



Use of Thermal Black in PLA films for industrial applications

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Sustainable polymer products at NRC *Mission and Research areas*

Support to NRC's Advanced Manufacturing Program Automotive & Surface Transportation Research Center

Support to Government Policies Zero Plastic Waste Strategy and Circular Economy (2018)





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Cancarb overview

PLATINUM

ecovadis Sustainability Rating

2023

- The only company solely dedicated to the manufacturing of <u>Thermal Carbon Black</u>
- Thermax is one of the <u>purest and cleanest carbon</u>
 <u>blacks</u> available at the industrial scale
- Annual capacity of <u>120 million pounds</u>
- Thermax thermal black is produced by cracking natural gas into its constituent elements C and H
- Production of zero-emission power by capturing hot exhaust gases to produce stream that drives an electric generator
- Thermax can be used in rubbers, insulation, refractories, metallurgy, concrete, ceramics, in <u>thermoplastics, elastomers and their composites</u>

RC CRC

Thermal Black vs. Furnace Black

Particle Size Diameter and Structure



N110

N762



Thermax N990

- Carbon black can be defined as very fine particle aggregates of carbon, possessing a paracrystalline molecular structure, while Thermal Black has an amorphous structure
- > The main distinction between Thermal Black and Furnace black are particle size and structure
- Thermal Black, due to its higher particle size and lower structure, compared to even the most coarse furnace black, can be translated into excellent properties of the thermoplastic composites

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PLATINUM Top 1% 2023 ecovadis Sustainability Rating



Outline

- State-of-the-art on PLA films
- Extrusion of compounds and cast-film extrusion
- > Mechanical/Thermal/Morphology Characterizations
 - Cast films
 - Biaxially stretched films
 - Blown films
- Complementary characterization
 - Biodegradability
 - Barrier properties
 - Optical properties
- Conclusions

CNRC-NRC 2.0kV 7.0mm x5.00k SE(M)

Thermal Black Thermax® N990 from spray drying



State-of-the-art on PLA films

Results from manufacturers of PLA - grades recommended for film applications

TS at break	Elongation at break	Film thickness	Grado/Mapufacturor	Applications
MD/TD (MPa)	MD/TD (%)	(µm)	Grade/Manufacturer	Applications
37.6 / NA	290 / NA	30	EcoHumer BFX303, WooSung Chemical Co., Ltd.	Blown films for different applications
29 / 21	280 / 400	20	Bio-Flex [®] F 1138, FKuR Kunststoff GmbH	T-shirt bags, waste bags
103 / 145	180 /100	25	4032D, NatureWorks [®] LLC	Biaxially oriented film, use temperatures up to 150°C
47 / 40	440 / 350	50	Ecovio® F2224, Copolyester, BASF Corporation	Blown films for different applications

Results from manufacturers of PLA films - values from TDS

TS at break MD/TD (MPa)	Elongation at break MD/TD (%)	Film thickness (µm)	Grade/Manufacturer	Applications
28 / 26	400 / 650	25	Ginegar Plastic Products Ltd.	Commercialized Mulch films
20 / 16	480 / 580	30	Bio-Flex® FX 1824, FKuR Kunststoff GmbH	Compostable bags made by film blowing

Results from scientific literature published on PLA films

TS at break	Elongation at break MD/TD	Film thickness		Recommended Applications
MD/TD (MPa)	(%)	(µm)	Reference	
60 MPa - 1.5 mm cast 100 MPa - 0.7 mm BO	20 % - 1.5 mm cast 120% - 0.7 mm BO film	1.5 mm - cast film 0.7 mm - bi-ax film	ACS Sustainable Chem. Eng. 2021, 9, 6296–6304	PLA 4032D / silica particles composite Superhydrophobic biodegradable PLA films, excellent self-cleaning and mechanical properties, Combining casting-thermal stretching and surface treatment.
50 - 60 MPa for films of 500-550 μm 90 - 120 MPa for films of 170 - 190 μm	5% for films of 500-550 μm 35 - 50% for films of 170 - 190 μm	500 to 550 μm 170 to 190 μm	Int J Biol Macromol, 2021 Jun 30;181:521-527.	PLA 4032D with hydroxyl-terminated polybutadiene (HTPB) films Extruded film, great mechanical O2 barrier properties. 7

Compounding and cast-film extrusion

Extrusion:

PLA 4032D from NatureWorks[®] - grade for films for industrial, shopping and trash bags. **Thermal Black (TB) Thermax ® N990 - from Cancarb Ltd.**

Matrix		TB N	990 cor	CB N762 content wt.%			
PLA	0	3	10	20	15	40	20

All compounds were used further in cast-film extrusion, biaxial stretching, and film blown processes.

