Sustainable Production of Non-wood MFC Biocomposites for Compostable Personal Care and Packaging Applications

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TAPPI NANO DIVISION

Brief Update from Queensland…

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Australian spinifex grass is being turned into an innovative medical gel

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$2.6 million is being invested into Trioda Wilingi Pty Ltd to develop innovative medical gels using unique cellulose nanofibers extracted from Australian spinifex grass. Image source: Mark Marathon, CC BY-SA 4.0.

$2.6 million is being invested into Trioda Wilingi Pty Ltd to develop innovative medical gels using unique cellulose nanofibers extracted from Australian spinifex grass.

Trioda Wilingi Pty Ltd is a company born from the partnership of the University of Queensland and

https://newscop.com.au/2023/03/08/australian-spinifex-grass-is-being-turned-into-an-innovative-medical-gel/

Our visits to TAPPI Nano over the years have featured Spinifex

This is where that work is heading now

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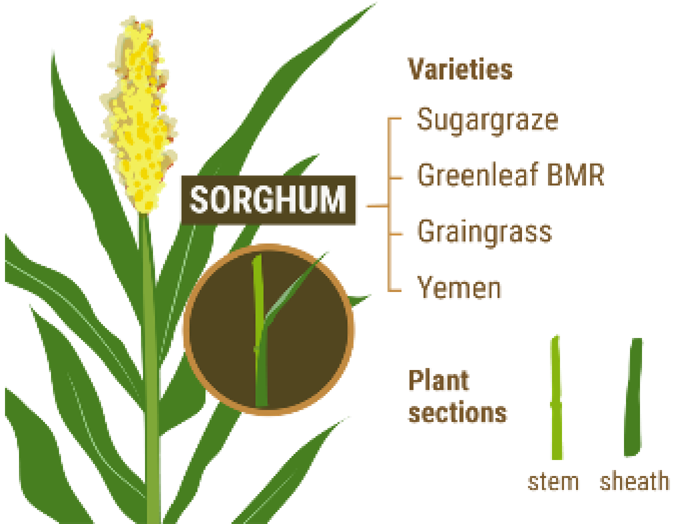
Overview

- Introduce sorghum as an attractive feedstock for ligno- CNF and MFC
- Twin-screw extrusion for sustainable ligno-MFC production

- **Application 1** – MFC-PHA composites for compostable razor handles

- **Application 2** – MFC-thermoplastic starch composites for compostable films

- Methods, results and discussion for each application
- Conclusions and acknowledgements



Sorghum Characteristics

- ✓ **Abundant** – 42 M ha cultivated in 105 countries around the world, sorghum is an ultra-resilient crop of increasing global importance (600,000 ha grown in Australia)[1]
- ✓ **Resilient** – drought and salinity resistant, excellent nitrogen usage efficiency[2]
- ✓ **Amenable** – high hemicellulose, low lignin content allows for sustainable micro/nano fibrillation[3]
- ✓ **Agronomically viable** – hundreds of genetic varieties and ability to be easily cultivated from seed[4]
- ✓ **Widely applicable** – potential production of bioenergy, biofuels, biochemicals, bio-based materials, food & feed

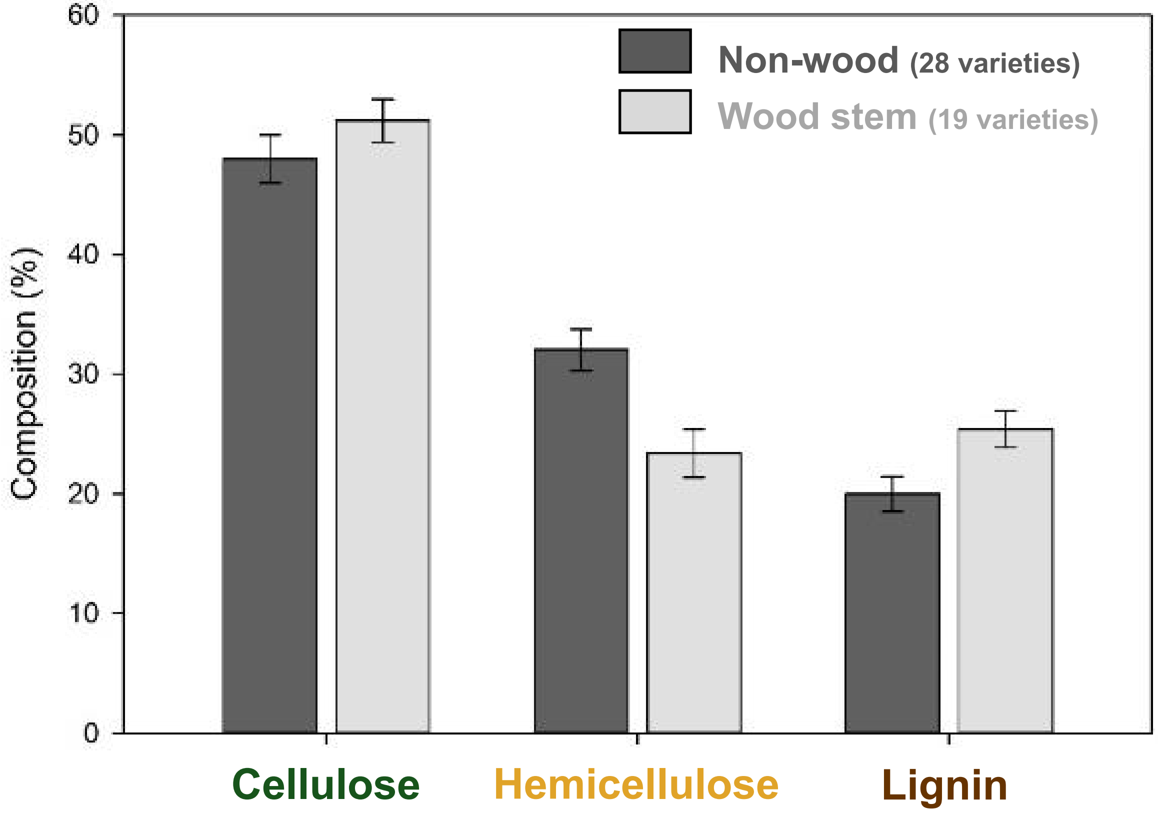
Sources:

1. FAOSTAT statistical database [Internet]. FAO, 2019. Available from: http://www.fao.org/faostat/en/#home
2. Borrell A, van Oosterom E, George-Jaeggli B, et al (2021) Chapter 5 - Sorghum. In: Sadras VO, Calderini DFBT-CPCH for MC (eds). Academic Press, pp 196–221
3. J. Pennells et al. 2021 'Sorghum as a novel biomass for the sustainable production of cellulose nanofibers', *Industrial Crops and Products*, 171, 113917
4. J. Pennells et al. 2022, 'Statistical genetics concepts in biomass-based materials engineering', *Frontiers in Bioengineering and Biotechnology*, 10, 1022948

Wood vs Non-wood Biomass

Biochemical composition

- Comparative study of 93 different biomass types

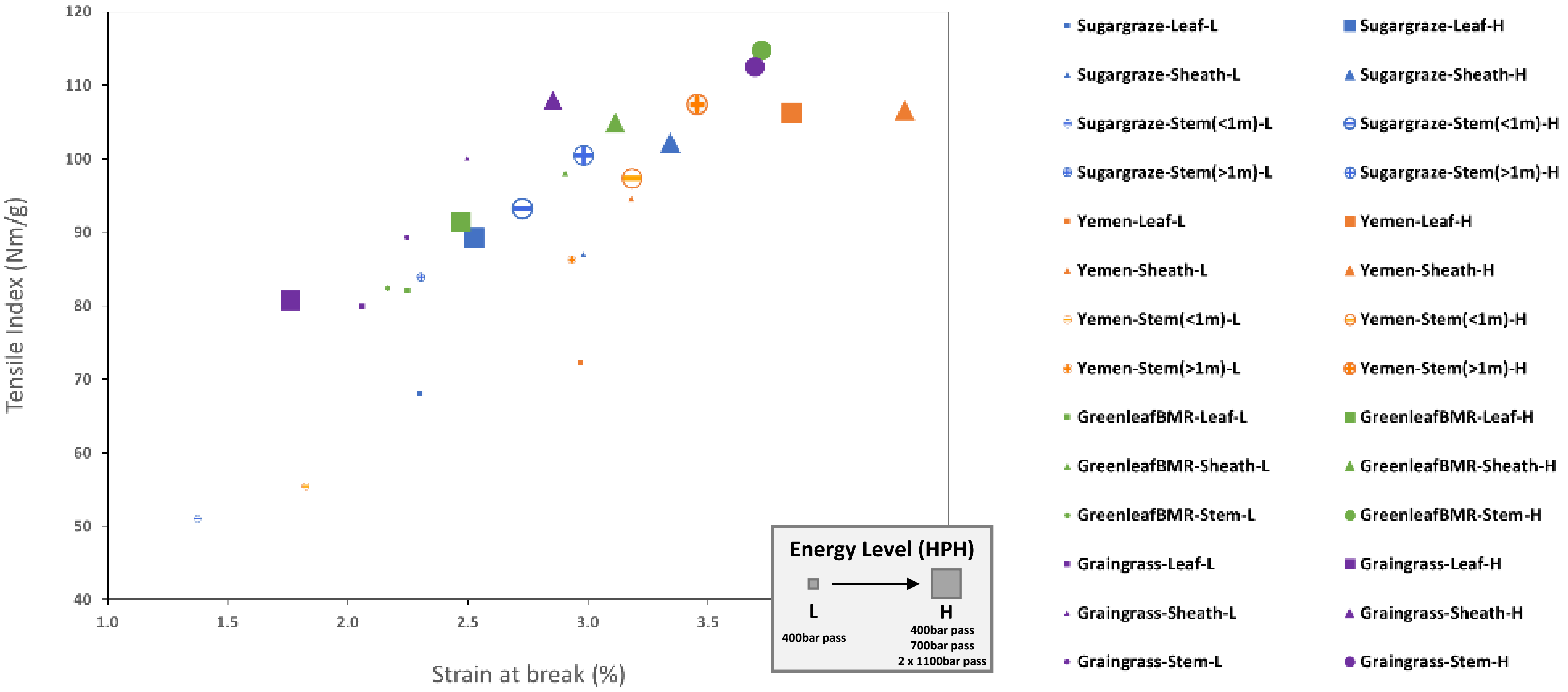


	Wood biomass	Non-wood biomass
Abundant	✓	✓
Renewable	✓	✓
Biodegradable	✓	✓
Recyclable	✓	✓
Non-toxic	✓	✓
High biomass density	✓	x
Established infrastructure	✓	x
Low lignin content	x	✓
High hemicellulose content	x	✓
Mild pulping conditions	x	✓
Growth on marginal land	x	✓/ x

Sources: Vassilev et al., (2012) *Fuel*
J Pennells et al., (2019) *Cellulose*

Highly Amenable to Fibrillation after Mild Pulping + Variety & Plant Section Matter

➢ **Processing:** Washing, Grinding, Chemical pre-treatment (NaOH, 2wt%, 80°C, 2hrs), Mechanical treatment (HPH: 'Low' or 'High' energy)



Sugargraze: Commercially grown variety with high sugar content

Yemen: Tall, thick stemmed variety originating from Yemen

GreenleafBMR: Thin stemmed Greenleaf variety with bmr6 gene modification

Graingrass: Fine stemmed primitive grain variety

J. Pennells et al. 2021 'Sorghum as a novel biomass for the sustainable production of cellulose nanofibers', Industrial Crops and Products, 171, 113917

