BIO-PLASTIC INNOVATIONS

Characterisation Techniques
Mechanical, Chemical and Physical Testing
Durability, Safety & biodegradability

Co-funded by the European Union

This project has received funding from the European Union's Horizon Europe, grant number 101057765. This includes funds from the UK Research and Innovation (UKRI) under the UK government's Horizon Europe funding guarantee, grant number 10038028.

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GREENLOOP

Characterisation Techniques

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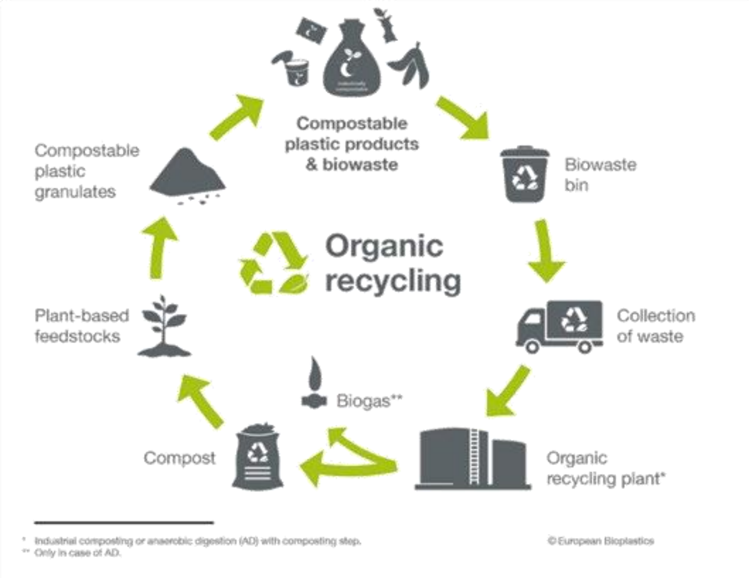
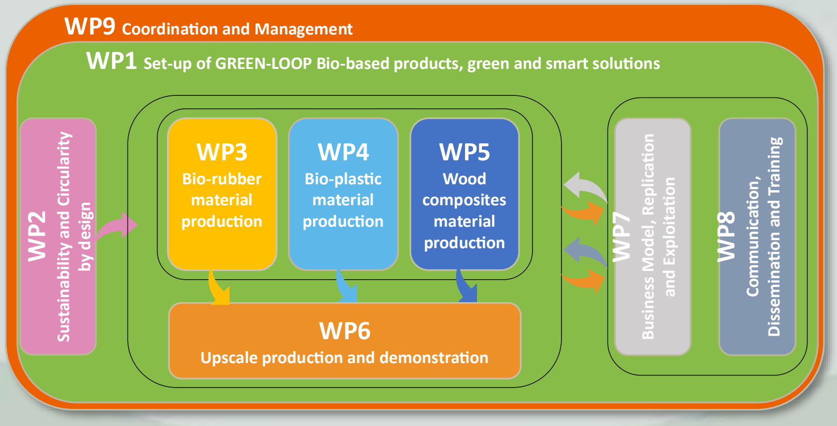
USING BIOPLASTIC TO REPLACE TRADICIONAL MATERIALS

- Replacement of traditional materials by bioplastic poses obvious advantages, but … Relevant properties must be demonstrated.
- Recycling of materials is more difficult than anticipated and composting might be an attractive way of reuse some materials
- WP 4 objective was to replace these traditional materials with bioplastics more sustainable and environment friendly.
- Baseline material property requirements were defined in T4.1 based on previous experience of the partners Mixcyling (material producer), Guala (closure manufacturer) and Terre di Zoe (end user).





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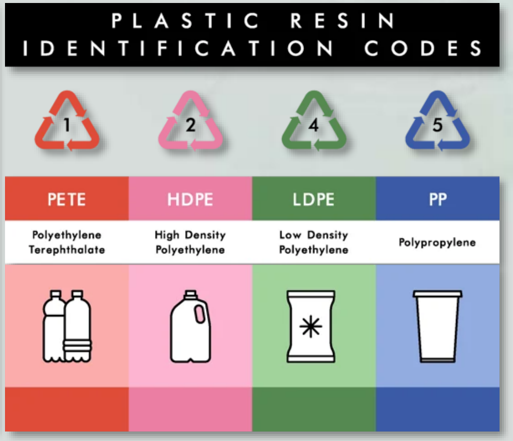
BIOPLASTIC MATERIAL REQUIREMENTS FOR BOTTLE CLOSURES

Current food container and closure materials include aluminum and polymers like PE, PET and PP. With high carbon footprint and/or eventual recycling problems.

