17.74	44.78	26.75	-	27.04	9.01	-	21.56	7.03	-
23	20.47	54.70	30.16	-	34.23	9.69	/ -	27.29	7.56	2-
26	22.00	60.62	31.87	-	38.26	9.87	2	30.50	7.70	- (
30	24.35	69.70	34.99	11.89	45.35	10.64	-0.61	36.15	8.30	-0.42
35	27.81	85.09	39.61	14.12	57.28	11.80	0.35	45.66	9.20	0.24
40	29.86	92.94	42.36	15.69	63.08	12.50	0.68	50.29	9.75	0.47
45	32.27	102.59	45.69	17.8	70.32	13.42	1.27	56.06	10.47	0.88
50	34.63	111.39	48.99	18.69	76.76	14.36	1.18	61.19	11.20	0.82
55	36.81	119.05	51.99	20.15	82.24	15.18	1.14	65.56	11.84	0.79
59	38.50	124.59	54.45	T	86.09	15.95		68.63	12.44	-
65	40.53	129.88	57.35	23.08	89.35	16.82	2.03	71.23	13.12	1.41
70	42.18	133.74	59.63	24.38	91.56	17.45	2.22	72.99	13.61	1.54
75	43.95	137.64	62.27	25.63	93.69	18.32	2.37	74.69	14.29	1.64
80	45.68	141.14	64.64	26.63	95.46	18.96	2.25	76.10	14.79	1.56
85	47.69	145.51	67.55	27.97	97.82	19.86	2.60	77.98	15.49	1.80
90	49.31	149.25	70.34	29.19	99.94	21.03	2.77	79.67	16.40	1.92

Table A.5 Cumulative CO₂ production and %biodegradation of blank, PBS, sample (PBS/CAB/Paraplex G40) and CAB.



Figure A.3 Net cumulative CO₂ production as a function of time for Blank(■), cellulose (●), LDPE (▲), CAB(►), PBS(♦), 70/30/10 (◄).



Figure A.4 Net cumulative CO₂ production as a function of time for cellulose (●),
 LDPE (▲), CAB(►), PBS(◆), 70/30/10 (◄).



Figure A.5 Degree of biodegradation as a function of time for cellulose (●), LDPE
(▲), CAB(►), PBS(◆), 70/30/10 (◄).

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APPENDIX B

SUPPORTING PROCEEDING

The Pure and Applied Chemistry International Conference (PACCON 2012)

"Formulation Development for Biodegradable Poly(butylene succinate) Based Film"

11-13 January 2012

at Chiang Mai Thailand

A l i g h t s i e s e r v e d



The 6th PACCON / Symposium on Biomaterials, Bioplastics and Bionanotechnology (BIP) Le 2_{th} baccon / Symbosium on Biomaterials, Bioplastics and Bionanotechnology (BIb)

BIP-P-110

FORMULATION DEVELOPMENT FOR THE PRODUCT OF BIODEGRADABLE POLY(BUTYLENE SUCCINATE) BASED FILM

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ABSTRACT

Nowadays, majority packaging product made from plastic to cause increase amount of waste plastic. Moreover, almost all plastics today are made from crude oil and natural gas, which are becoming scarce and more expensive. Degradable polymer, therefore is a wise alternative to get interested especially poly(butylene succinate)(PBS). In order to improve the properties of PBS, in particular its transparency and lower its unit cost, the PBS has been melt blended with cellulose acetate butyrate (CAB) and a polymeric plasticizer (polyester adipate, Paraplex G40) using an internal mixer and then films prepared by compression molding. The thermal and mechanical properties of the PBS/CAB blends of various compositions (100/0, 70/30, 50/50, 30/70 and 0/100 wt %) both without and with 10% by weight of Paraplex G40 were investigated by differential scanning calorimetry (DSC), scanning electron microscopy (SEM), tensile testing and UV spectrometry. The results indicate that PBS and CAB are compatible all of blend composition which notice by single glass transition temperature (T_g) and no phase separation from SEM images. Addition of Paraplex G40 to the PBS/CAB blends increase with the increase weight% of PBS and incorporation of Paraplex G40 increase the film flexibility and elongation at break. Moreover, CAB can increase the transparency of polymer blends.

