agrimax

Agri & food waste valorisation co-ops based on flexible multi-feedstocks biorefinery processing technologies for new high added value applications

Patrizia Cinelli, Norma Mallegni

INSTM, Inter University Consortium of Materials Science and Technology Unit, University of Pisa, Department of Civil and Industrial Engineering

23rd September 2021

Webinar training session on agricultural by-products valorisation through bio-refineries

(FCAC - Virtual Event)

Horizon 2020 European Union Funding for Research & Innovation

Bio-based Industries Consortium

This project has received funding from the Bio Based Industries Joint Undertaking under the European Union's Horizon 2020 research and innovation programme under grant agreement No. 720719.

INSTM unit of Pisa University, Department of Civil and Industrial Engineering

Who we are and where we are

Our Mission

Understanding and controlling, by careful processing, structure properties relations in polymers and composites with particular interests to mechanical properties, interphase adhesion, plasticity, creep, fatigue and fracture mechanics.

Our Vision

- Production of bio-based polymers and composites to solve the problems related to materials derived from oil.
- Improvement of mechanical and thermal properties of techno-polymers composites and ceramic composites.
- Development of new materials in the biomedical sector with particular interest in tissue engineering.

- University of Pisa: Public University (established in 1343), 56.000 students, 2.311 researcher and teaching staff.
- Department of civil and industrial engineer includes the departments of: Aerospace, Mechanical, Nuclear Engineering and also Industrial Chemistry and Materials Science.

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Funded projects

wheylayer

Lequval

BioBoard

OliPHA

PERFORMANCE

n-chitopack

dibbio pack

ecofunco

POLYBIOSKIN

FORBIOPLAST

biontop

AGROCYCLE for a circular economy

EVOLUTION

ROBO-IMPLANT

PHA

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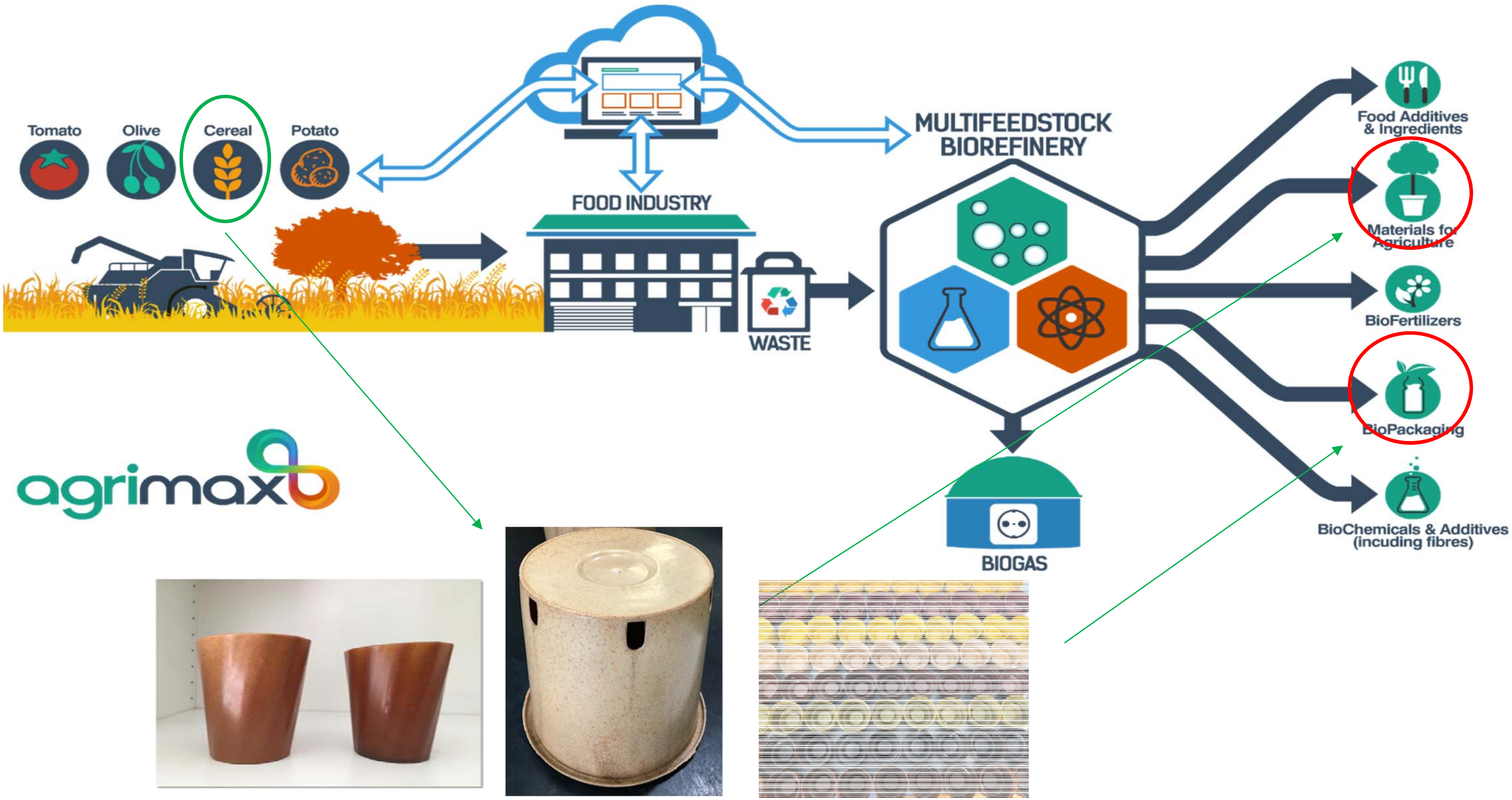