However, we must take into consideration that:

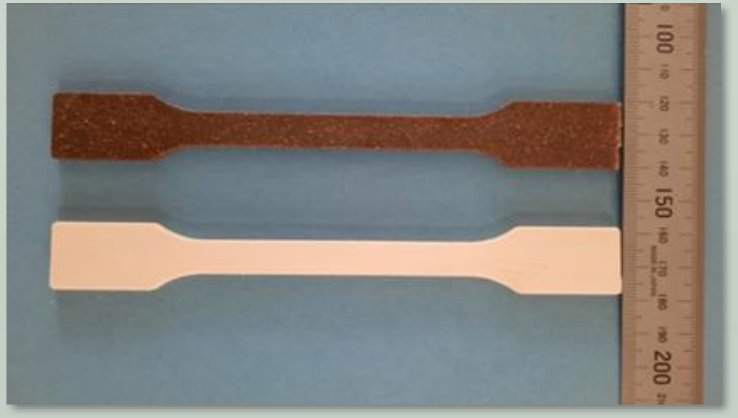
- Bioplastics properties are generally inferior to their petrochemical based counterparts.
- Composites reinforced with natural fibers, particularly with powder or short random fibers, also yield materials “weaker” than those reinforced with oriented glass or carbon fibers.
- Although the developed materials were not intended for a structural application, they must ensure some level of mechanical strength together with some ductility to give them enough toughness.
- New materials must respect very stringent rules concerning food contact.



BIOPLASTIC FORMULATIONS

Within WP3 many formulations were prepared to optimize the properties of the bioplastic.

A formulation with rice husk was also attempted within WP6



BATCH	REFERENCE	MATERIAL / MANUFACTURE PROCESS
1 WP3	BIOD FIBERFORM 1% NUOVO	100% fiberform
	BIOD FIBERFORM VECCHIO	100% fiberform
	T23016	56 % PLA + 24% PBAT + 20% calcium carbonate
	T23017	45,5 % PLA + 19,5% PBAT + 35% calcium carbonate
	T23018	40% PHB + 40%PLA + 20% Cork
	T23036	56% PLA + 24% PBAT + 20% biofill
	T23037	45,5% PLA + 19,5% PBAT + 35% biofill
	T23038	56% PLA + 24% PBAT + 20% micronized eggshell
	T23039	45,5% PLA + 19,5% PBAT + 35% micronized eggshell
	MATER-BI E102A2	100% Mater Bi
2 WP3	MP1800001	PHBH 100%
	T23088	PHBH 70% - PLA30%
	T23089	(PHBH+PLA) 80% - Cork 20%
	T23090	PHBH 80% - 20% Cork
3 WP6	PFT24078	80% (PHBH+PLA) + 20% Rice Husk
	PFT24162	80% (PPHBH+PLA+SEBS) + 20% Rice Husk
	PFT24164	LLDPE+LDPE+MASTER COAL

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BIOPLASTIC PROPERTIES REQUIREMENTS

- Based on previous experience and properties of competitor materials some baseline goals were stablished for the materials to be developed.
- These goals must be understood as development targets and not acceptance criteria.

PHYSICAL PROPERTIES

Natural Fibers content	15 – 45 %
Biodegradable thermoplastic carriers' content	55 – 85 %
Biobased additives content	< 5 %
Vicat softening temperature	93 °C
Specific weight	2.7 kg/m^3
Weight	1.85 g

MECHANICAL PROPERTIES

Compressive strength	> 80 MPa
Compressive modulus	> 2.5 GPa
Flexural strength	> 71 MPa
Flexural modulus	> 2.3 GPa
Tensile strength	> 49 MPa
Tensile modulus	> 2.3 GPa

FUNCTIONAL PROPERTIES

Must comply with European and international standards and directives such as:

ASTM D6866

EN 13432

DM 21/03/73

Council Directive 82/711/EEC

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CHARACTERISATION TECHNIQUES AND STRATGEY

- Different types of tests were chosen for properties characterization of the bioplastics.
- Several other techniques were also used to determine properties not initially considered.
- Preference for internationally accepted standards and procedures.

PROPERTY		TEST / Standard
Mechanical Properties	Tensile	ISO 527
	Compression	ASTM D695
	Flexural	EN ISO 14125
	Hardness Shore A	ISO 48
Physical Properties	Density	ASTM D792
	Microstructure	Microscopy
	Porosity / void content	Microscopy
	TMA	
	Thermal expansion	ISO 11359
	Moisture absorption	ASTM D5229
	VICAT Softening Temperature	ASTM D1525
	NMR/SEC/FTIR	
Functional Properties	Global migration	EN 1186
	Specific migration	EN 13130
	Organoleptic properties	???
	Metals and Amines	???
	NIAS - Nonintentional added substances	???
	Specific mechanical tests	?????

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Mechanical, Chemical and Physical Testing