Keywords Poly(butylene succinate) (PBS); Cellulose acetate butyrate (CAB); Paraplex G40; Polymer blends





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Polydutylene succinate (PBS) has a number of interesting properties including biodegradability, flexibility, good thermal stability and perfect processability. It has proved to be strongly resistant to heat. In addition PBS is environmentally friendly because it is not a source of carbon dioxide as a socalled green house gas. However, PBS by itself is rather turbidity and low strength material which makes it unsuitable for use in applications. Consequently, PBS tends to be blended with cellulose acetate butyrate (CAB) and a polymeric plasticizer for potential use as biodegradable packaging material. Based on this methodology, a logical starting point for this research could be a three-component blends consisting of PBS, CAB and plasticizer polyester adipate or PG-40

Introduction

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\blacksquare & & & \\
\Box^{(1)} & \Box^{(1)} & \Box^{(2)} & \Box^{($$

Methods

Blends of PBS and CAB in forms of thin films were prepared by melt blending. The thermal and mechanical properties of the PBS/CAB blends of various compositions both without and with plasticizer (PG-40) were investigated by differential scanning calorimeter (DSC) and tensile mechanical testing.

Results and Discussion

DSC

TEC

The results shown correspond to the second heating. Table 1 show the T_g and second DSC scans of PBS/CAB blends with 10% by weight or without the use of a plasticizer (PG-40). A single T_g is observed for all blend compositions, which indicates that the PBS/CAB system is compatible.

Table 1: Thermal properties (DSC second run) of pure PBS and blends of PBS and CAB with or without plasticizer (PG-40)

	Thermal properties					
PBS/CAB	T _g (°C)		T _c (°C)		T _m (°C)	
	0	10	0	10	0	10
	(wt.%)	(wt.%)	(wt.%)	(wt.%)	(wt.%)	(wt.%)
Pure PBS	-30.9	-34.6	97.5	95.1	110.7	110.8
70/30	-31.7	-32.5	35.2	25.6	100.4	98.4
50/50	-16.6	-22.5	n/d	n/d	n/d	n/d
30/70	23.9	-3.2	n/d	n/d	n/d	n/d
Pure CAB	107.0	94.0	n/d	n/d	n/d	n/d

n/d = not detectable

The main effect of the plasticizer (PG-40) at 10% by weight when added to the PBS/CAB blends was to lower the glass transition temperature, T_g , as shown in Figure 1.

Also, the plasticization effect of the PBS by the PG-40 increased its miscibility with the CAB. The melting and crystallization temperatures, $T_{\rm m}$ and $T_{\rm c}$, were observed at weight % of PBS ≥ 70 but not for lower weight % of PBS and pure CAB. These results suggest that the CAB inhibits crystallization of PBS in the blends and that the PBS is in an amorphous state when the weight % of PBS is low.



Figure 1. Effect of weight % PBS and PG-40 on the T_g of the blend films.

Mechanical properties

As shown in Figure 2, the blending of PBS with CAB showed increasing % EB at break with the increase weight % of PBS. These findings suggest a compatibility of those polymer. The addition of PG-40 in PBS/CAB blends reduced considerably the tensile strength in all the blend composition and increase % EB. And found that elongation at break which the most flexible composition blends is PBS/CAB/PG-40 = 70/30/10.



Figure 2. Stress at break and elongation at break as a function of PBS/CAB composition with or without plasticizer (PG-40) $\,$

Conclusions

PBS/CAB were compatible all of blend composition. Addition of PG-40 to the PBS/CAB blends decreased the T_g as would be expected. From tensile testing, the elongation at break of the polymer blends increased with increasing weight % of PBS. Incorporation of PG-40 increased film flexibility and elongation at break.

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FORMULATION DEVELOPMENT FOR BIODEGRADABLE POLY(BUTYLENE SUCCINATE) BASED FILM

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Abstract: Nowadays, the majority of packaging product made from plastic cause increasing amounts of waste plastic. Moreover, almost all plastics today are made from crude oil and natural gas, which are becoming scarce and more expensive. Degradable polymers are therefore a wise alternative and there is interest especially in poly(butylene succinate)(PBS). In order to improve the properties of PBS, in particular its transparency and lower its unit cost, the PBS has been melt blended with cellulose acetate butyrate (CAB) and a polymeric plasticizer (polyester adipate, Paraplex G40) using an internal mixer and then films prepared by compression molding. The thermal and mechanical properties of the PBS/CAB blends of various compositions (100/0, 70/30, 50/50, 30/70 and 0/100 wt %) both without and with 10% by weight of Paraplex G40 were investigated by differential scanning calorimetry (DSC), scanning electron microscopy (SEM), tensile testing and UV spectrometry. The results indicate that PBS and CAB are compatible in all blend compositions as indicated by a single glass transition temperature (T_s) and no phase separation from SEM images. Addition of Paraplex G40 to the PBS/CAB blends decreased the Tg of the three-components blends. From tensile testing, the elongation at break of the polymer blends increase with the increase weight% of PBS and incorporation of Paraplex G40 increases the film flexibility and elongation at break. Moreover, CAB can increase the transparency of the polymer blends.