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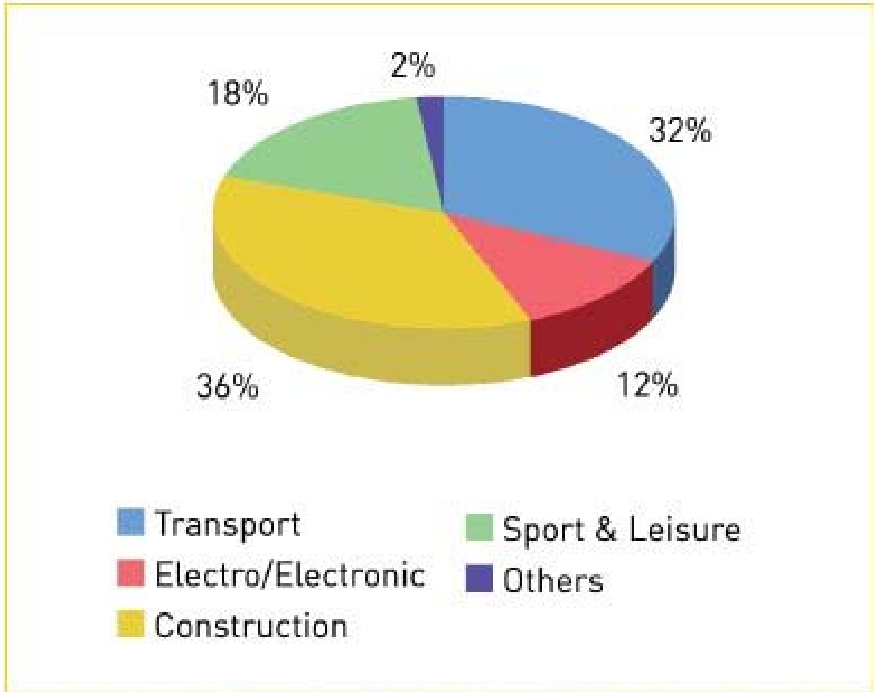
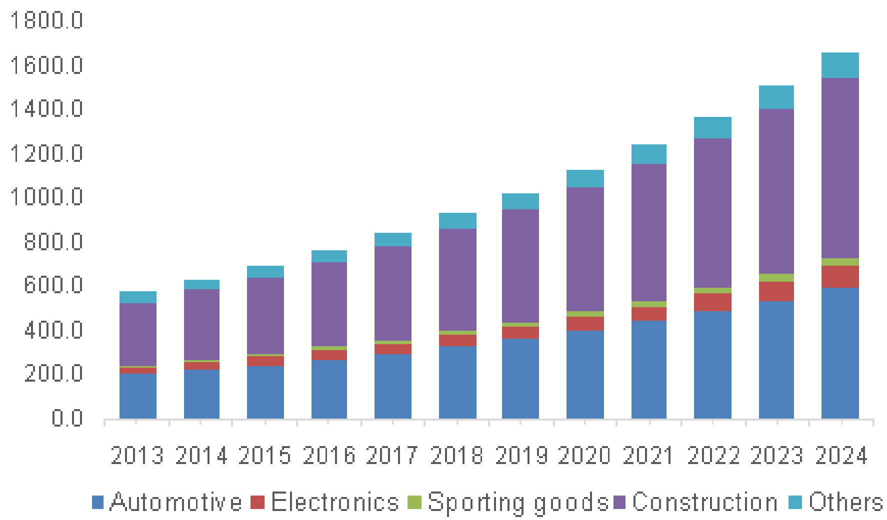


Fig. 2: GFP production in Europe for individual application industries (year: 2009)

Europe natural fibre composites market revenue, by application, 2013 - 2024 (USD Million)



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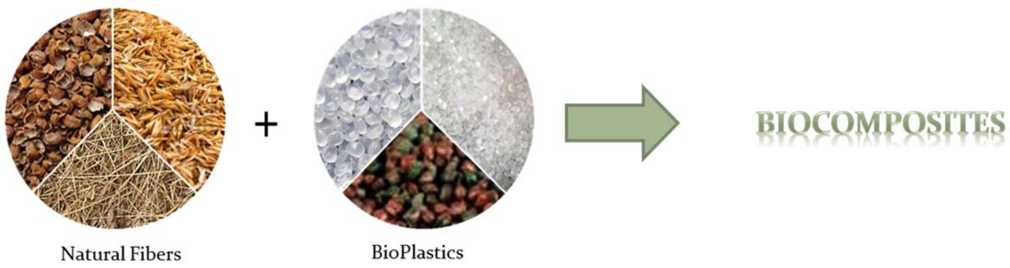
Biocomposites

- Next generation of materials: Natural/Biofibre composites (Bio-Composites)
- Viable alternative in many applications, agriculture, packaging but even automotive field
- Special attention required by natural composites in order to be competitive with synthetic composites (in particular biofibre–matrix interface and new extrusion processing).
- The advantage of fibre-reinforced plastics results from their structure. The controlled structure in which the fibres are lying in the polymer matrix increases the stiffness of the composite.
- In particular this is important in automotive or building product.
- The presence of natural fibres promotes degradability and thus meeting compost ability



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Advantages of Composites Containing Natural Vegetable Fibres

- They are environmentally friendly materials at the stage of production, processing and waste.

- Environmentally friendly production of natural vegetable fibres - annual renewability and lower energy inputs in production per unit.

- They display acoustic insulation and absorb vibrations and large quantities of energy when subjected to destruction.

- Lower density of polymer composites reinforced with natural fibres than those reinforced with glass fibre.

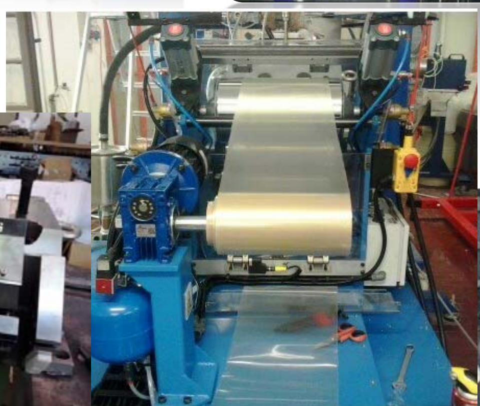
- The price of polymer composites reinforced with natural fibres is from two to three times lower than that of polymers reinforced with glass fibre.

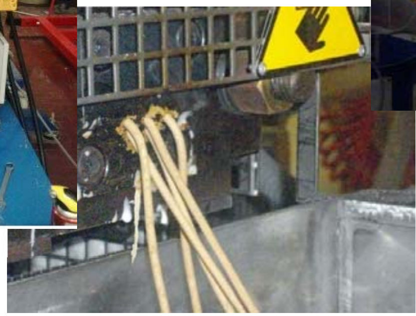
- Natural vegetable fibres can be applied to the reinforcement of the natural polymers such as starch, lignin, hemicellulose and the material obtained in this way is 100% biobased and biodegradable.

- Reaction to fire of composites based on lignocellulosic fibres is much more beneficial comparing to polymers – significant reduction of heat release rate.

POLYMERS PROCESSING

Haake mini extruder e minijet





COMAC Estrusore

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MATERIALS CHARACTERIZATION

- *Differential Scanning Calorimetry*
- *Dynamic Mechanical Analyzer*
- *Mechanical Tests: MTS; Instron, Impact Charpy*
- *SEM, FTIR, ... etc*