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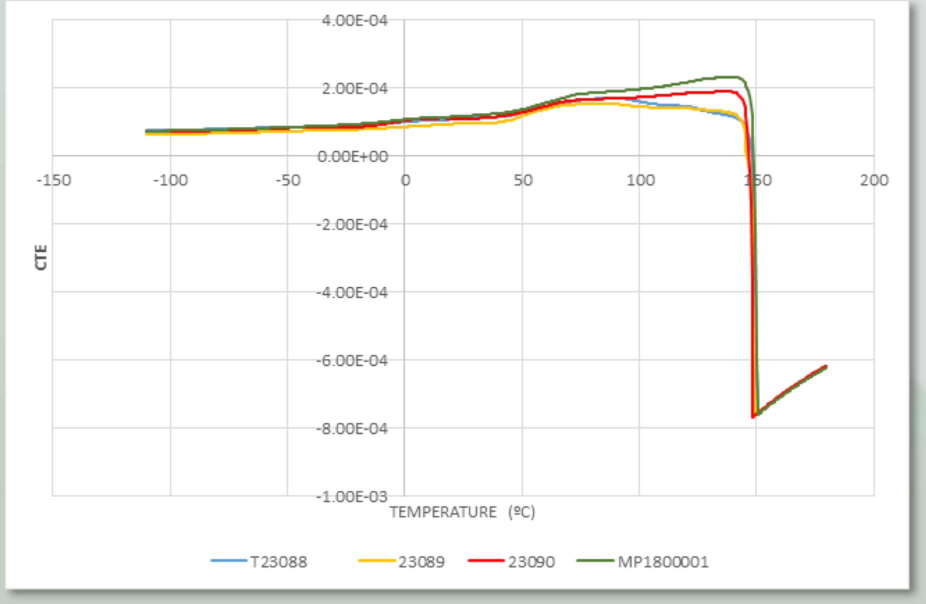
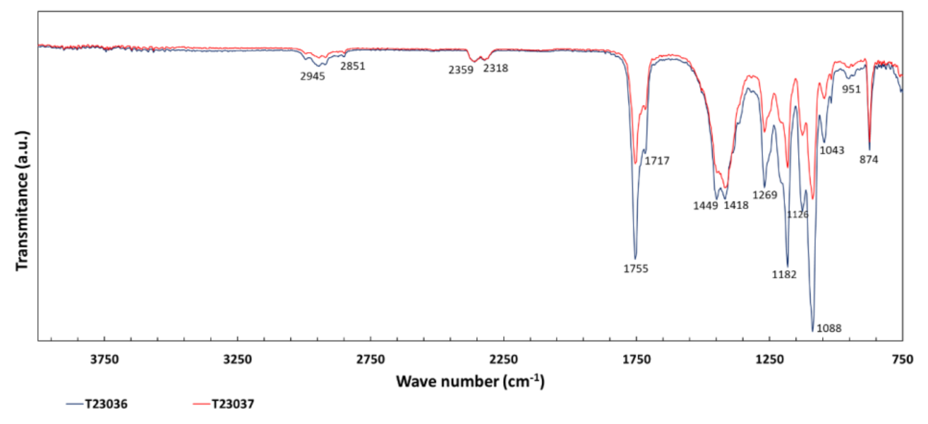
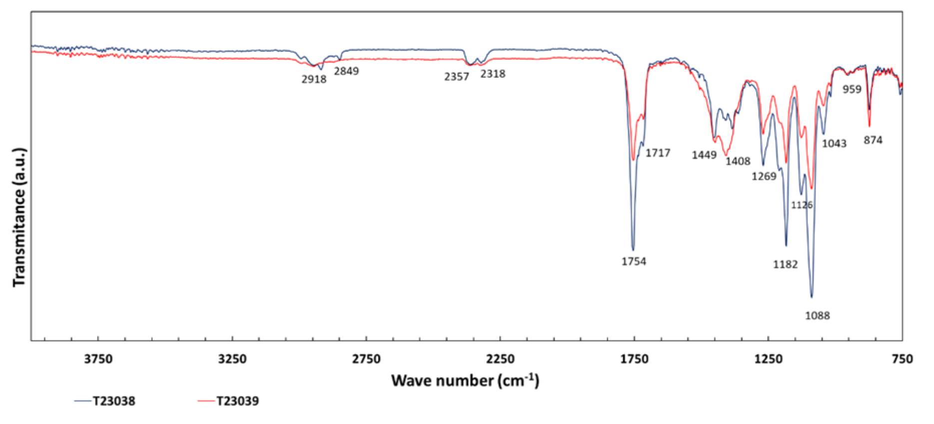
PHYSICAL/CHEMICAL PROPERTIES

- FTIR analysis was carried out in all formulations. Spectrums were as expected. No major interaction between material components detected.
- Thermal analysis performed by TMA/TDA identified CTE and structural stability up to 150ºC