Film casting:

Extrusion of cast films with a thickness of ca. 100 μ m from each formulation and from pristine PLA as well.

The cast film obtained from PLA containing 40 wt.% TB proved to be very brittle and was discarded. A dilution was done down to PLA / 15 wt.% TB and extruded to obtain again cast films.

Compounding



Extrusion casting



Mechanical testing:

- Tensile properties according to ASTM D882-18: *Standard Test Method for Tensile Properties of Thin Plastic Sheeting;* Parameters used: 5 kN cell, 100 mm grips distance, 10 mm/min speed
- All samples were conditioned at 23 °C, 50% RH and 40 hours and tested in Machine Direction (MD)
- For TB contents from 3 up to 20 wt.%, TS presented values of 41 46 MPa and the Elongation was around 2.5%, therefore those values are similar to pure PLA.
- This performance of cast films can fit in applications having UV blocking properties

	TB content	Thickness	SD	Tensile Modulus	SD TM	Tensile Strength	SD TS	Elongation	SD
	wt.%	μm	mm	MPa	MPa	MPa	MPa	%	%
PLA-0	0	118	0.01	2385.27	82.10	49.17	1.02	2.62	0.13
PLA-1	3	121	0.01	2015.99	151.22	44.75	1.62	2.99	0.13
PLA-2	10	125	0.01	2593.41	156.13	46.23	3.63	2.61	0.33
PLA-5	15	138	0.01	2722.38	82.88	44.80	2.69	2.64	0.30
PLA-3a	20	145	0.01	2878.00	84.56	40.59	4.05	2.09	0.23
PLA-3b	20	139	0.00	2759.42	71.79	48.15	1.77	2.52	0.18
PLA-4	40	-	-	-	-	-	-	-	-



Differential Scanning Calorimetry - DSC

The DSC analysis was done between 20 °C and 200 °C, at a rate of 10 °C/min

		Heat 1							He	at 2			1.0
TB Content	Behavior after casting							Innate behavior					0.8 -
wt.%	Tg-1	ΔHc-1	Tc-1	ΔHm-1	Tm1-1	Tm2-1	Tg-2	ΔHc-2	Tc-2	ΔHm-2	Tm1-2	Tm2-2	
	°C	J/g	°C	J/g	°C	°C	°C	J/g	°C	J/g	°C	J/g	0.6 -
0	63.5	33.7	123.8	36.9	164.5	-	61.5	35.7	124.0	36.7	160.3	-0	(B//N) 0.4 -
3	63.7	34.2	113.6	37.1	162.6	168.8	61.3	33.2	114.3	38.5	163.0	169.1	at Flow
10	63.9	31.9	113.2	33.0	162.4	168.9	61.7	32.3	113.2	36.1	162.3	168.7	±
15*	57.7	27.1	105.1	33.3	151.6	167.5	61.3	26.8	105.1	36.7	152.0	167.5	0.0 -
20	63.3	26.2	111.6	32.1	161.7	168.5	61.3	26.9	111.2	32.7	161.3	168.2	-0.2 -
20 (CB)	63.6	25.2	109.0	25.2	162.0	168.3	61.9	25.0	109.5	29.5	161.9	168.3	-0.4
40	63.1	20.5	109.1	27.7	159.8	166.9	60.8	21.4	109.2	26.6	159.2	166.3	0 Exo Down



Tg-1 and Tg-2, remained unchanged for all formulations.

Tm, for both heating cycles, remained also unchanged with increasing TB content.

