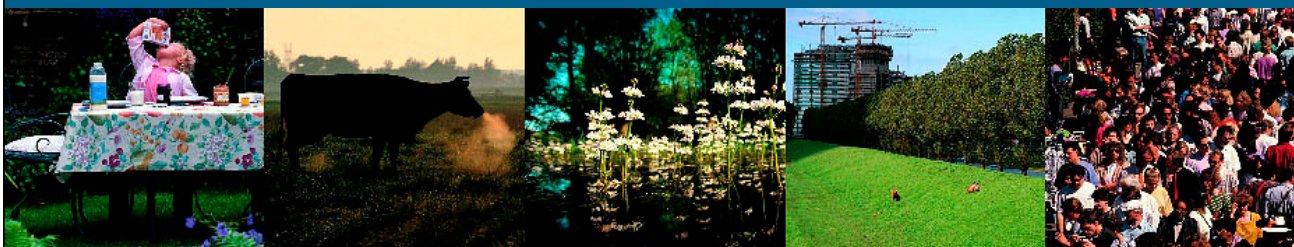


Processing window of Natural fibre-PLA composites

Martien van den Oever
Prof. dr.-ing. Jörg Müssig
Benjamin Beck

6th N-FibreBase Congress, Essen, 22 September 2008



Introduction

■ Natural fibre-Polyolefins:

- NMT, Fibre mat based (door trims, etc.)
- WPC (decking, siding, etc.)
- Injection moulding (entering market *¹)

■ Natural fibre-Poly(lactic acid):

- PLA has properties that could make it a competitor of PP



*¹ Carus & Gahle, Reinf Plast, April 2008

Background: Moisture sensitivity of PLA

- PLA hydrolyses in the presence of water in the molten state
- Moisture content < 0.025% *2
- Agrofibres: 6-10% moisture at regular RH cond.

Worst case: Maximum cleaving of PLA chain

- Assume PLA of $M_w = 245,000$ Da
 - 30% natural fibre in PLA
 - All water present causes chain scission
- ➔ Resulting in:

	Moisture:PLA (wt.%)	# Cleaves (-)	Mw (Da)
Pure PLA	0.025	3.4	72,000
Fibre, 6% moisture	2.6	350	700
Fibre, 10% moisture	4.3	580	420

Experimental: material

- PLA NatureWorks 4042D

- 4-8 h dried at 80°C

- Ramie

- Flax

- Cotton

- Dried at 105°C for 16 h
 - not-dried

best practice
worst case



AGROTECHNOLOGY &
FOOD SCIENCES GROUP
WAGENINGENUR

Experimental: kneading

- Under flow of pre-dried N
- 10-50 wt.% fibre in PLA
- Set Temperature: 175°C
- Rotor speed: 100 RPM
- Total compounding time: 13 min



AGROTECHNOLOGY &
FOOD SCIENCES GROUP
WAGENINGENUR

Experimental: injection moulding + moisture analysis

- Granules dried at 80°C for 4-8 h
- Injection moulding to flexural test bars
4 mm * 10 mm * 80 mm
- Moisture analysis of fibres:
 - Undried fibres: weight reduction after 16 h at 105°C
 - Dried fibres: Mettler Toledo DL39 Karl Fisher Coulometer at 200°C

Experimental: Intrinsic viscosity + MFI

- IV:
 - Viscosity of PLA in chloroform
 - Extrapolated to 0 concentration
 - IV relates to M_v according to Mark-Houwink
- MFI: flow of material (g/10 min) through a standard die
 - Pre-drying of granules at 80°C for 4 h and for 24 h
 - 190°C
 - 2.16 kg
 - The higher MFI, the lower viscosity, the more degradation

Experimental: SEM + Flexural + Charpy impact

- SEM, Jeol JSM-6300 F:
 - Fracture surface resulting from Charpy test
 - 10 nm layer of platinum, Oxford CT1500
- Flexural testing, Zwick universal testing machine:
 - Support span=16 * thickness
 - $v_E = 2\text{mm/min}$, $v_\sigma = 10\text{mm/min}$
- Charpy Impact, Ceast pendulum:
 - 4J hammer
 - 2.9 m/s
 - Flatwise, unnotched