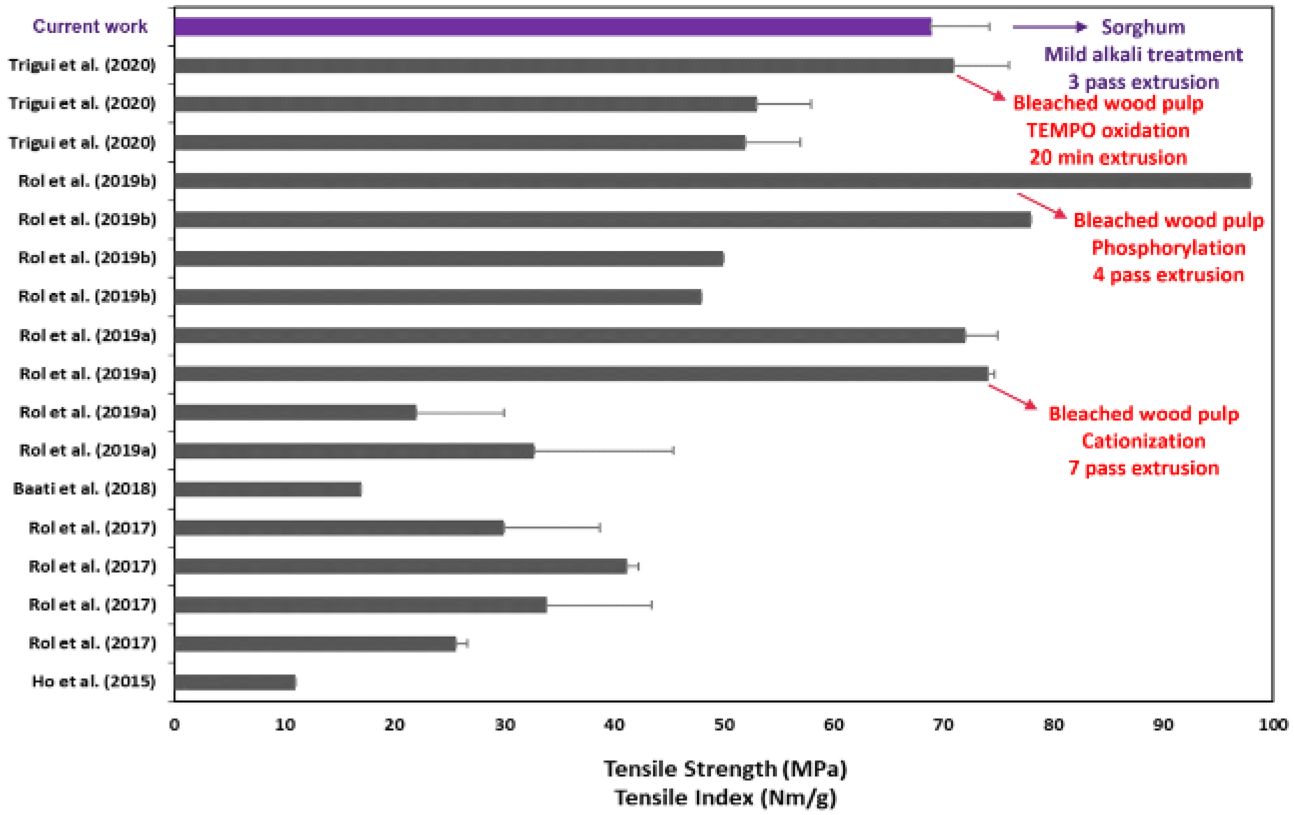
J. Pennells et al. 2022, 'Statistical genetics concepts in biomass-based materials engineering', Frontiers in Bioengineering and Biotechnology, 10, 1022948

Sorghum-derived MFC using Twin-screw Extrusion

We have found twin-screw extrusion to be very attractive in the preparation of non-wood MFC materials

- Scalable, continuous process
- High solids content (up to ~30%)
- Cost-effective, and it is a process very familiar to those working in the bioplastics and biocomposites sector

- Sorghum lignocellulose pulp can achieve similar quality to other wood-derived feedstocks which have necessitated more aggressive chemical pre-treatments



Sources:

1. T. T. T. Ho et al., Nanofibrillation of pulp fibers by twin-screw extrusion, *Cellulose* 22 (1) (2015) 421–433
2. F. Rol et al., Pilot-Scale Twin Screw Extrusion and Chemical Pretreatment as an Energy-Efficient Method for the Production of Nanofibrillated Cellulose at High Solid Content, *ACS Sustainable Chemistry and Engineering* 5 (8) (2017) 6524–6531
3. R. Baati, A. Magnin, S. Boufi, High Solid Content Production of Nanofibrillar Cellulose via Continuous Extrusion, *ACS Sustainable Chemistry and Engineering* 5 (3) (2017) 2350–2359
4. F. Rol, N. Belgacem, V. Meyer, M. Petit-Conil, J. Bras, Production of fire-retardant phosphorylated cellulose fibrils by twin-screw extrusion with low energy consumption, *Cellulose* 26 (9) (2019) 5635–5651
5. K. Trigui et al., Cellulose nanofibrils prepared by twin-screw extrusion: Effect of the fiber pretreatment on the fibrillation efficiency, *Carbohydrate Polymers* 240 (15) (2020) 116342