Tc, for both cycles, decreased with increasing TB content: TB particles play the role of nucleating agent for crystallization

*15% TB was obtained by dilution 40% TB. Therefore the PLA was extruded 2 times and a possible slight degradation took place (normal for PLA)



Differential Scanning Calorimetry - DSC

Theoretical melting enthalpy of a 100% crystalline PLA is $\Delta H_{100\% PLA}$ = 93 J/g.

The crystallinity was calculated as follows: X % = Δ H / (1 – TB%) Δ H _{100% PLA} Where TB% is: 0, 0.03, 0.1, 0.15, 0.2 and 0.4 corresponding at 0, 3, 10, 15, 20 and 40% TB in PLA

The PLA and PLA/TB crystallize during the heating, therefore the materials were not fully crystallized during the film casting process. The calculated crystallization % is presented in red in the figure.

Increasing TB%, from 3 to 40 wt.%, increased slightly the crystallinity of cast films from 3.5 up to 12.9%. Therefore, TB played a role of nucleating agent.

Very similar values were obtained for the cycle Heat 2

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Heat 1: cycles comparison (Heat 2 cycles presented very similar trend)

Differential Scanning Calorimetry - DSC

	Thermal Black Content wt.%/100		Heat 1 Behavior after casting								
		Tg-1	ΔHc-1	Xc H1	Tc-1	ΔHm- 1	Xm H1	Tm1-1	Tm2-1		
		°C	J/g	%	°C	J/g	%	°C	°C		
PLA-0	0	63.5	33.7	36.2	123.8	36.9	39.7	164.5	-		
PLA-3a TB	0.2	63.3	26.2	35.2	111.6	32.1	43.1	161.7	168.5		
PLA-3b CB	0.2	63.6	25.2	33.9	109	25.2	33.9	162.0	168.3		

PLA / 20 wt.% TB and PLA / 20 wt.% CB presented very similar thermal behaviors, i.e. similar Tg and Tm.

Exception for the crystallization behaviors: Tc and crystallinity content were lower for PLA-CB composite.

Reason: CB particles, being much smaller than TB particles, around 50 nm compared at 230-250 nm, at 20 wt.% CB there are a higher number of particles compared at 20 wt.% TB.

Therefore, at the same wt.% content, CB would accelerate the crystallization moment while hindering the crystal growing.



Characterization of PLA - TB cast films SEM morphology



Characterization of PLA - TB cast films SEM morphology



Characterization of PLA - TB cast films SEM morphology



Bi-axial stretching of PLA-TB films

Objective:

To test the processability of PLA-TB cast films in bi-axial stretching process and to evaluate the performance of obtained stretched films using similar characterization methods as for cast films.



Biaxial stretcher apparatus (Karo IV from Bruckner)



Clamping section of the stretcher

Stretching conditions:

Pre-heating time: 30 sec Pre-heating temperature: 100 °C



20 wt.%TB films obtained at different stretching rates

Bi-axial stretching Characterization of stretched PLA-TB films

Mechanical testing:

- Tensile properties using ASTM D882 18: Standard Test Method for Tensile Properties of Thin Plastic Sheeting
 5 kN cell, 100 mm grips distance, 10 mm/min speed
- All samples were conditioned at 23 °C, 50% RH and 40 hours and tested in Machine Direction (MD)
- PLA-TB bi-axial stretched films presented increasing tensile strength and modulus at increasing TB from 3 up to 20 wt.% (comparison at the same stretching ratio)
- PLA-TB bi-axial stretched films demonstrated similar and higher performance compared at pristine PLA
- PLA-TB bi-axial stretched films can replace pure PLA in applications such as laminations, industrial packaging etc.

Stratch ratio	Thermal Black	Thickness	SD Thickness	Tensile Modulus	SD TM	Tensile Strength	SD TS	Elongation	SD
Stretch ratio	content wt.%	μm	mm	MPa	MPa	MPa	MPa	%	%
2x2	0	35.0	0.00	2232.6	158.6	43.7	5.1	5.8	2.3
5x5	0	12.0	0.00	1024.3	270.3	18.9	5.1	2.9	0.6
2x2	3	36.0	0.01	2242.3	157.1	37.3	2.5	3.2	0.4
5x5	3	10.0	0.00	1207.5	206.9	18.9	2.9	3.2	0.8
2x2	10	33.0	0.00	2702.6	397.8	44.9	6.6	2.5	0.2
5x5	10	19.0	0.02	1993.4	353.7	29.8	5.7	4.6	2.1
2x2	20	33.0	0.00	3301.1	340.1	51.6	2.0	2.6	0.3
5x5	20	10.0	0.00	1627.9	141.7	22.8	2.1	2.9	1.3
2x2	20 (CB)	41.0	0.00	3241.8	175.3	56.0	2.5	2.5	0.1
5x5	20 (CB)	17.0	0.00	1815.9	630.6	28.2	5.9	2.5	0.3