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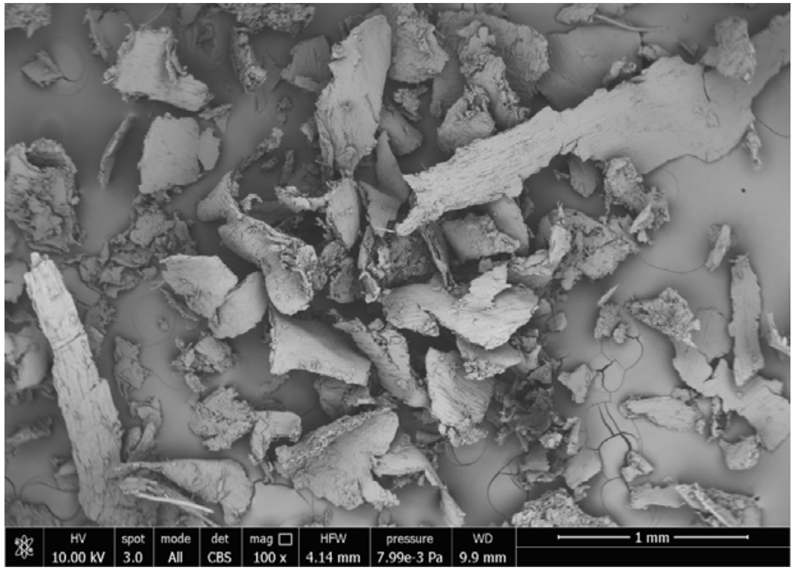
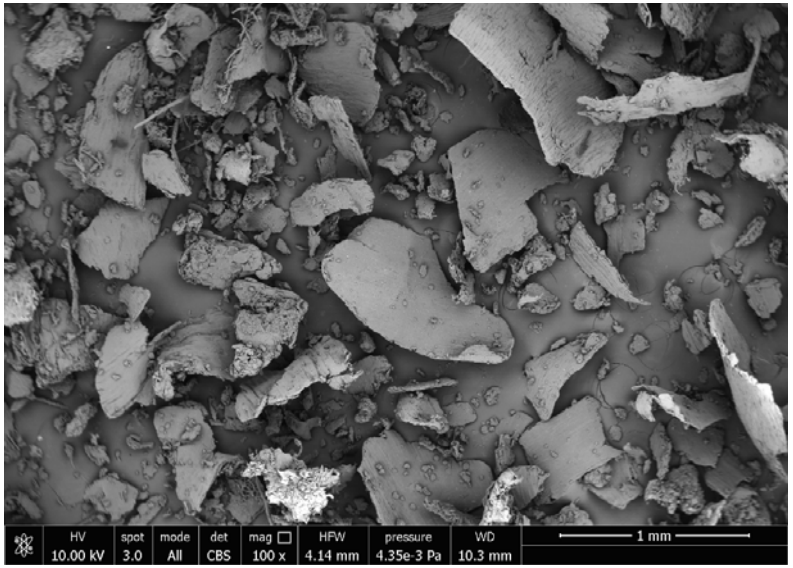
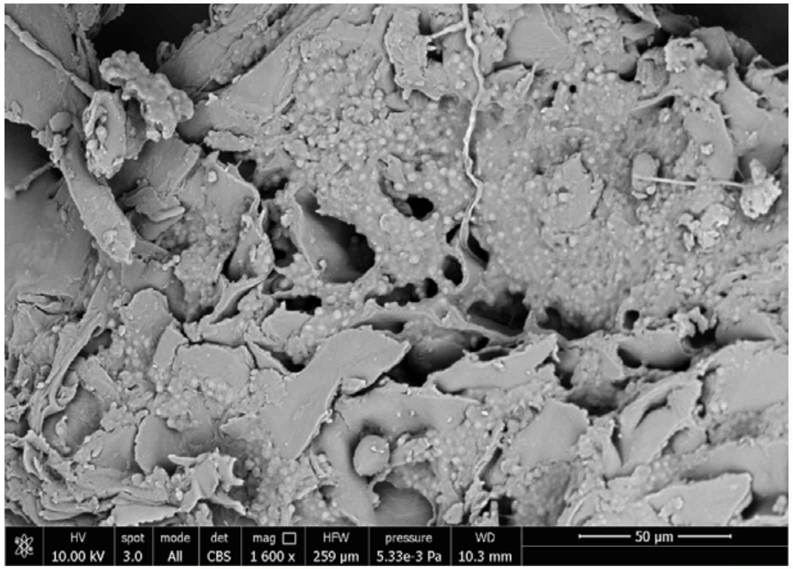
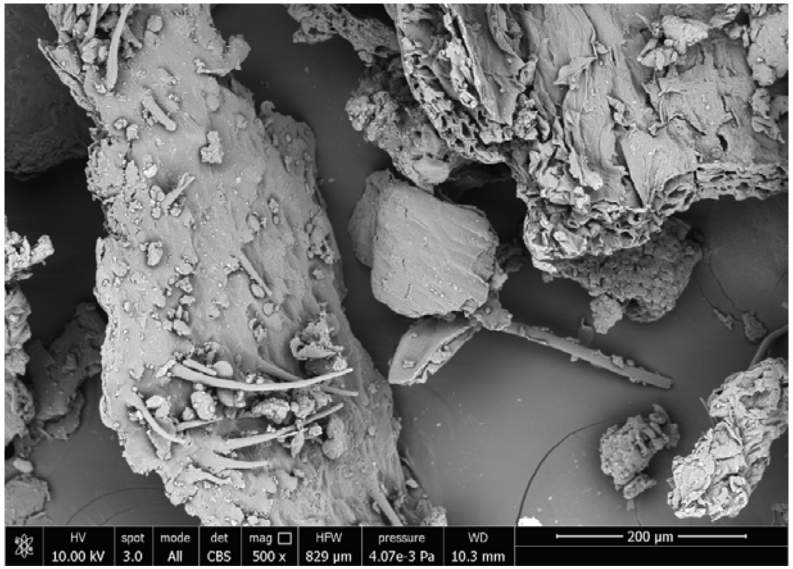
Bran





Micronised Bran





No fibres shape but fillers size/ shape !!