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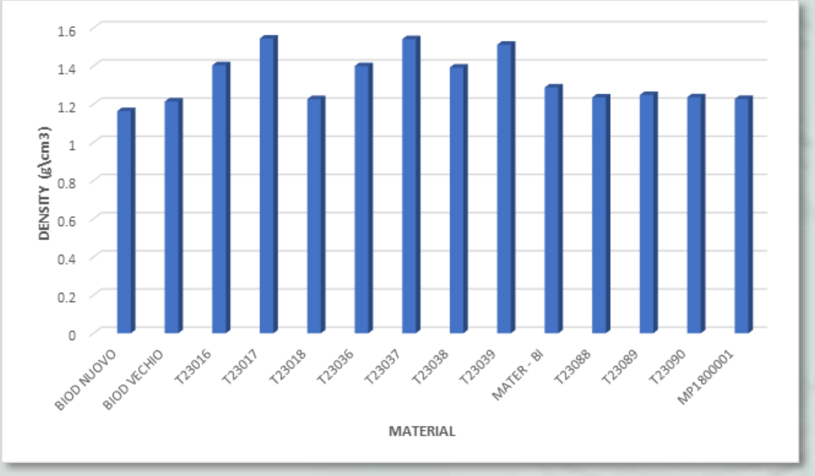
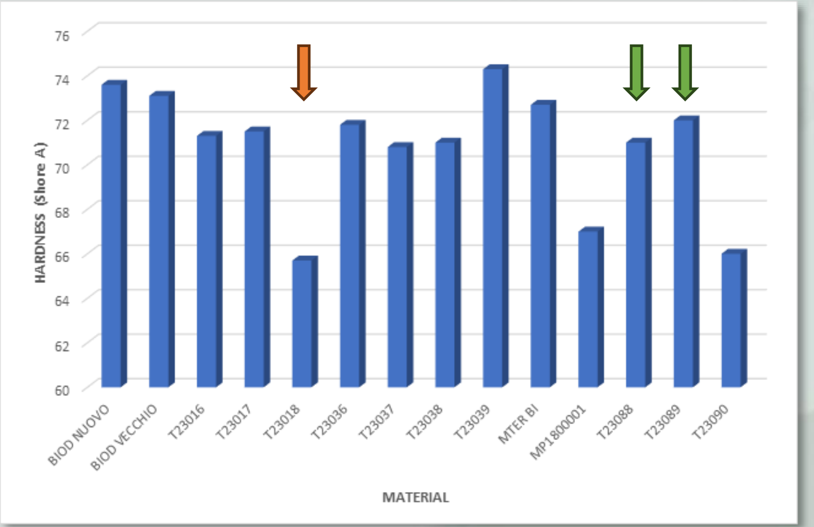
PHYSICAL/CHEMICAL PROPERTIES

- Density determined by gravimetric method in alcohol. Similar values for all materials ~1.2-1.5 g/cm^3.
- Considerable variations of hardness depending on the reinforcement material but specially on the polymer used.
- Significant increase of hardness on the cork reinforced material from the first batch associated with the introduction of PLA.
- Vicat test at with 10N and 50 °C/h. Softening temperature quite above the intended target.

DENSITY



HARDNESS



VICAT TEST (93 °C)

SPECIMEN	MATERIAL					
	23088	23089	23090	24078	24162	24164
1	122.6	123.8	123.5	124.8	112.4	83.1
2	119.6	125.2	123.6	126.7	111.2	84.8
3	119.5	123.7	125.3	126.4	115.3	84.6
4	124.2	125.5	123.8	126.7	116.1	85
5	124.2	-----	-----	-----	111.2	-----
6	119.3	-----	-----	-----	112.8	-----
Average	121.6	124.6	124.1	126.2	113.2	84.4
STD	2.38	0.93	0.84	0.91	2.08	0.87

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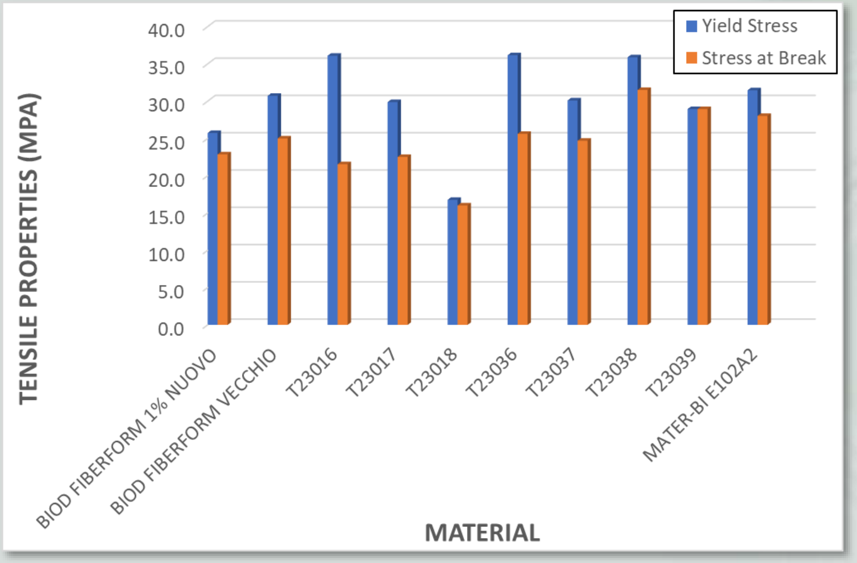
TENSILE PROPERTIES

- Reasonable results although strength on the lower side.
- Additions of biofill and eggshell of 20% with better results than 35%.
- Ductility on the lower side but good elastic modulus.
- Some concerns about the compatibility with food contact regulations of some fillers
- Most promising filler (cork) with disappointing results.