Bi-axial stretching DSC of stretched PLA-TB films



Bi-axial stretching DSC of stretched PLA-TB films

<u>Tg of bi-ax films</u>: decreased to 59 °C compared to cast films (64 °C); <u>Tm of bi-ax films</u>: similar or slightly lower compared to cast films <u>Tc of bi-ax films</u>: decreased compared to cast films due to crystals developed rapidly during stretching at 100 °C and 1 m/min

	TD Contont		Heat ²	l: on bi-a	cially stre	tched fil	ms		
Stretch ratio	wt.%	Тg	ΔHc	Xc	Тс	Xm	ΔHm	Tm	X%
		°C	J/g	%	°C	%	J/g	°C	From bi-ax
cast	0	63.5	33.7	36.2	123.8	39.7	36.9	169.0	NA
2 x 2	0	58.7	28.7	30.9	104.5	38.9	36.2	167.6	8.1
5 x 5	0	58.9	6.5	7.0	99.3	44.1	41	167.2	37.1
cast	0.03	63.7	34.2	37.9	113.6	41.1	37.1	168.8	NA
2 x 2	0.03	58.6	23.5	26.1	95.8	39.4	35.5	166.5	13.3
5 x 5	0.03	NA	1.7	1.9	82.6	43.3	39.1	166.5	41.5
cast	0.1	63.9	31.9	38.1	113.2	39.4	33	168.9	NA
2 x 2	0.1	58.3	15.1	18.0	92.1	41.9	35.1	166.3	23.9
5 x 5	0.1	NA	NA	0.0	NA	46.2	38.7	166.6	46.2
cast	0.15	57.7	27.1	34.3	105.1	42.1	33.3	167.5	NA
2 x 2	0.15	58.9	11.2	14.2	91.2	43.8	34.6	166.4	29.6
5 x 5	0.15	NA	NA	0.0	NA	52.1	41.2	166.7	52.1
cast	0.2	63.3	26.2	35.2	111.6	43.1	32.1	168.5	NA
2 x 2	0.2	58.4	8.3	11.2	90.4	46.4	34.5	166.2	35.2
5 x 5	0.2	NA	NA	0.0	NA	55.8	41.5	166.1	55.8
cast	0.2 (CB)	63.6	25.2	33.9	109	33.9	25.2	168.3	NA
2 x 2	0.2 (CB)	58.9	1.6	2.2	93.8	44.2	32.9	166.4	42.1
5 x 5	0.2 (CB)	NA	NA	0	NA	49.3	36.7	168.5	49.3

Crystallinity content increases with TB % and stretching degree.

High crystallinity was developed during stretching, that can further could conduct to pores formation while fingerprinting the films permeability performance



Bi-axial stretching SEM of stretched PLA-TB films

x 2,500 surface



2x2

5x5

Bi-axial stretching SEM of stretched PLA-TB films



x 5,000 surface

PLA - TB Films blowing

Objective:

To test two (2) optimal PLA-TB formulations in film blowing process using the NRC's semi-industrial scale film blowing line. The obtained films were tested using the same methods as for cast and bi-axial stretched films.



Formulation	Thickness
PLA 4032D reference	around 50 microns
10 wt.% TB	around 90 microns





Film blowing Characterization of blown PLA-TB films

Tensile testing (ASTM D882 - 18):

- Samples were tested in MD direction
- In terms of TS & thickness, the blown film with 10 wt.% TB demonstrated adequate properties for single use packaging and protective films. The elongation is low, further improvement can be done. It has equivalent properties to the PLA / 20 wt.% TB film obtained in biaxial stretching.