1. Introduction

In recent years, the world's environment has deteriorated, partly due to plastic pollution since these materials are hard to dispose of. If all the conventional plastics were made from biodegradable polymers, the environmental problems would be reduced. Therefore, we are attempting to produce a new biodegradable composite material by using a blending technique.

Poly(butylene succinate) (PBS) is a biodegradable aliphatic thermoplastic polyester. This polymer is semi-crystalline and flexible due to its structure. PBS has a number of interesting properties including biodegradability, flexibility and good thermal stability. It has proved to be strongly resistant to heat [1]. In addition PBS is environmentally friendly because it is not a source of carbon dioxide as a so-called greenhouse gas. The monomer precursors for making PBS are succinic acid and 1,4-butanediol. Both of these monomers can be prepared from petrochemical products and natural raw materials [2]. However, PBS by itself is a rather turbid and low strength material which makes it unsuitable for use in applications where transparency and strength are required. Consequently, PBS tends to be blended with other components to modify its properties and lower its cost.

Cellulose esters are one of the thermoplastics derived from biomass feed stocks. CAB esterified by acetyl and butyryl groups is a brittle and transparent material. It has improved compatibility and solubility. The properties of CAB are easily modified by addition of plasticizer and/or resins to modify adhesion, flexibility, heat sealing ability, and moisture resistance. Therefore, CAB has been used as a photofilm and a coating material. Some researchers reported that CAB acted as a plasticizer for some polyesters, such as poly(hydroxy butyrate) [3-5]. Frores and et al. reported that biobased furfural was a good plasticizer for PBS [6].

Plasticizers are widely used to improve process ability, flexibility and ductility of polymers. In the case of a semi-crystalline polymer like PBS, an efficient plasticizer has to reduce the Tg. Recently, plasticizers such as polyester adipate (Paraplex G-40), a high molecular weight permanent plasticizer, have been found to give excellent durability. Paraplex G-40 has low volatility, excellent resistance to extraction by

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hydrocarbon solvents and can improve the flexibility and process ability of polymer blends.

In this work, the preparation of some novel threecomponent blends of PBS, CAB and poly (ester adipate) are described and their properties as potential packaging materials are examined.

2. Materials and Methods

2.1 Materials

PBS was obtained from Mitsubishi Chemical Corporation (China). The PBS pellets were milky white in colour with a number-average molecular weight of 30,000 g/mol and a T_g of -30 °C and a T_m of 110°C.

Cellulose acetate butyrate (CAB500-5) was obtained in powder form from Eastman Chemical Company (Kingsport, Tennessee). The content of CAB with average molecular weight of 57,000 was butyryl 51%, acetyl 4%, and of hydroxyl 1%.

Poly(ester adipate) with a number-average molecular weight of 6,000 was supplied by HallStar Company (USA) under the trade name of Paraplex G40 or PG-40.

2.2 Preparation of Polymer Blends

The PBS and CAB were dried in a vacuum oven at 60 °C for 24 h prior to use. PBS and CAB were mixed together in a molten state at various blend ratios, i.e., PBS/CAB = 100/0, 70/30, 50/50, 30/70 and 0/100 both without and with PG-40 10% by weight. Compounding was performed in an internal mixer at 175 °C for 20 min. The obtained samples were compressed into flat sheets with 0.5-1.0 mm thickness by compression molding machine at 175 °C and then cooled down to room temperature. After cooling, the sheets were cut into dumbbell-shaped specimens for tensile testing.

2.3 Blend Properties

2.3.1 Thermal Properties

The thermal properties of the various blended films were characterized by a Differential Scanning Calorimeter (Mettler Toledo, DSC822). The samples were sealed in aluminum pans and heated from -60 to 200 °C at a heating rate of 10 °C/min under a nitrogen atmosphere. Both first and second scans were recorded in all cases. The glass transition temperature (T_g) , crystallization temperature (T_c) and melting temperature (T_m) were determined from the second heating scans.