Results: Intrinsic viscosity (IV) and Molecular Weight (M_v)

Mark-Houwink: $IV = 0.0153 * M_v^{0.759}$ *3

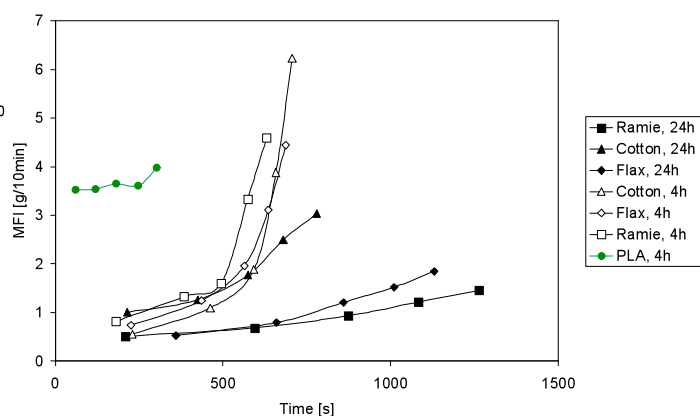
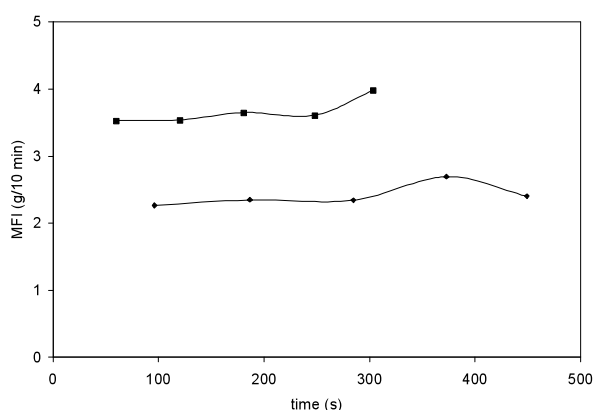
	IV (ml/g)		M _v (Da)	
	Dried	Undried	Dried	Undried
Blanc	188.4		245,000	
Kneaded	129.4		149,400	
30% Ramie	105.6		114,300	
30% Flax	126.7	117.1	145,300	131,000
30% Cotton	62.9	61.6	57,800	56,200

Results: Mv, worst case vs. experimental values

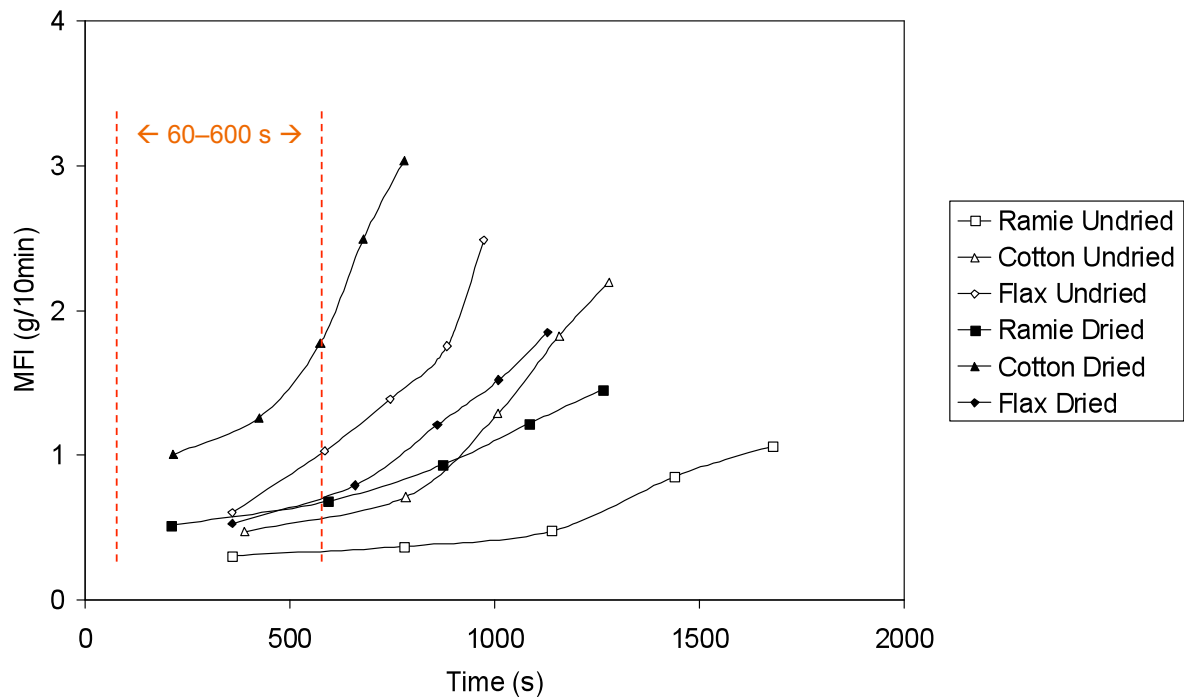
- Dried fibre: 0.22-0.40 wt.% moisture
- Undried fibre: 6.6-9.0 wt.% moisture