Blown film	Thermal Black	Thickness	SD	Tensile Modulus	SD TM	Tensile Strength	SD TS	Elongation	SD %
BIOWITHIII	content wt.%	μm	mm	MPa	MPa	МРа	MPa	%	
PLA-0, 4032D, Ref*	0	35	NA	NA	NA	48 MD (82 TD)	NA	5.7	-
PLA-2, 10 wt.% TB	10	11	0.01	3585.3	470.0	33.7	2.1	1.60	0.19

*https://www.ncbi.nlm.nih.gov/pmc/articles/PMC8705587/

Morphology and Microstructure by SEM (x 2,500 surface):







Film blowing Characterization of blown PLA & PLA-TB films

Differential Scanning Calorimetry analysis:

Only a heating cycle was applied with the purpose to evaluate the crystallinity in film blowing process.

The Δ Hc and Δ Hm of blown PLA films are roughly similar.

This means that the blown film obtained contain low levels of crystallinity, due to the low blow-up ratio used (BUR) to maintain the bubble, around 1.4.

	TB Contont	Heat : Behavior after casting								
Process	ID Content	Tg∘	ΔHc	Tc	ΔHm	Tm1	Tm2			
	WU.70	Ċ	J/g	°C	J/g	°C	°C			
Cast	0	63.5	33.7	123.8	36.9	164.5	-			
Blown	0	57.8	28.0	130.0	38.4	165.5	-			
Cast	10	63.9	31.9	113.2	33.0	162.4	168.9			
Blown	10	58.6	32.1	112.0	34.7	161.9	168.5			



Biodegradability	Phases : Date :	Start-up 0 days	1 st validation 20 days	2 nd validation 37 days
	PLA / 0 wt.% TB Cast			
Method:		PLA-O	85	84
 ASTM D5338 Test at 58 °C, 55 days, 50% H.R. Monitor the Oxygen consumption Used references: 	PLA / 3 wt.% TB Cast	PLA-1	B4	- B2
 Positive: Cellulose-Sigma Aldrich-310697 Pure PLA Compost: Peat and Bark Biofor[®] 	PLA / 3 wt.% TB Bi-axe 2x2	PIA -1 2×2	A7	AB
3 samples were tested from each film. The samples were monitored at an time interval of 20 days (see table).	PLA / 10 wt.% TB Cast	PLA - 2-100	88	c1

PLA / 20 wt.% TB

Cast

PLA / 20 wt.% TB Bi-axe 5x5

PLA-3A

PLA-3A

SX5

End of the test 55 days

C3

00

C3

C6

The final visual aspect - the level of disintegration is presented in the last column.

All the tested PLA-TB films disintegrated in a similar way compared to pristine PLA.

Biodegradability PLA-TB films



Oxygen consumption is presented as a function of time.

Oxygen consumption is proportional to CO_2 produced during the samples degradation.

All PLA/TB films demonstrate an O_2 consumption equal or slightly higher than the pristine PLA cast film and less compared at positive control, the cellulose.

All PLA/TB films present similar Aerobic Biodegradation, slight differences are due to TB content, porosity and the thickness of samples.



Barrier properties of PLA - TB films

			ASTM D-3985		ASTM F2714-08 2013	ASTM F-1249-06	
Sample	TB content wt.%	Film Thickness µm	OTR cc/m ² .day	SD	OTR cc/m ² .day	WVTR g/m ² .day	SD
PLA 4032D NatureWorks [®] LLC*	0	25	675	-	-	380	-
Cast	3	121	148	1	NA	40.1	0.9
Bi-axe 2 x 2	15	40	566	67	NA	123.2	23.6
Bi-axe 3.5 x 3.5	15	20	† 7721	1847	NA	337.5	34.6
Bi-axe 5 x 5	20	10	NA	NA	65493, 1542, 166554	781.5	76.6

* Data on PLA 4032 permeability from TDS from manufacturer

OTR & WVTR increased with films crystallinity. Crystallinity development in PLA-TB films depends on parameters used for cast and bi-ax stretching processes and TB content. The PLA-TB crystallinities can be <u>fine-tuned to obtain other values for OTR and WVTR</u>

Conclusions OTR:

An industry rule-of-thumb is that a film material is considered a **"high oxygen barrier" if its OTR is less than 15.5 cc/m²/day**. Pristine reference PLA film: has **a good permeability to oxygen (*)**, i.e. 675 cc/m²/day.