Materials and Methods

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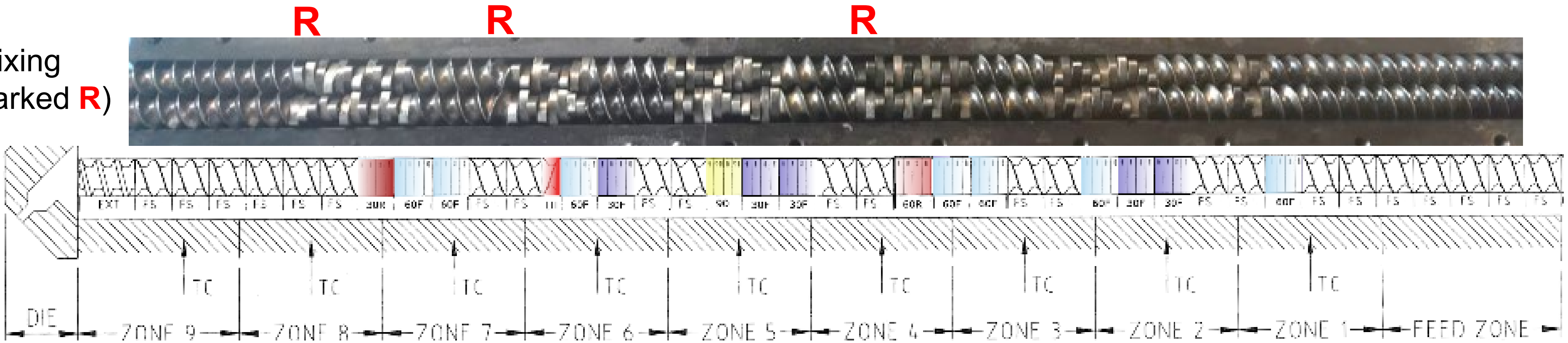
Sorghum-derived MFC using Twin-screw Extrusion

Dried, ground forage sorghum (Surgargraze™) was **pulped** (**1% or 2% NaOH** (w/v) at 80°C for 2h) and rinsed extensively
Fibrillated via twin-screw extrusion at ~19% (w/w) solids. The resulting **MFC** “crumb” extrudate was at **~23% (w/w) solids**

3 reverse mixing sections (marked **R**)



Haake Polylab 16 mm 40:1 L/D co-rotating twin-screw extruder – adiabatic conditions
Screw speed: 80 rpm

Application 1 – Compostable Razor Handles

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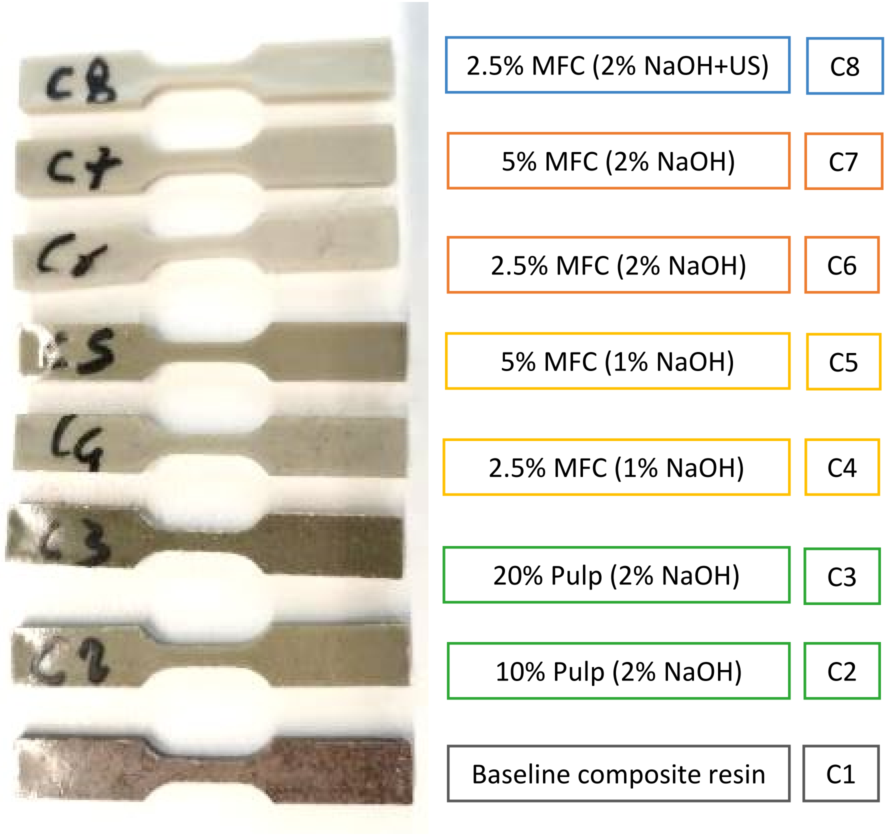
Materials/Formulations: PHBV Enmat Y1010, Plasticiser
MFC were incorporated to the PHA powder as per below:

- Extruded **MFC** diluted down to 8% (w/w). One sample treated with an **ultrasonic probe**
- PHBV dried overnight at 60°C
- Blend PHA + diluted MFC + Plasticiser for a couple of minutes in kitchen blender at 3000rpm
- Dry at 60°C for 48h before extrusion

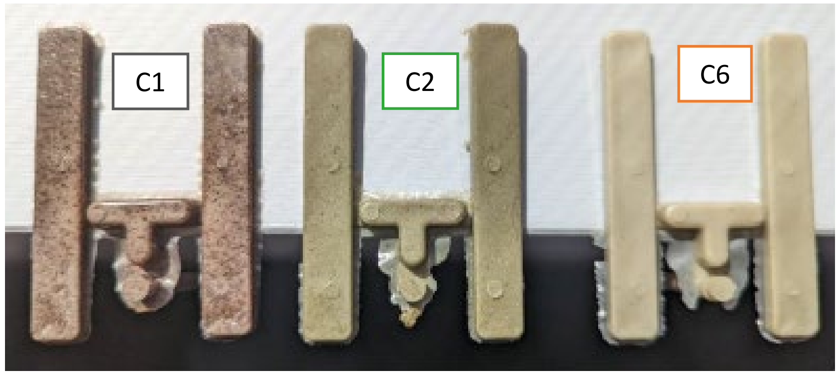
- Extrusion: Haake Polylab 16 mm 40:1 L/D co-rotating twin-screw extruder, screw speed: 170 rpm, temperature profile ranging from 140°C to 175°C (proven polymer nanocomposites screw config.)