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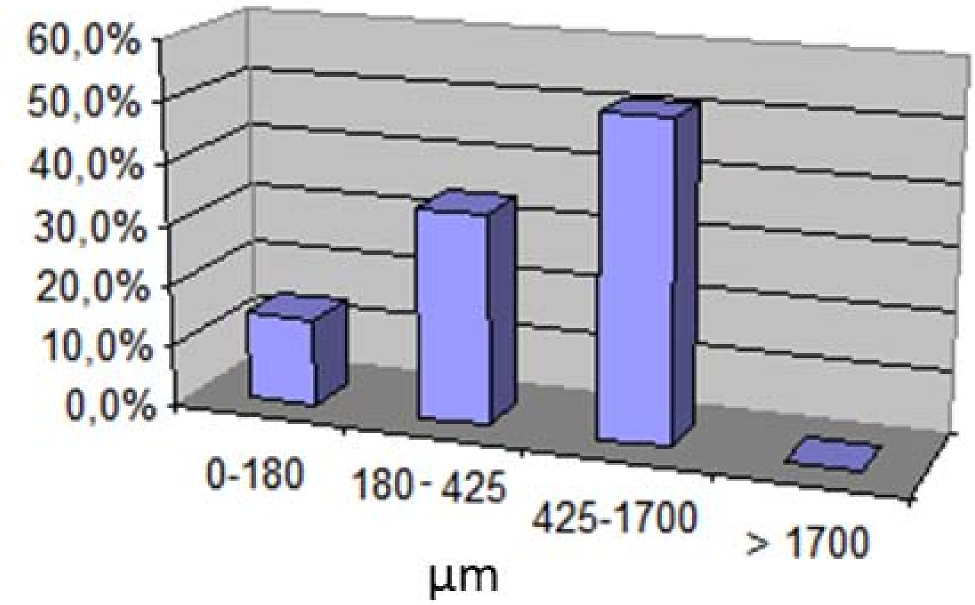
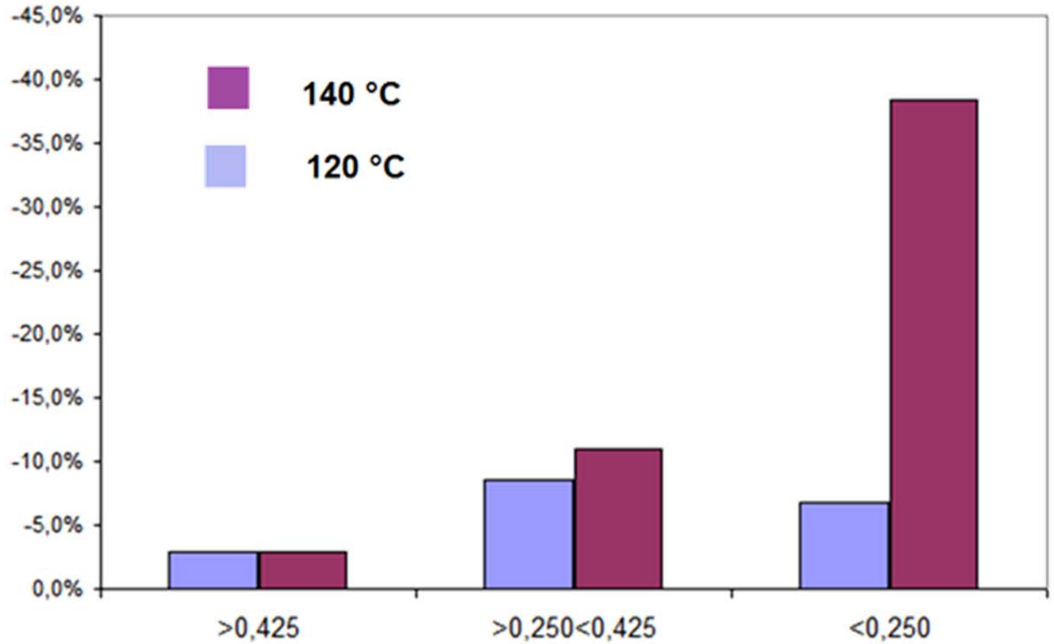
AGRIMAX bran fiber size distribution





Thermally pretreating the fiber at increasing temperatures (under the T of pyrolysis start estimated at 150 °C) we avoid the fraction below 300 microns, gas is present in the compounds with consequent formation of shrinkage defects in the injection molded pieces

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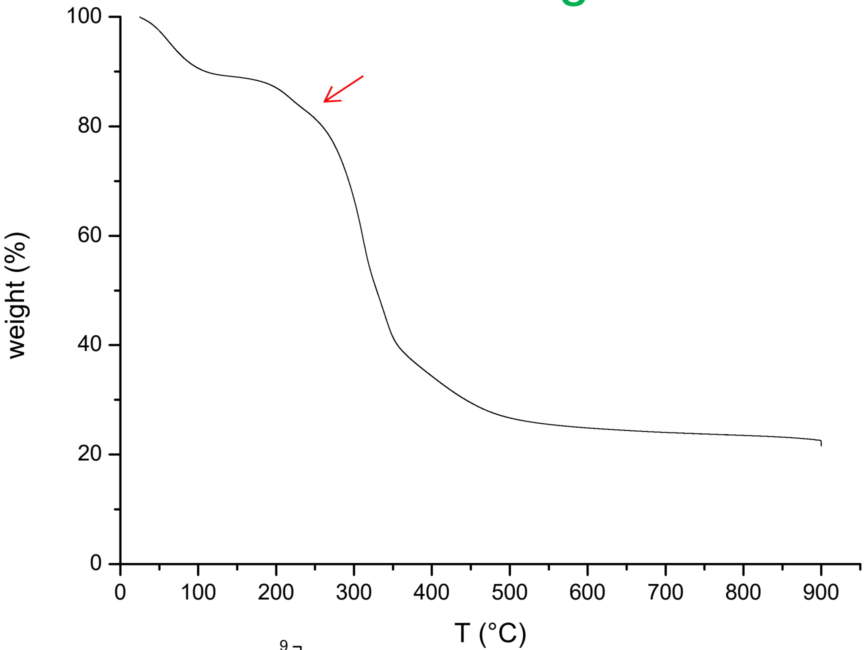
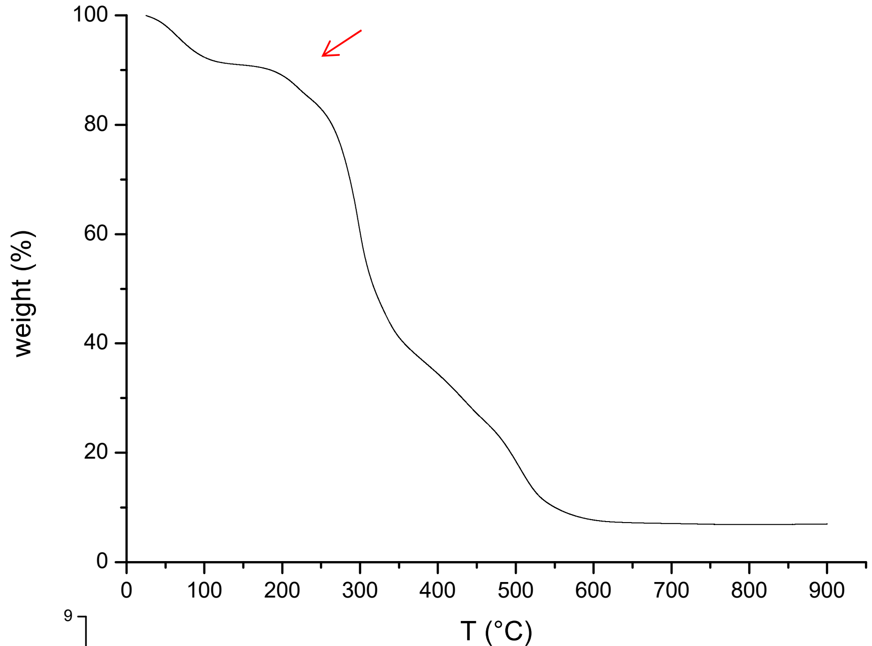
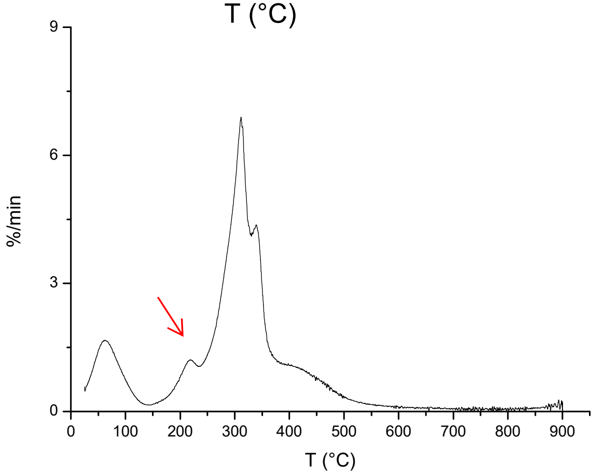
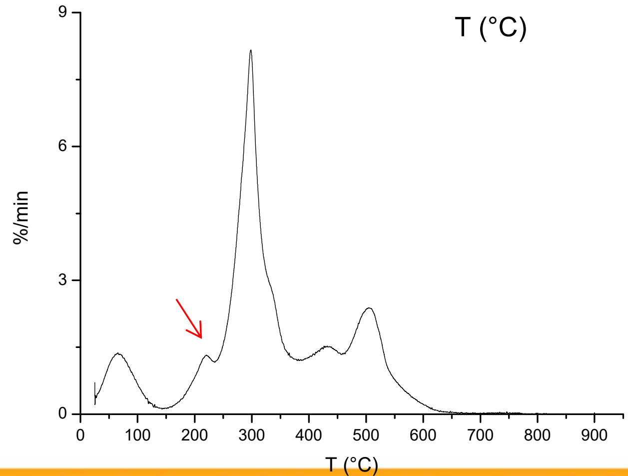
T degradation compatible with processing