Target values

σ_M= 49 Mpa

E= 2300 MPa



REFERENCE	TENSILE STRESS AVERAGE (MPa)			TENSILE STRAIN AVERAGE (%)			MODULUS (MPa)
	σ_y	σ_M	σ_B	ε_y	ε_M	ε_B	E
BIOD NUOVO	25.7	25.7	22.9	1.6	1.6	6.9	2089
BIOD VECCHIO	30.7	30.7	25.0	1.3	1.3	16.9	2833
T23016	36.1	36.1	21.5	2.6	2.6	8.4	2480
T23017	29.9	29.9	22.5	2.3	2.3	4.2	2863
T23018	16.8	16.8	16.0	2.0	2.0	2.9	1606
T23036	36.2	36.2	25.6	2.6	2.6	5.7	2497
T23037	30.1	30.1	24.7	2.5	2.5	4.2	2684
T23038	35.9	35.9	31.5	2.5	2.5	3.5	2794
T23039	---	28.9	28.9	---	2.1	2.1	3126
MATER-BI E102A2	31.5	31.5	28.0	1.8	1.8	3.7	3437

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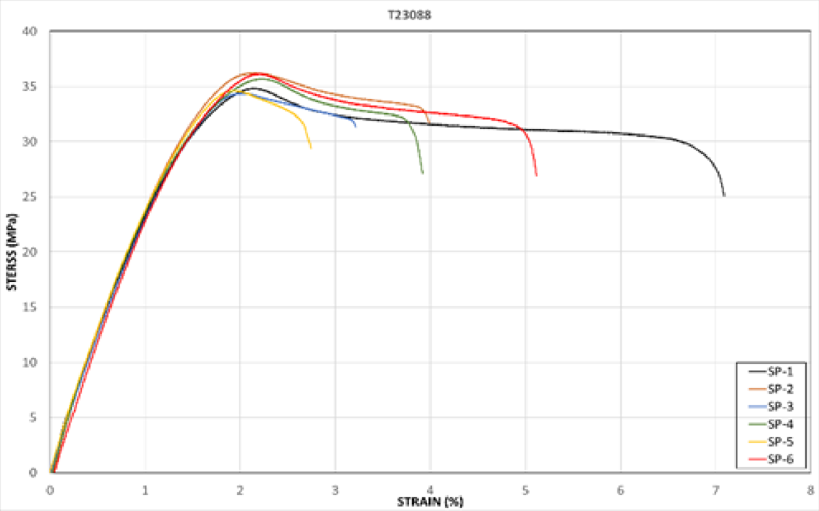
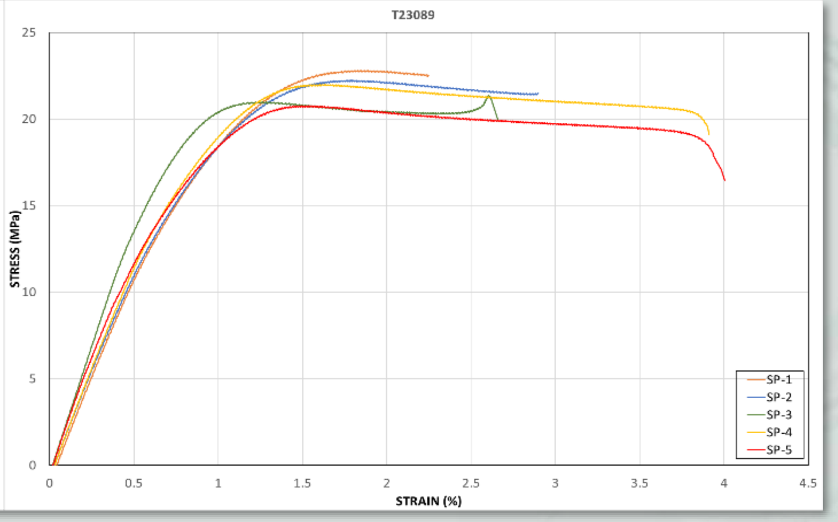
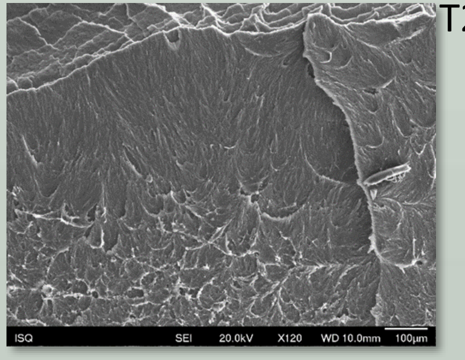
TENSILE PROPERTIES

- Major improvements in cork filled composite.
- Tensile properties match the best of the previous batch
- PHBH has a quasi-brittle behavior
- Blends of PHBH and PLA yielded the best results.

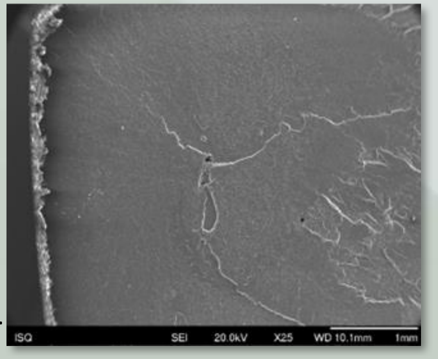






T23088

MP1800001



REFERENCE	TENSILE STRESS AVERAGE (MPa)			TENSILE STRAIN AVERAGE (%)			MODULUS (MPa)
	σ_y	σ_M	σ_B	E_y	ε_M	ε_B	E
MP1800001	-----	25.86	25.86	-----	2.17	2.17	2175
T23088	35.30	35.32	31.76	2.11	2.11	4.15	2782
T23089	21.80	21.76	20.75	1.60	1.60	3.05	2612
T23090	17.20	17.20	16.10	1.42	1.42	2.18	2117