Application 1 – Compostable Razor Handles

Moulding



Injection moulding parameters	
Plastification chamber temp	168°C
Injection Chamber temp	168°C
Nozzle temp	170°C
Mould temp	60°C
1st Injection Pressure	80 bar
1st injection pressure time	7 s
2nd injection pressure	90 bar
2nd injection pressure time	12 s
Cooling time	20 s

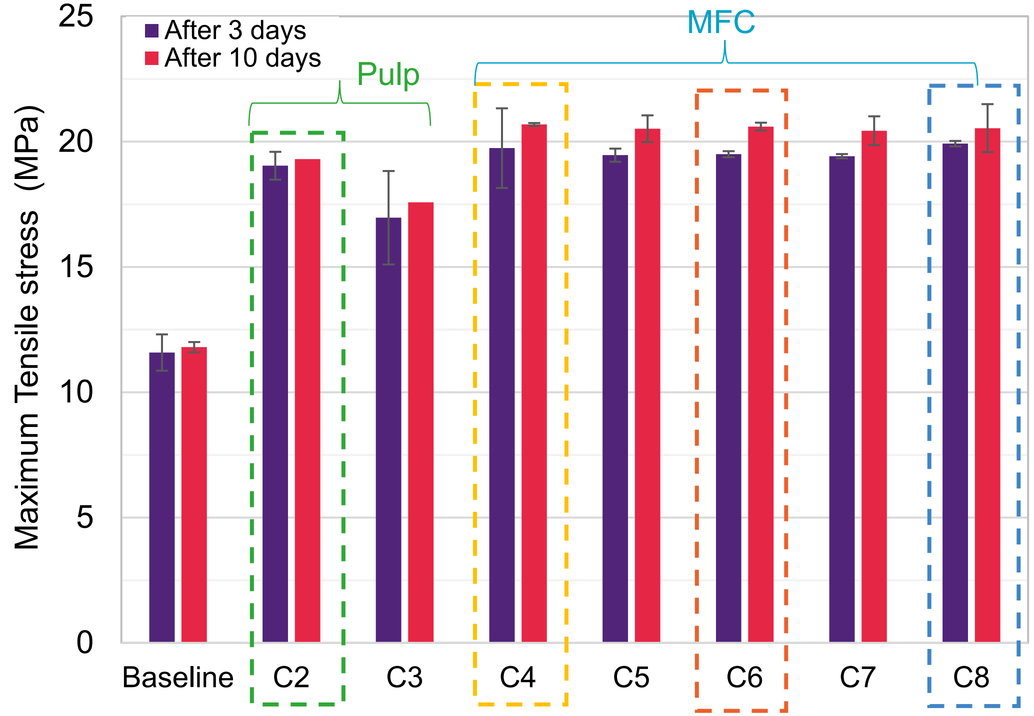


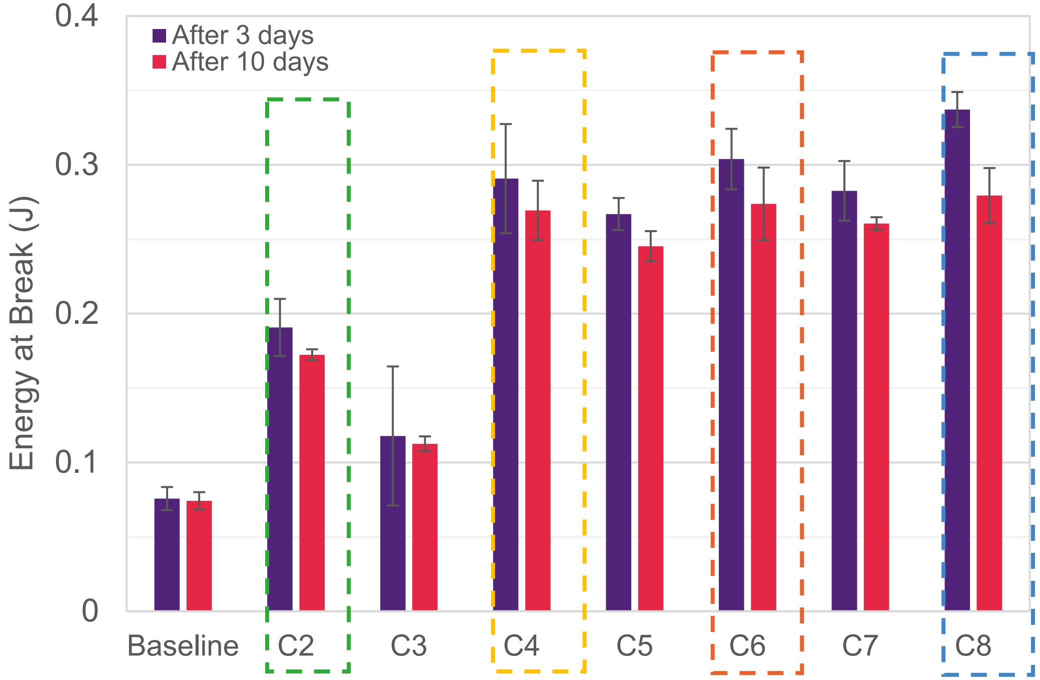
Tensile testing

- Injection-moulded dogbones (ATSM D638-22 Type V) aged at 23°C for 3 days and 10 days prior testing
- Instron model 5543 universal testing machine with a 500 N load cell and pneumatic grips
- Crosshead speed of 1 mm/min
- A minimum of five replicates