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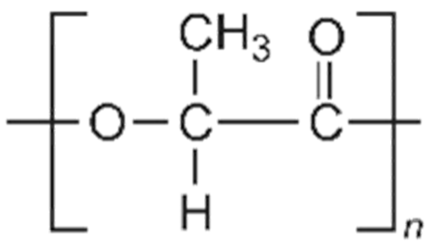
POLYLACTIC ACID (PLA)

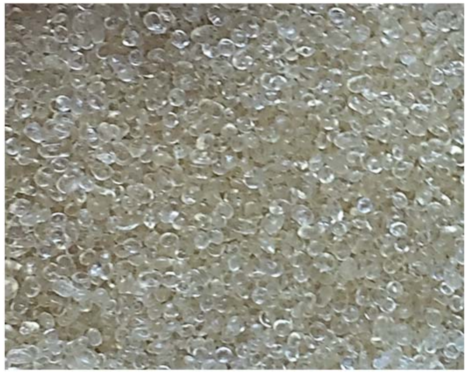
- Poly(lactic acid) (PLA) is a biodegradable and biobased thermoplastic aliphatic polyester.

- It derives from renewable resources such as corn starch (in the United States and Canada), tapioca roots, chips (mostly in Asia), or sugarcane (in the rest of the world).

- In 2010, PLA had the second highest consumption volume of any bioplastic of the world[2].

Monomeric Unit





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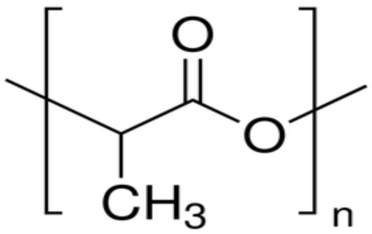
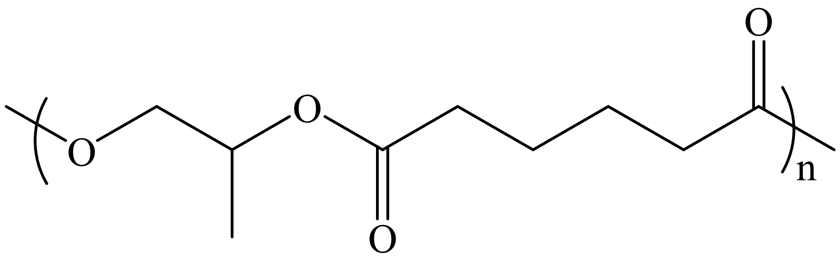


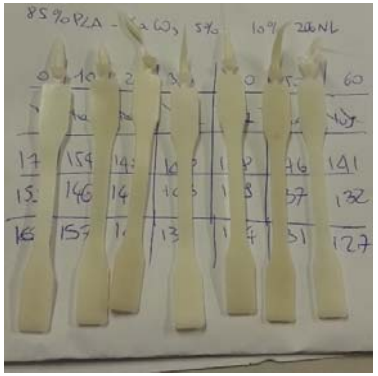
Polylactic acid



GLYPLAST®206 3NL (polyester of adipic acid with 1,3-butanediol, 1,2-propanediol and 2-ethyl-1-hexanol) a saturated polyester with the density of 1,07 (g/cm3 at 25 ºC) kindly provided by Condensia Quimica S.A (Spain).

$CaCO_3$

PLA85 -$CaCO_3$ 5 – Plasticizer 10 + 10-20% Bran



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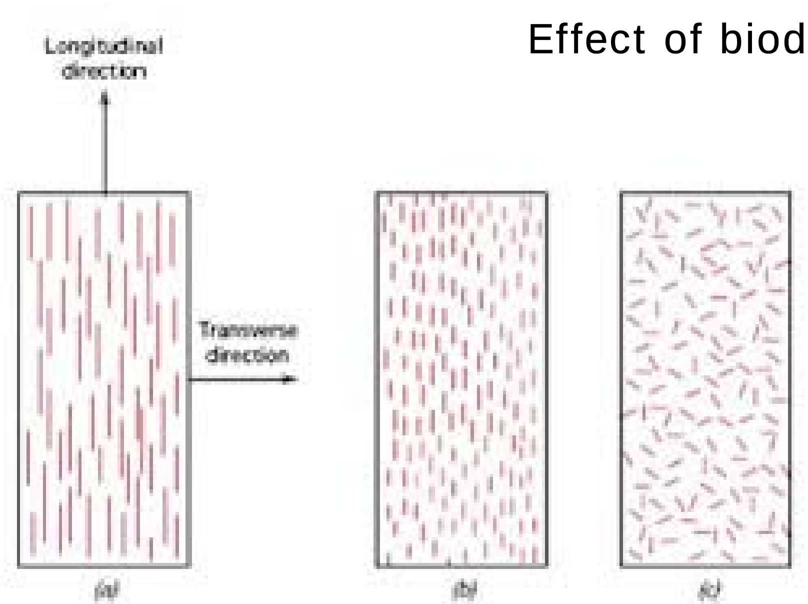
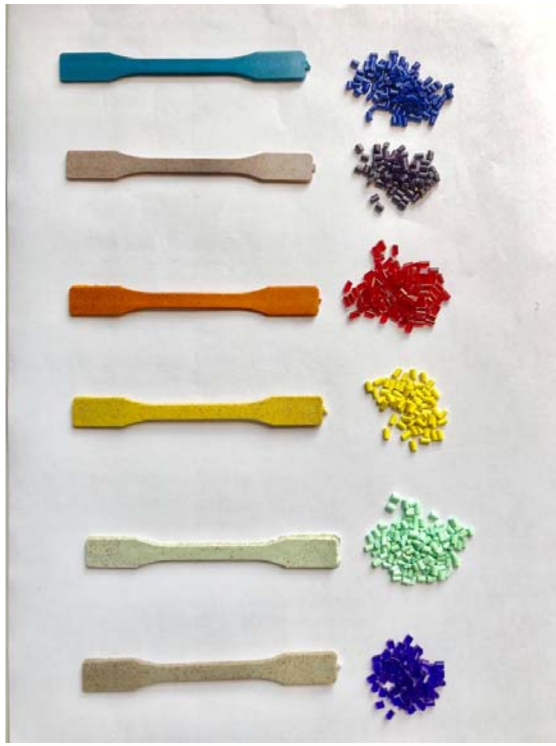
	E (Gpa)	Elongation (%)	Ultimate Tensile Strength (Mpa)
PLA matrix	4.3	1.7	54.5
PLA- 10% Bran	3.3	2.8	39.1
PLA -20% Bran	3.6	2.9	33.6