Calculated Worst Case		Experimental	
Dried	Undried	Dried	Undried
Blanc		245,000	
Kneaded	72,000	149,400	
30% Ramie	19,310 630	114,300	-
30% Flax	10,590 467	145,300	131,000
30% Cotton	14,360 636	57,800	56,200

Melt Flow Index = indicator for PLA degradation

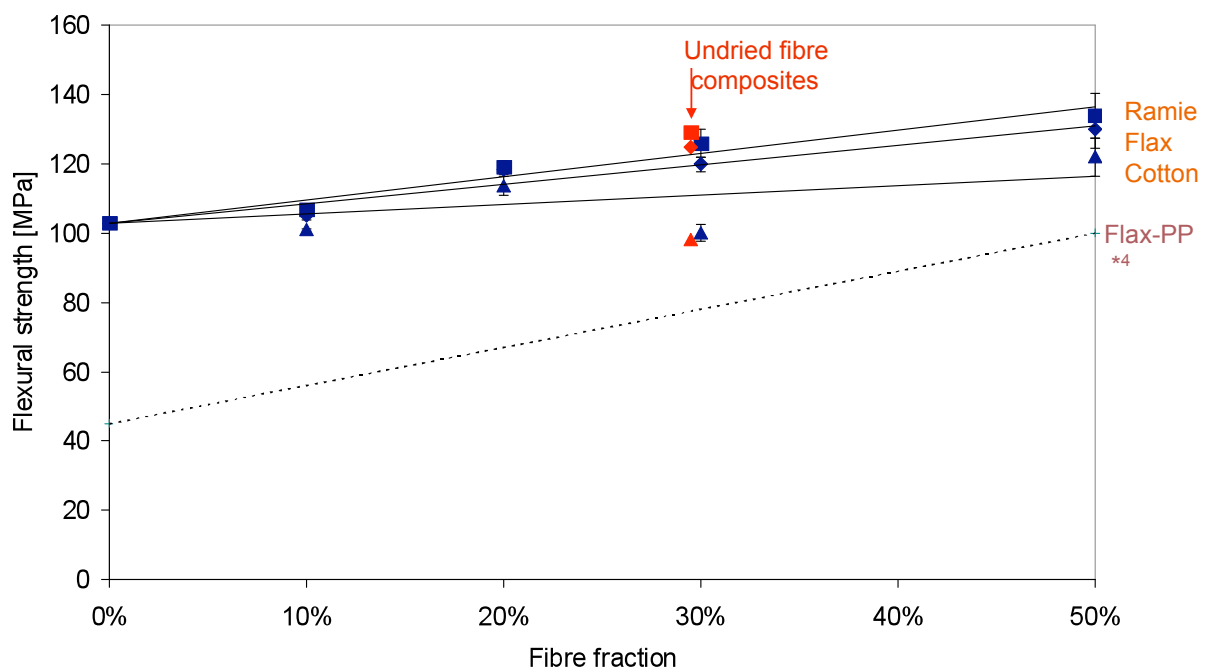


Melt Flow Index = indicator for PLA degradation



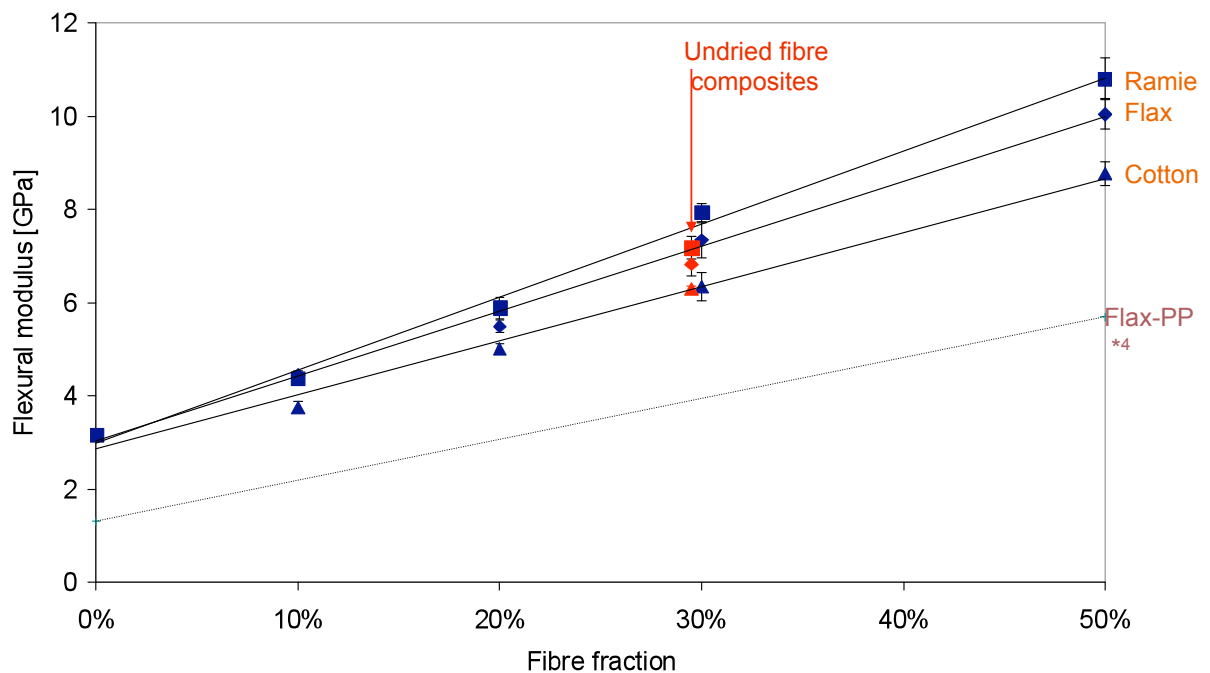
Typical processing times: 60–600 s.