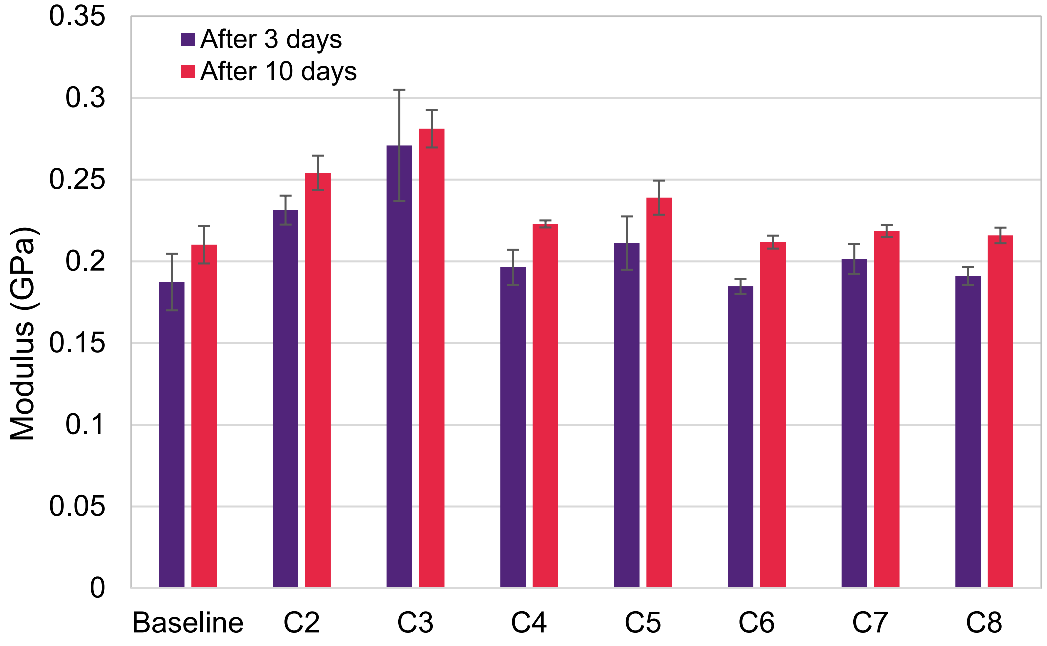
Impact testing

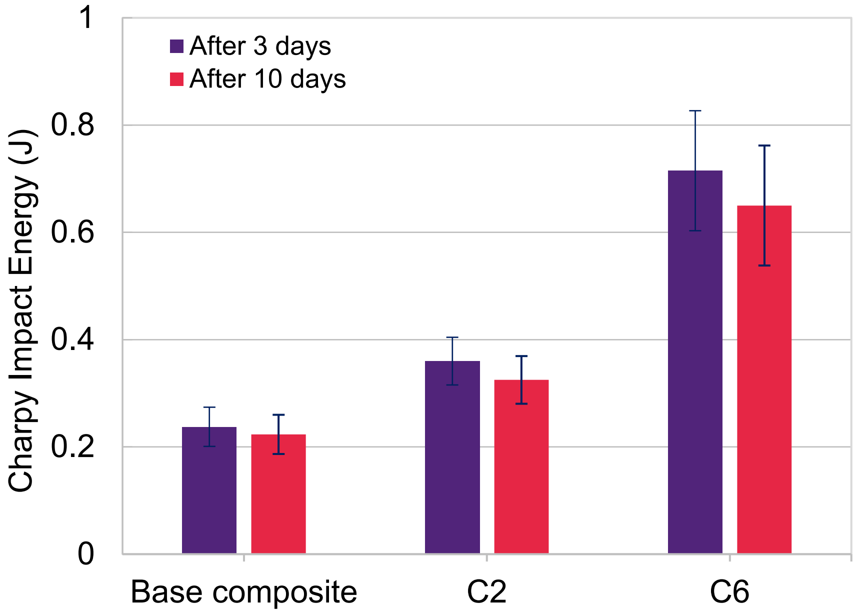
- Unnotched injection-moulded rectangular samples aged at 23°C for 3 days and 10 days prior testing
- Charpy impact tester fitted with a 1J load and a span of 40 mm
- A minimum of six replicates





2.5% MFC (2% NaOH+US)	C8
5% MFC (2% NaOH)	C7
2.5% MFC (2% NaOH)	C6
5% MFC (1% NaOH)	C5
2.5% MFC (1% NaOH)	C4
20% Pulp (2% NaOH)	C3
10% Pulp (2% NaOH)	C2
Baseline composite resin	C1

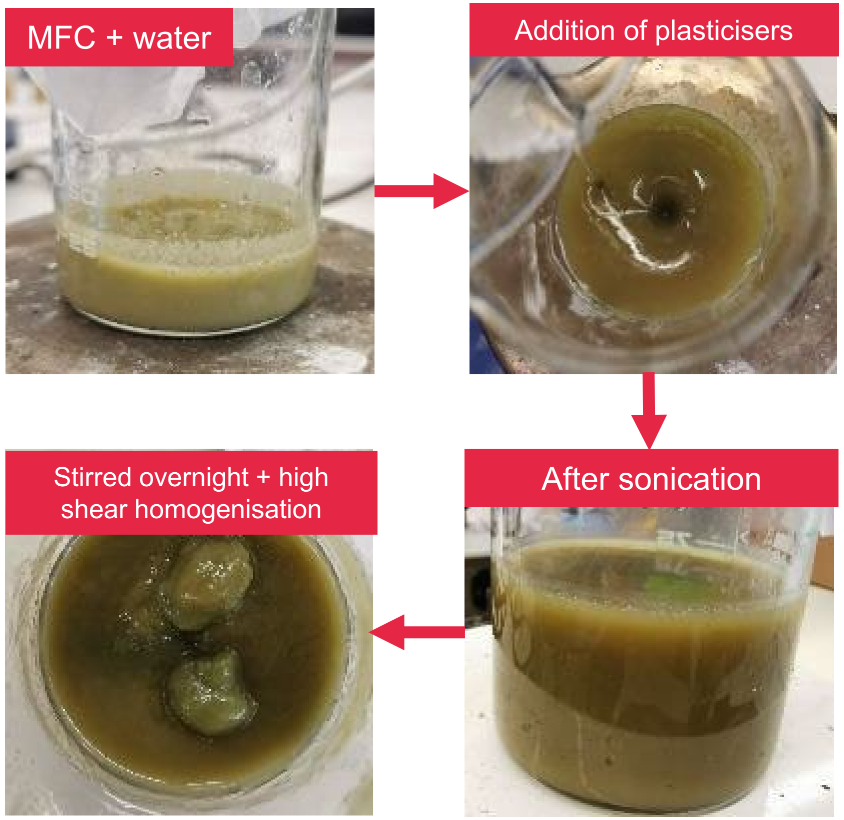




Application 2 – Low Cost Compostable Films

TPS composites – Feed material

Preparation of MFC suspensions



Preparation of composite starch feed mix

- **'Dry' blend mix:**
  Starch + additives (HPO*) weighed separately and mixed together for 2 min in a blender
- MFC suspension added to the 'dry' mix in the blender and mixed for another 2 mins until a homogenous 'light' powder blend was obtained