Fillers, no reinforcement, benefit of lower cost, higher degradability

Effect of biodegradable colourants





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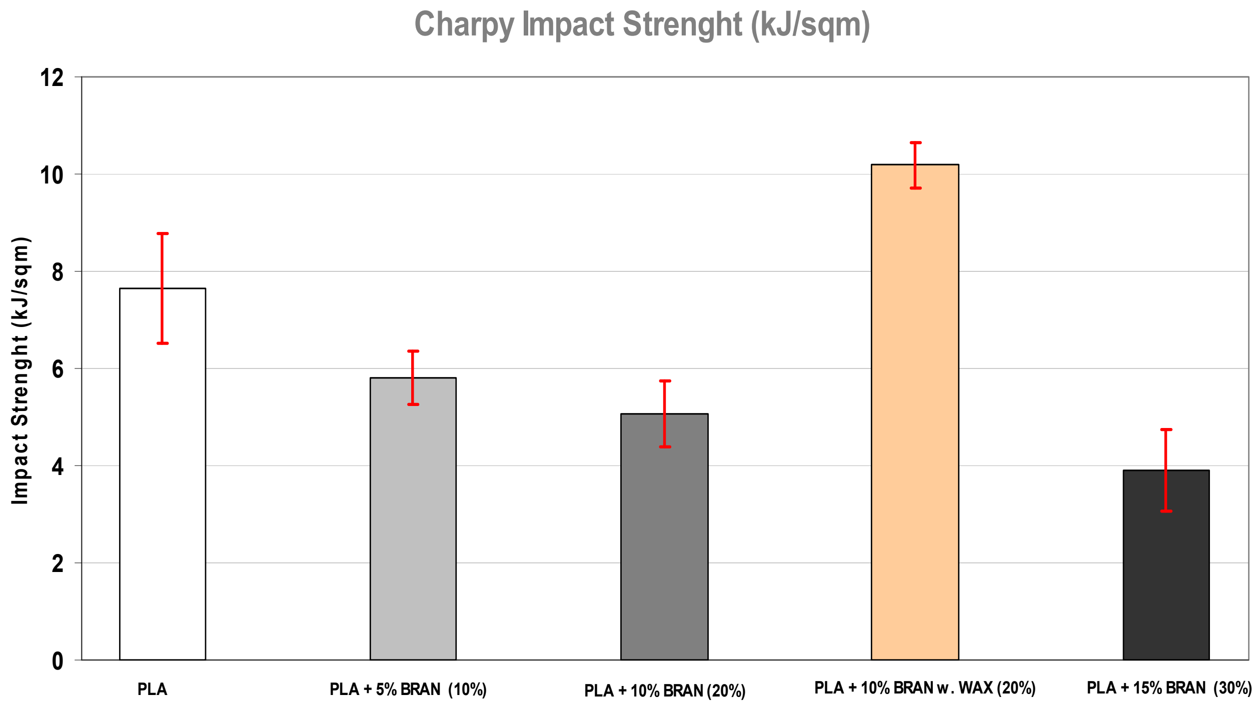
Bran Fibres treated with Natural waxes

- To optimize the adhesion and compatibility between bran fibers and biopolymeric matrices, a wax coating has been realized, comparing the results with or without the treatment.
- The base matrix is composed by a large amount of PLA, to achieve stiffness, PBAT, to improve impact resistance, and calcium carbonate as inorganic filler.
- In the graph it’s possible to see how the wax coating improves the impact resistence of the final compound.

The bran fibres were treated with a 1/6 weight ratio of bees wax to fibres, resulting in a final amount of wax (dry matter) to fibres of 1% by weight



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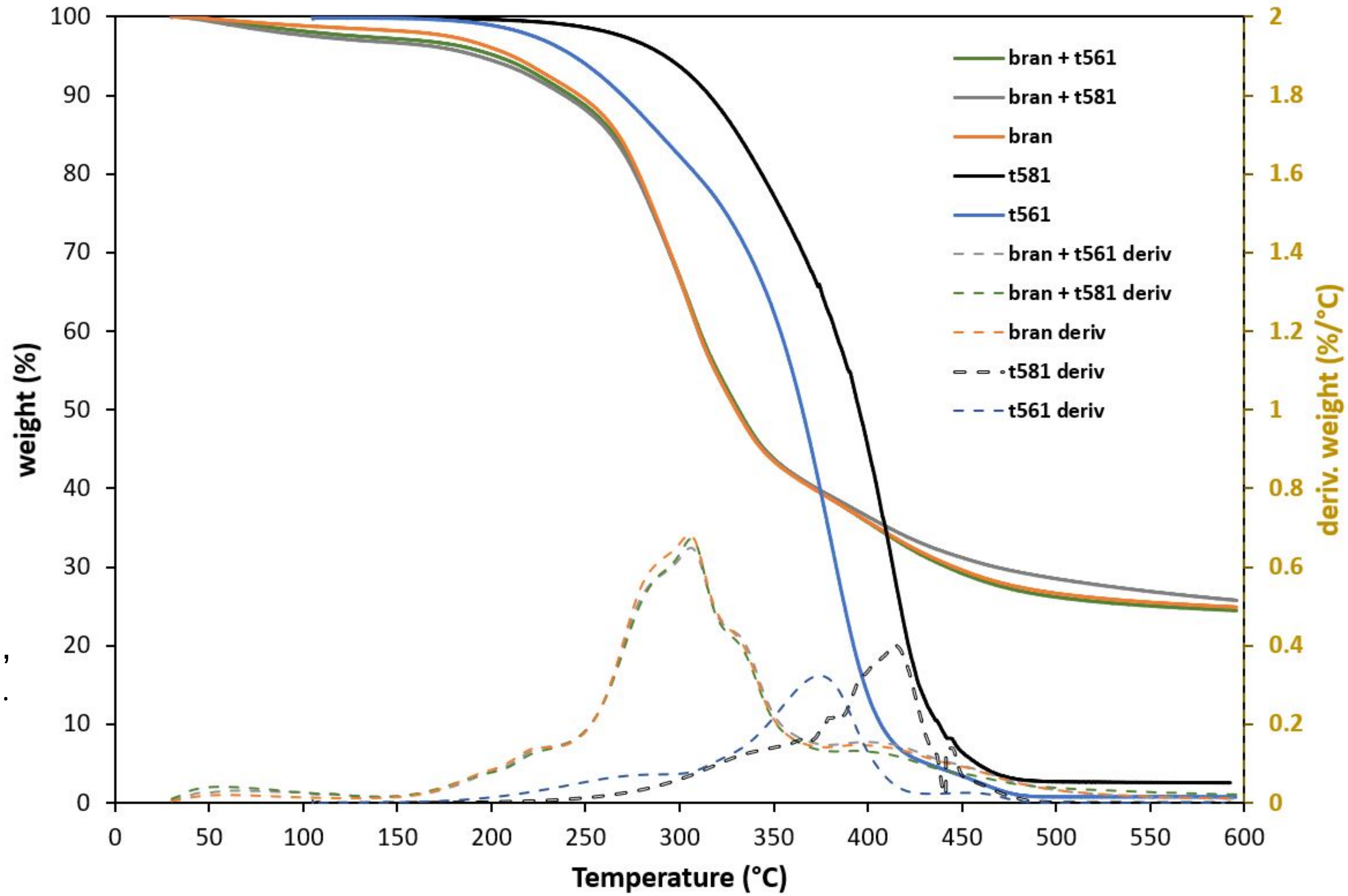
Bran Fibres treated with Natural waxes