PLA/TB films: OTR values increased with TB content and the stretching, i.e. proportionally to the crystallinity content of tested films. PLA/3 wt.% TB cast-film (121 μ m) has a low permeability to O₂ while Bi-ax PLA/15 wt.% TB, 2x2 (40 μ m), has an OTR similar to pure PLA.

Conclusions WVTR:

A polymer film is considered water vapor barrier for WVTR values of 4 - 10 g/m².day

Pristine reference PLA film: has rather good permeability to water vapors, i.e. 380 g/m².day.

PLA/3 wt.%TB has a low WVTR but this value is a function of film thickness, 121 µm.

PLA/TB films: WVTR values increased with TB content and the stretching, but remained near the value of PLA reference film.



Cast, bi-axial stretched, and blown films **Optical properties**

Bi

Transparency (transmittance):

- Measurement according to ASTM D1746-09
- Haze-guard plus from BYK Gardner was used
 - No sample \rightarrow 100% transparency
 - Blocked light path \rightarrow 0% transparency

Conclusions:

- All cast and blown films with fillers have 0 transparency
- For bi-axial stretched films with filler: ٠
 - At low stretch ratio (2x2) films are not transparent independent of filler content
 - At high stretch ratio (5x5) and minimum filler content semi-transparent films an be achieved
- High potential to block the UV •

Biax film	Average T Value	SD	Blown film	Average T Value	SD
PLA_0_2x2	95.7	0.0	PLA_0	95.0	0.1
PLA_0_5x5	95.7	0.1	PLA_2	0.0	0.0
PLA_1_2x2	5.6	1.3		1	
PLA_1_5x5	63.4	4.3	Cast film	Average T	SD
PLA_2_2x2	0.0	0.0		Value	-
PLA_2_5x5	12.6	2.3	PLA_0	95.5	0.1
PLA_3a_2x2	0.0	0.0	PLA_1	0.0	0.0
PLA_3a_5x5	1.0	0.3	PLA_2	0.0	0.0
PLA_3b_2x2	0.0	0.0	PLA_3a	0.0	0.0
PLA_3b_5x5	0.4	0.3	PLA_3b	0.0	0.0
PLA_5_2x2	0.0	0.0	PLA_4	0.0	0.0
PLA_5_5x5	15.2	9.1	PLA_5	0.0	0.0



Conclusions

Performance vs. thickness of PLA / TB films from this project – for industrial applications:

- > PLA / TB films from cast extrusion: Thickness = 140 120 μ m, TS MD = 40 45 MPa, ϵ MD = 3 %
- PLA / TB films from biaxial stretching: Thickness = 10 35 μm, TS MD = 22 (2x2) 50 (5x5) MPa, ε MD = 3-5 %
- > PLA / TB from blown film process: Thickness = 11 μm, TS MD = 34 MPa, ε MD = 1.6 % (process to be optimized)

Commercial Biodegradable Mulch Films:

Mater-Bi[®] - Novamont

Starch material + compostable polyesters Thickness = 15 μ m TS (MD/TD) = 41.0/34.2 MPa ϵ = 400% MD

Commercial PURE PLA films: (TDS data)

T-shirt bags and waste bags, thin film for laminate applications and packaging

Thickness: $60 \ge 30 \ge 15$, TS MD = $30 \cancel{47} \cancel{100}$ MPa, ϵ MD = $450 \ge 290 \ge 180\%$

Ecovio[®] - BASF

Biodegradable Ecoflex + PLA Thickness = 20 μ m TS (MD/TD) = 31.2/22.5 MPa ϵ = 100% MD

According to EN 13655: an elongation of 180% is expected for <u>black conventional mulches</u> with thickness less than 30 μ m.

Elongation of PLA / TB films can be easily improved by adding, for example, Ecoflex (BASF)

PLA / TB films demonstrated to be appropriate for many types of applications.

PLA / TB could be a lower-cost choice compared to pristine PLA (TB price is lower).





THANK YOU!

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