- Feed material prepared the day before extrusion
- Stored overnight in closed airtight container at room temperature

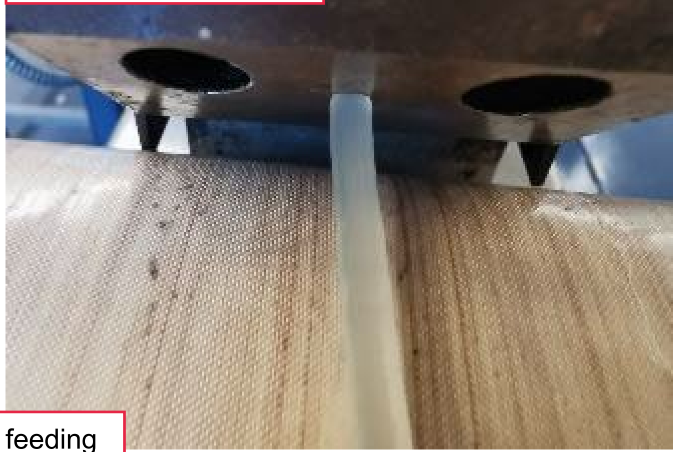
* Hydrogenated Palm Oil

TPS composites - Processing

Haake Polylab 16 mm 40:1 L/D co-rotating twin-screw extruder

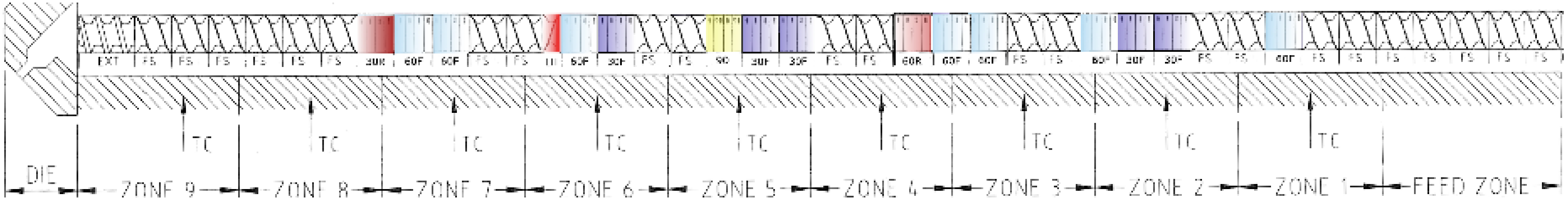
Single spring screw feeder – Starve feeding

3 mm single hole die



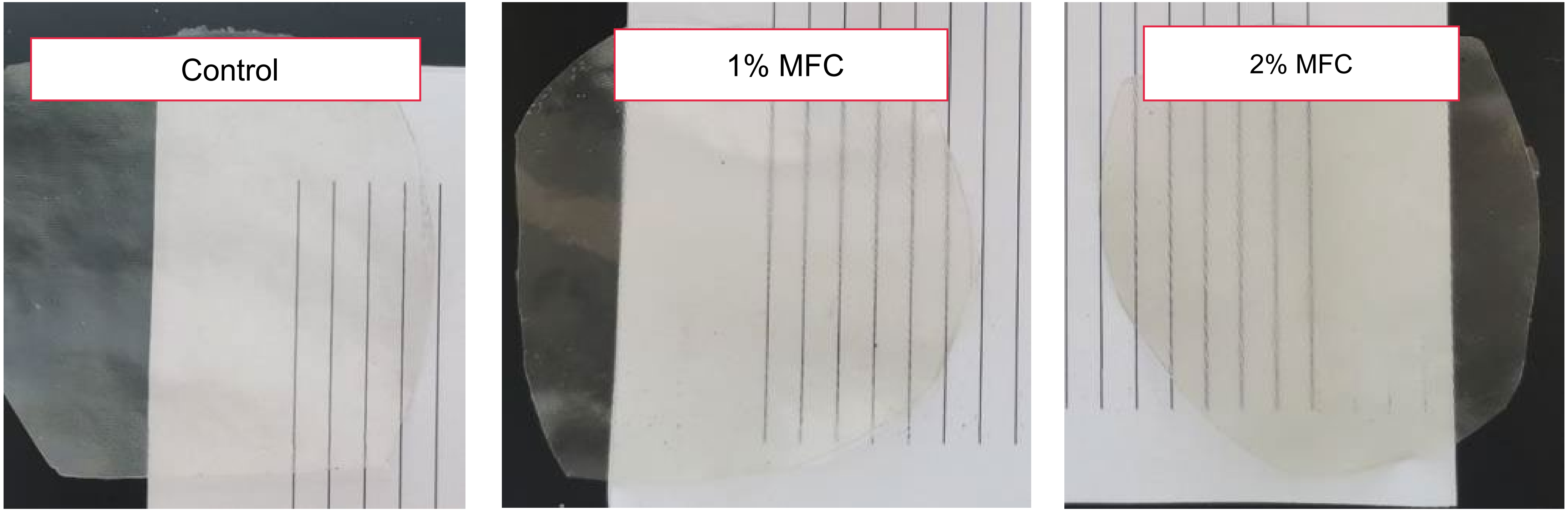
Screw speed: 120 rpm

Extruder zone	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**Die**
Set temperature (°C)	80	90	115	125	130	130	130	120	115	90