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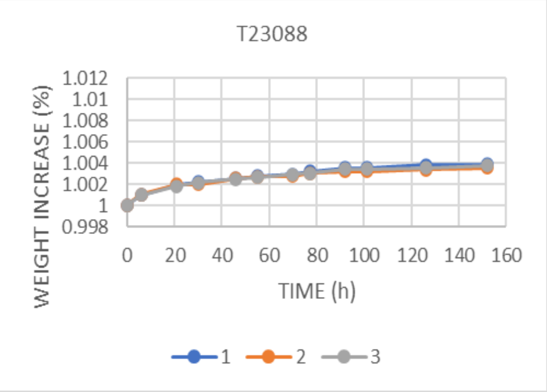
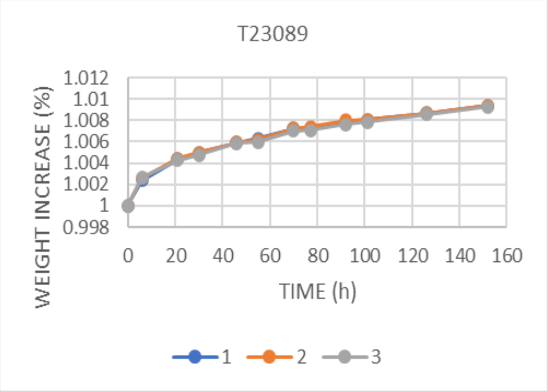
TENSILE PROPERTIES

- Tensile specimens were dried in oven at 40 °C and then immersed in distilled water for about 150 h.
- Moisture absorption more intense in cork filled material.
- Small effect upon the tensile strength.
- Higher effect on the ductility but still acceptable.





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FLEXURAL PROPERTIES

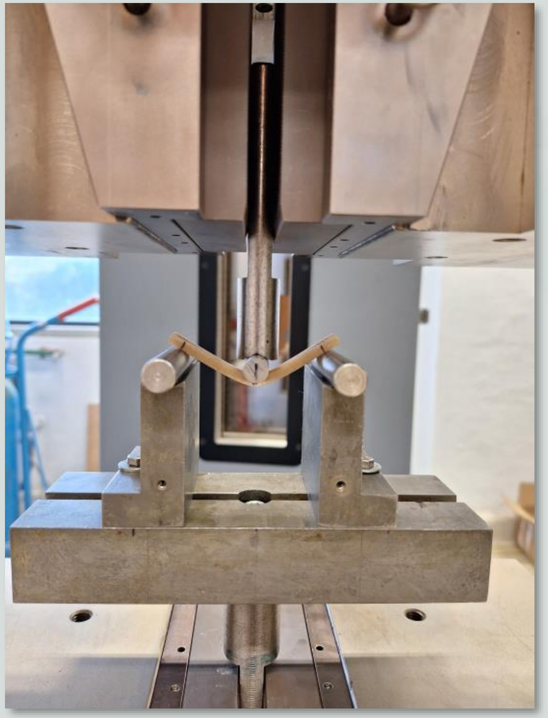
REFERENCE	FLEXURAL STRESS AVERAGE (MPa)			FLEXURAL STRAIN AVERAGE (%)			FLEXURAL MODULUS (MPa)
	σ_y	σ_M	σ_B	ε_y	ε_M	ε_B	E
MP1800001	-----	44.77	44.77	-----	3.4%	3.4%	1702
T23088	61.36	60.73	54.06	4.3%	4.1%	5.8%	2205
T23089	41.73	41.73	39.62	3.0%	3.0%	3.2%	1938
T23090	33.74	33.74	32.78	3.0%	3.0%	3.3%	1689

- Flexural strength with similar trends to the tensile behaviour
- Both strength and flexural modulus slightly lower than the initial target values
- Best performance with the PLA/PHBH blend.