Verified by Thermal Gravimetric Analysis the Thermal stability of the waxes and of the fibres after being treated with the waxes.
The fibres after wax treatment don't change significantly thermal stability.



ACQUACER 581 (Carnauba renewable),
ACQUACER 561 (bees wax, renewable).

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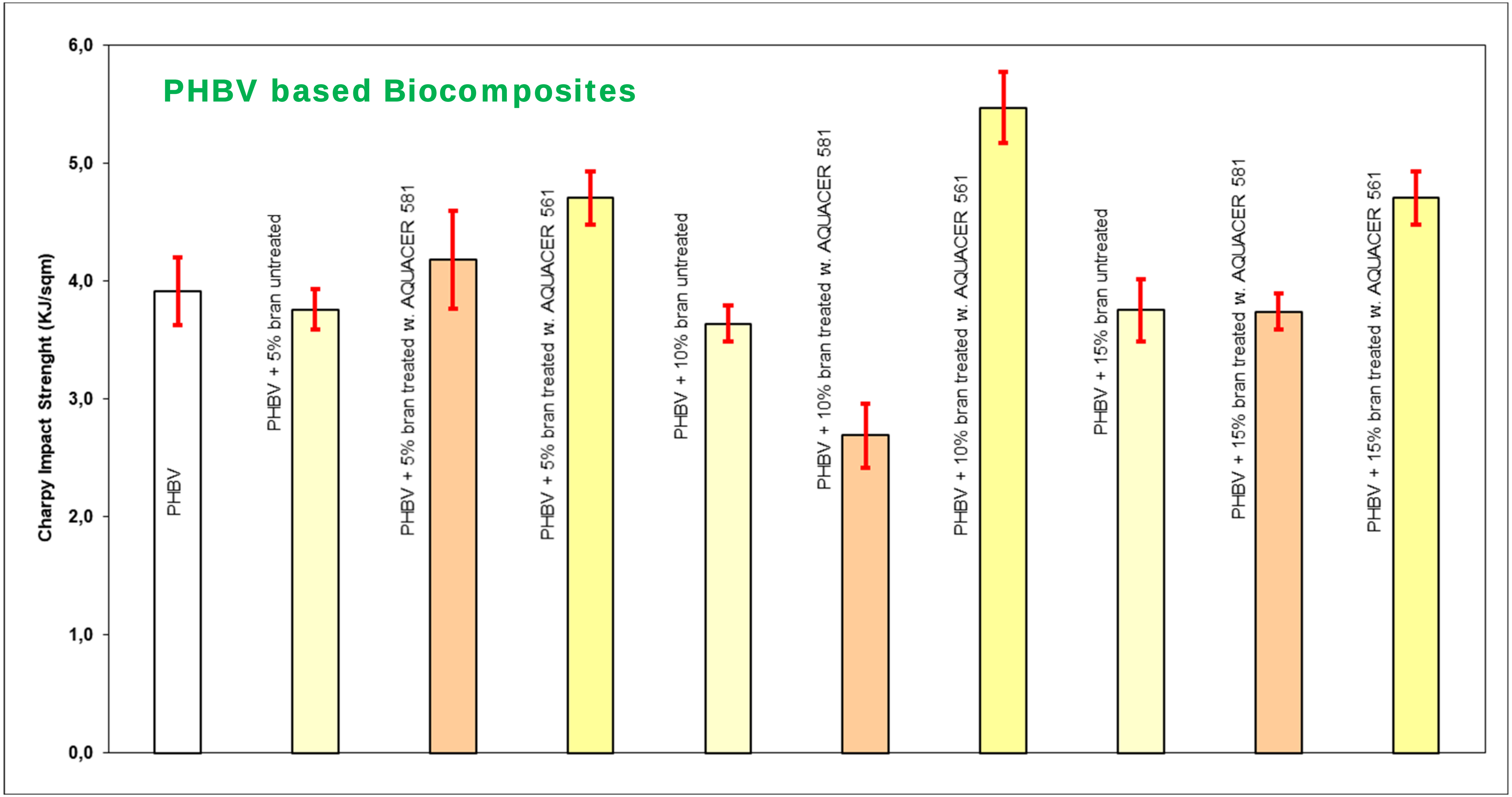
ACQUACER 581 (Carnauba renewable), ACQUACER 561 (bees wax, renewable).