Flexural Strength

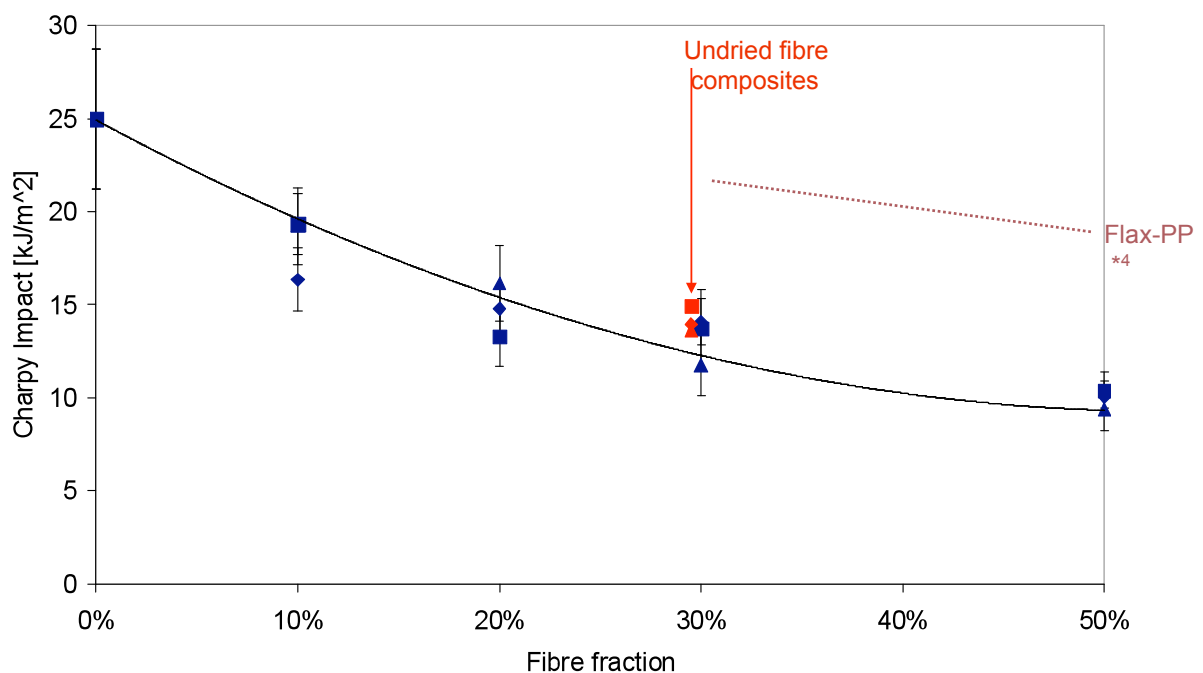


*4 Bos et al., Comp A, 37, 2006, 1591-1604

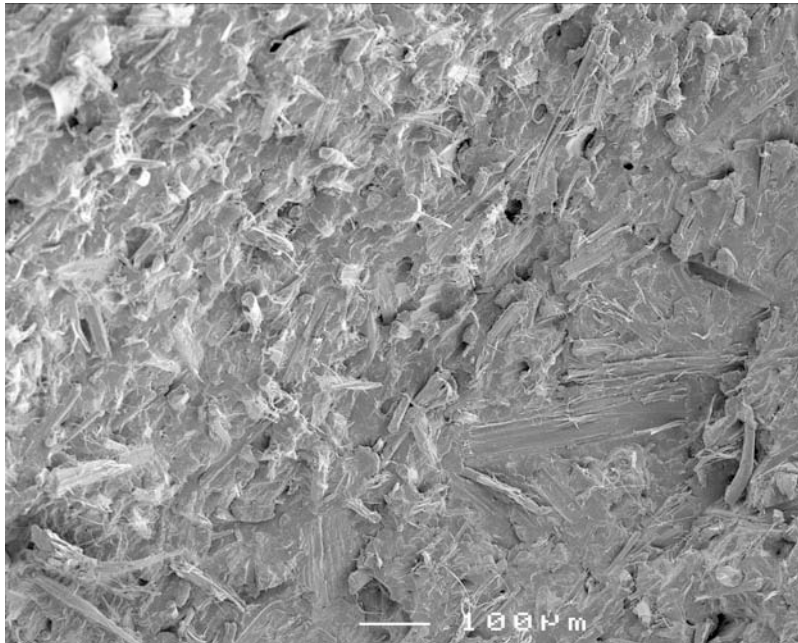
Flexural Modulus



Charpy Impact = energy absorption capacity



Scanning Electron Microscopy



30 wt.% Flax-PLA

- Fairly good
fibre-matrix
adhesion

Conclusions

- Undried natural fibres cause PLA chain scission during melt processing, though by far not to theoretical *worst case* level.
- Drying of natural fibres prior to compounding appears to be not critical for basic mechanical performance of PLA composites.
- MFI results indicate that natural fibre-PLA composites exhibit an onset point for degradation in the presence of too much water.
- Strength and stiffness properties of natural fibre-PLA composites is better than natural fibre-PP, impact strength is less good.