Target values

σ_M= 71 Mpa

E= 2300 MPa



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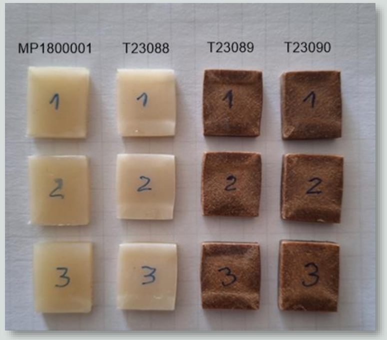
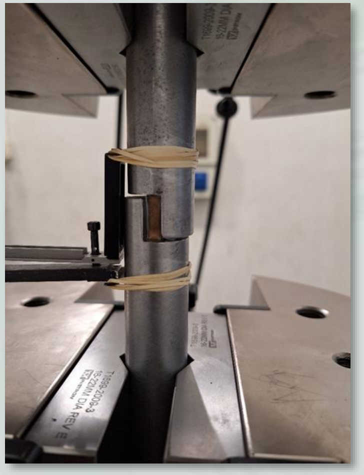
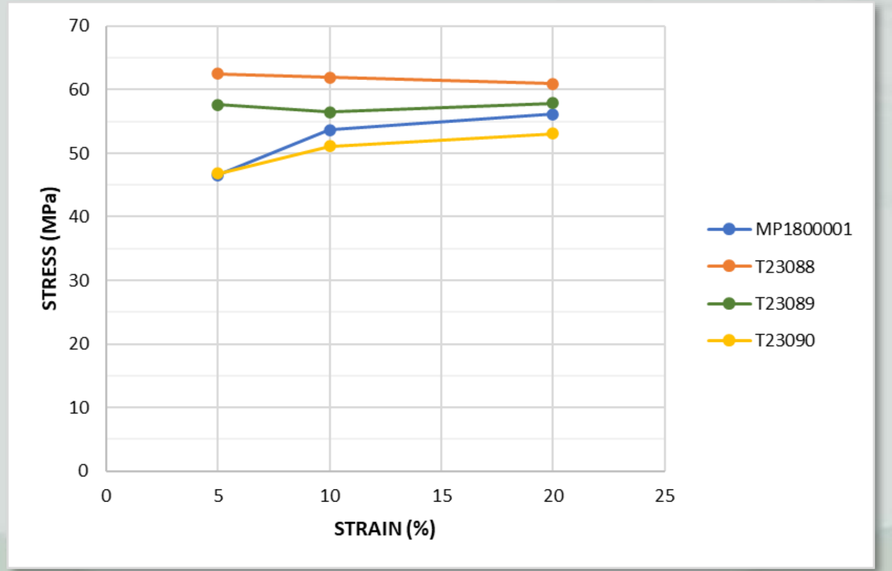
COMPRESSION PROPERTIES





- Standard test not possible due to the material dimensions
- A small compression tool was used to test specimens with 15 mm wide by 20 mm height.
- As with tensile tests the strength fell a bit short of the intended target value, but the modulus was very close to target.
- The PLA+PHBH blend produce the best results



Target values

σ_M= 49 Mpa

E= 2300 MPa

MATERIAL	MODULUS (MPa)	STRESS @ STRAIN		
		5.00	10.00	20.00
MP1800001	1907	46.58	53.66	56.18
T23088	2480	62.50	61.93	60.92
T23089	2539	57.65	56.50	57.90
T23090	1310	46.79	51.15	53.08

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Durability, Safety & Biodegradability

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SAFETY

- Safety issues are concern with the contact with food and the material capability to contain food stuffs without degradation or affect their quality
- There are strict rules at both nacional and international level for this purpose such as Directive 10/2011.
- Specific tests to evaluate the food contact suitability are:
  - Global migration
  - Specific migration
  - Organoleptic properties
  - Metals and Amines
  - NIAS - Nonintentional added substances

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GLOBAL MIGRATION TEST

- Oil and alcohol simulators
- Total immersion method
- Exposure for 10 days at 40ºC

Reference	Composition	SAMPLE	GLOBAL MIGRATION									
			OIL SIMULATOR					ALCOHOL SIMULATOR				
			VALUE	INCERTAINTY	AVERAGE	SPECIFICATION	RESULT	VALUE	INCERTAINTY	AVERAGE	SPECIFICATION	RESULT
T23090	PHBH + 20%Cork	1	56.5	3	60	10	NOK	18	0.6	16.5	10	NOK
		2	53.5					16				
		3	69.5					15.5				
T23088	PHB+PLA	1	44.5	2.3	45.5	10	NOK	6	0.2	5.5	10	OK
		2	46.5					4.5				
		3	45					5.5				
T23089	PHBH+PLA+CORK	1	18	2.9	58.5	10	NOK	27.5	1	27.5	10	NOK
		2	85					26.5				
		3	71.5					28				
PFT24078	80% (PHBH+PLA) + 20% rice husk	1	NA	-----	-----	10	-----	2.5	0.2	4	10	OK
		2	NA					3.5				
		3	NA					5.5				
PFT24162	80% (PPHBH+PLA+SEBS) + 20% rice husk	1	61.5	1.8	37.5	10	NOK	<0,2	-----	<0,2	10	OK
		2	17					<0,2				
		3	33					<0,2				
PFT24164	LLDPE+LDPE+MASTER COAL	1	22	1.3	25.5	10	NOK	<0,2	-----	<0,2	10	OK
		2	23					<0,2				
		3	31					<0,2				

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DURABILITY & BIODEGRADABILITY

- Bio polymers are generally less durable than oil derived ones. More easily degraded in the environment at the end of their life cycle.
- Does not mean that when disposed off in the environment they will be degraded in a fast way.
- Suitable for degradation by composting but often at industrial scale. Even then the time scale by be months.
- Several international standards regulate the testing and classification of bioplastics suitable for composting
  - EN 13432
  - ASTM D6400
  - ISO 17088
- Factors affecting the composting process include:
  - Composition of composting load.
  - Time
  - Temperature
  - Aeration
  - Worms & Micro-organisms