TPS composites – Film preparation

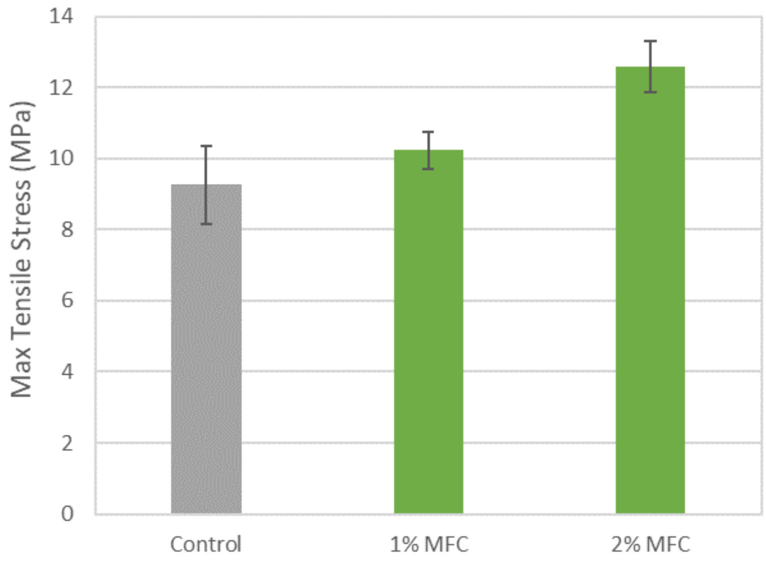
- Extruded samples compression-moulded into films at 145°C for 5 min at 50 kN pressure
- 250 μm thick films were produced for mechanical testing

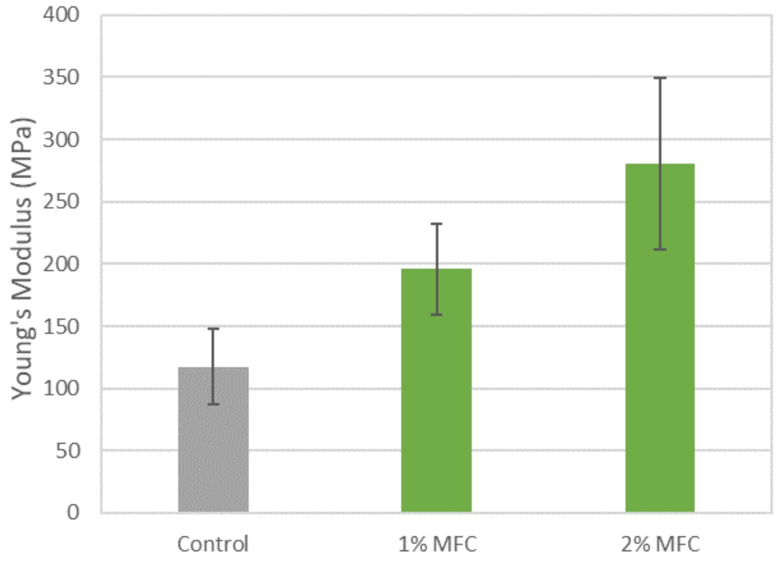


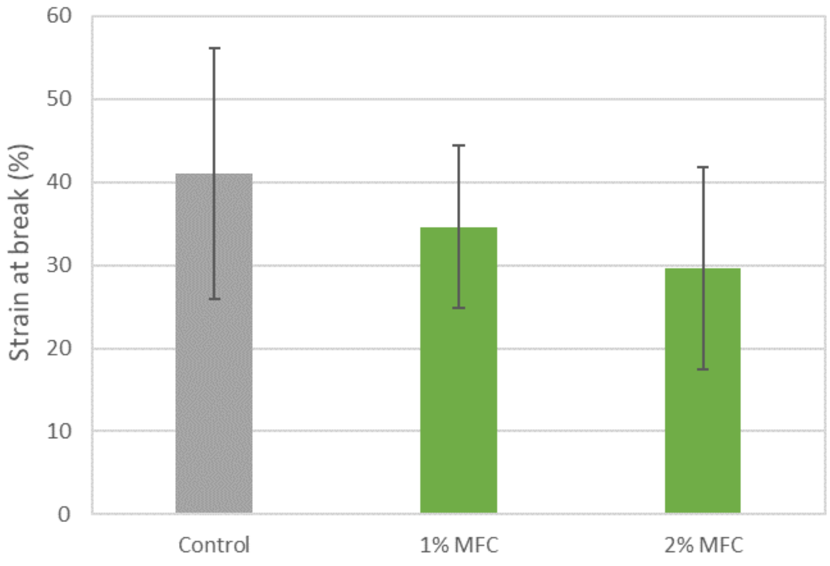
TPS composites – Tensile Testing

- Compression moulded films conditioned for 7 days at 23°C at a RH of 54% ($Mg(NO_3)_2$ sat. sol.)
- 60 mm x 10 mm rectangular strips cut out from each film
- Samples were conditioned at 54%RH for a further 3 days prior to testing
- Instron model 5543 universal testing machine with a 50 N load cell and pneumatic grips
- Crosshead speed of 20 mm.min^{-1} with a 20 mm-gauge length (following ASTM D882-18)
- A minimum of five replicates

Application 2 – Low Cost Compostable Films







36% improvement in UTS with 2% MFC

130% increase in Young's Modulus with 2% MFC

Strain at break was reduced by 25% with 2% MFC

Conclusions

- Forage sorghum is a very attractive feedstock for low cost ligno- MFC made by twin-screw extrusion
  - Mild pulping combined with high solids, continuous scalable processing
- **Application 1** - 2.5% w/w MFC addition demonstrated an improvement of 276% in toughness and 74% in tensile strength with respect to the client’s commercial control
- **Application 2** - 2% w/w MFC addition demonstrated an improvement of 36% in tensile strength and 130% Young’s Modulus with respect to the client’s commercial control
- Both commercial partners are progressing further with these applications

Acknowledgements

Funding

- Grains Research and Development Corporation (GRDC)
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Industry Partners

- GSA Innovation
- Cardia Bioplastics