2.3.2 Mechanical Properties

Mechanical properties of the various blended films of size 65 mm \times 6 mm \times 0.5-1.0 mm were evaluated on a universal mechanical testing machine (Model 55R4502) according to the ASTM D 638, with a crosshead speed of 50 mm/min. The stress-strain curves were used to determine the tensile strength and % elongation at break (% EB). Seven specimens were tested for each blend and the results reported as average values.

2.3.3 Scanning Electron Microscopy (SEM)

The morphologies of thin films (PBS, CAB, PG-40 and their blends) were investigated using SEM at an accelerating voltage of 15 KV and a magnification of 2000.

2.3.4 Optical Properties

Optical properties of PBS, CAB, PG-40 and thin film polymer blends were measured using a UV-VIS spectrometer (Lambda 25, Perkin-Elmer) at a wavelength of 500 nm in transmission mode compared to air as a standard.

3. Results and Discussion

3.1 Thermal Properties

The thermal properties of PBS and the PBS/CAB blends with 10% by weight or without the use of a plasticizer (PG-40) were investigated by DSC; the results shown correspond to the second heating. Table 1 shows the T_g and second DSC scans of PBS and PBS/CAB blends. A single T_g is observed for all blend compositions, which indicates that the PBS/CAB system is compatible.

	/						
11 /	Thermal Properties						
PBS/CAB	T _g ((°C)	T _m (T _m (°C)			
	0	10	0	10			
	(wt.%)	(wt.%)	(wt.%)	(wt.%)			
Pure PBS	-30.9	-34.6	110.7	110.8			
70/30	-31.7	-32.5	100.4	98.4			
50/50	-16.6	-22.5	n/d	n/d			
30/70	23.9	-3.2	n/d	n/d			
Pure CAB	107.1	94.0	n/d	n/d			
1							

n/d = not detectable

Table 1: Thermal properties (DSC second run) of pure PBS and blends of PBS and CAB with or without plasticizer (PG-40)

The main effect of the plasticizer (PG-40) at 10% by weight when added to the PBS/CAB blends was to lower the glass transition temperature, T_g , as shown in Figure 1. Also, the plasticization effect of the PBS by the PG-40 increased its miscibility with the CAB. The melting and crystallization temperatures, T_m and T_c , were observed at weight % of PBS \geq 70 but not for lower weight % of PBS and pure CAB. These results suggest that the CAB inhibits crystallization of PBS in the blends and that the PBS is in an anorphous state when the weight % of PBS is low.

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Figure 1. Effect of weight % PBS and PG-40 on the T_g of the blend films.

3.2 Mechanical Properties

Tensile strength and elongation of PBS, CAB and without and with 10% by weight of PG-40 in PBS/CAB blends are summarized as shown in Figure 2. The blending of PBS with CAB showed increasing % EB at break with the increase weight % of PBS. The PBS/CAB blends showed increasing % EB all composition. These findings suggest a compatibility of those polymers.



Figure 2. Stress at break and elongation at break as a function of PBS/CAB composition with or without plasticizer (PG-40)

The addition of PG-40 to the PBS/CAB blends decreased considerably the tensile strength in all of the blend compositions and increased the % EB. The highest elongation at break which gave the most flexible composition was for the PBS/CAB/PG-40 = 70/30/10 blend.

3.3 Scanning Electron Microscopy (SEM)

The SEM images shown in Figure 3 show the surfaces of the PBS, CAB, and their blends both without and with 10% by weight of PG-40. No obvious phase separation was observed for the blend compositions, suggesting that they were compatible. This is consistent with the DSC results which gave only a single glass transition temperature.



Figure 3. SEM images of PBS/CAB without or with plasticizer (PG-40)

3.4 Optical Properties

The transparency of the blended films was around 70% up to a weight % of CAB = 70. The transparency increased slightly when PG-40 10% by weight was added which was an indication that plasticization aided miscibility.

4. Conclusions

PBS is a promising biodegradable polymer which can be blended with CAB and PG-40 to modify its properties and lower its cost. The properties and compatibility of these biodegradable polymer blends were determined through thermal, mechanical and optical property testing. The changes in the tensile

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properties and the DSC results suggest that blends of PBS and CAB are compatible. Addition of PG-40 to the PBS/CAB blends decreased the T_g as would be expected. From tensile testing, the elongation at break of the polymer blends increased with increasing weight % of PBS. Incorporation of PG-40 increased film flexibility and elongation at break. Blending could increase the transparency of the polymer blends films.

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