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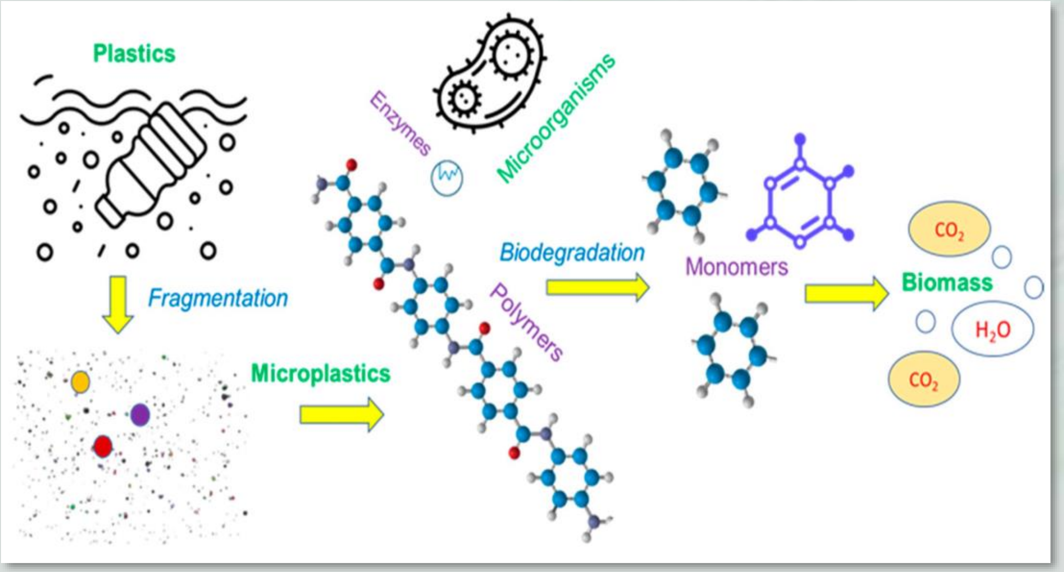
COMPOSTING METHODS

The time it takes to compost can vary widely depending on the method and conditions. Timesacle can reach a few month even in industrial conditions.

- **Cold Composting**
  - Timeframe: 6 months to 2 years.
  - This method is less labor-intensive but slower. It involves adding organic materials to a pile and letting nature take its course with minimal turning.
- **Vermicomposting**
  - Timeframe: 2 to 3 months.
  - Using worms to break down organic matter can be faster than traditional composting methods, especially for kitchen scraps.



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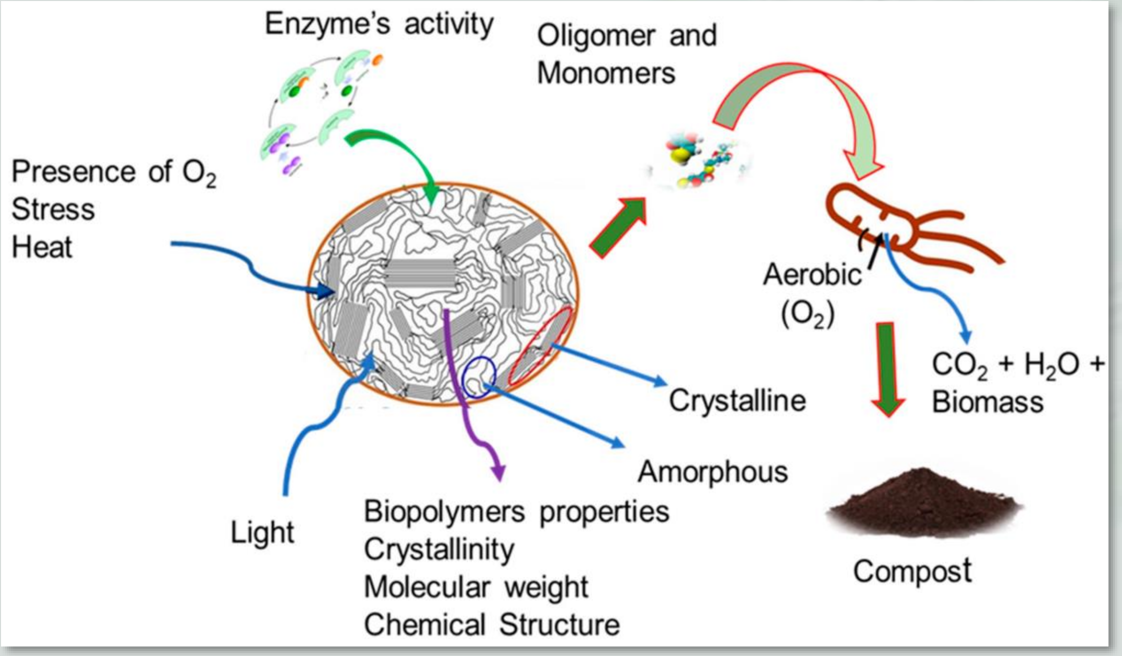
COMPOSTING METHODS

- **Hot Composting**
  - Timeframe: As short as a few weeks to a few months.
  - This method involves maintaining high temperatures (around 50-60°C) by regularly turning the compost pile and ensuring a good balance of green (nitrogen-rich) and brown (carbon-rich) materials.
- **Industrial Composting**
  - Timeframe: Few weeks to few months.
  - Industrial facilities use controlled environments to optimize the composting process, often speeding it up compared to home composting.



CURRENT/FUTURE ACTIVITY

- Further food contact tests under consideration
- Permeability tests being considered/planned
- Composting tests to start shortly

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