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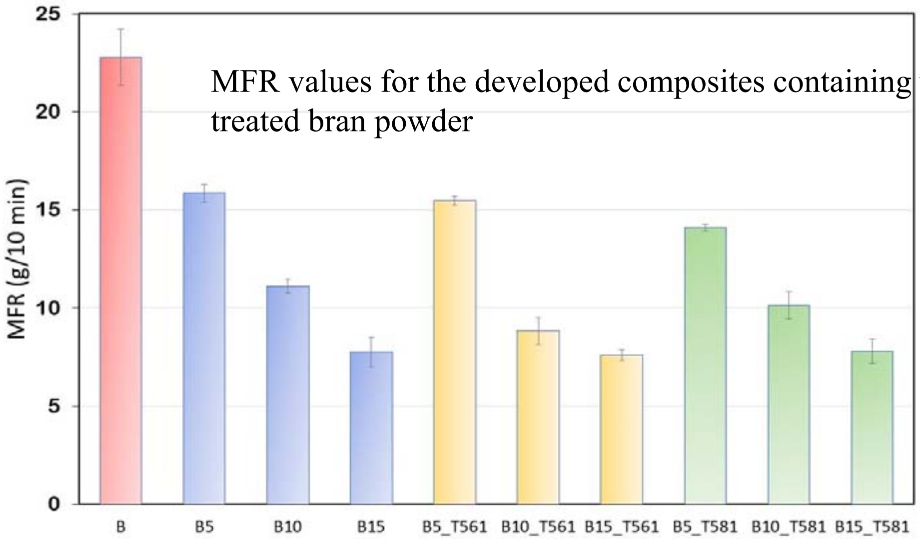
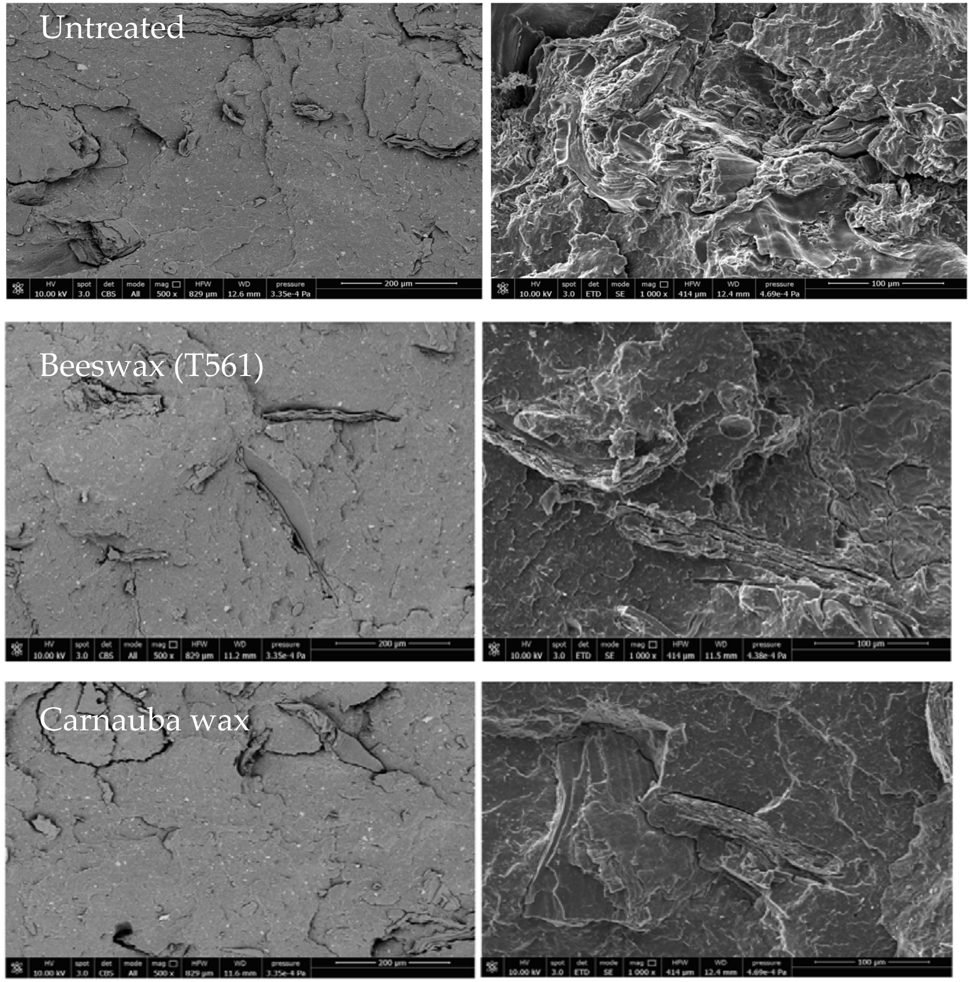
Bran Fibres treated with Natural waxes and PHBV

Table 1 – Composition (wt%) of the PHBV/bran biocomposites

Sample	PHBV	$CaCO_3$	ATBC	bran	T561 wax	T581 wax
Polymeric Matrix (M)	85	5	10	-	-	-
M + 5 bran (B5)	80.75	4.75	9.5	5	-	-
M+ 10 bran (B10)	76.5	4.5	9	10	-	-
M + 15 bran (B15)	72.25	4.25	8.5	15	-	-
M + 5 bran T561 (B5_T561)	80.75	4.75	9.5	4.75	0.25	-
M + 10 bran T561 (B10_T561)	76.5	4.5	9	9.5	0.5	-
M + 15 bran T561 (B15_ T561)	72.25	4.25	8.5	14.25	0.75	-
M + 5 bran T581 (B5_T581)	80.75	4.75	9.5	4.75	-	0.25
M + 10 bran T581 (B10_T581)	76.5	4.5	9	9.5	-	0.5
M + 15 bran T581 (B15_T581)	72.25	4.25	8.5	14.25	-	0.75



MFR values for the developed composites containing untreated and treated bran powder



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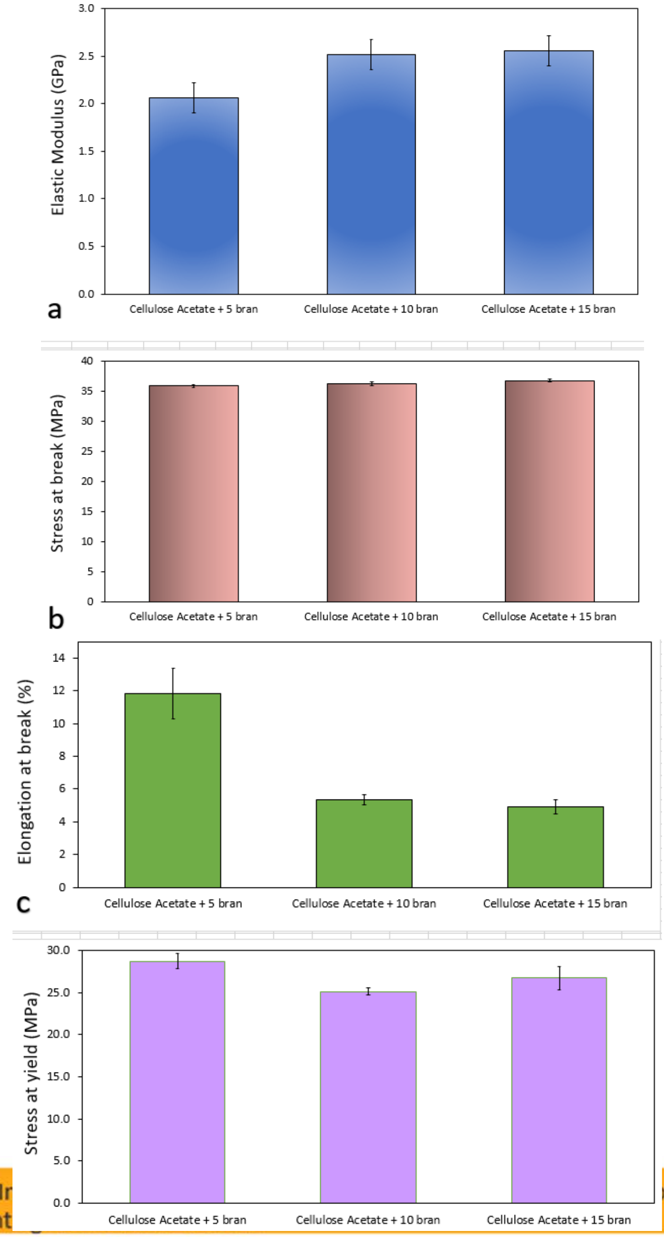
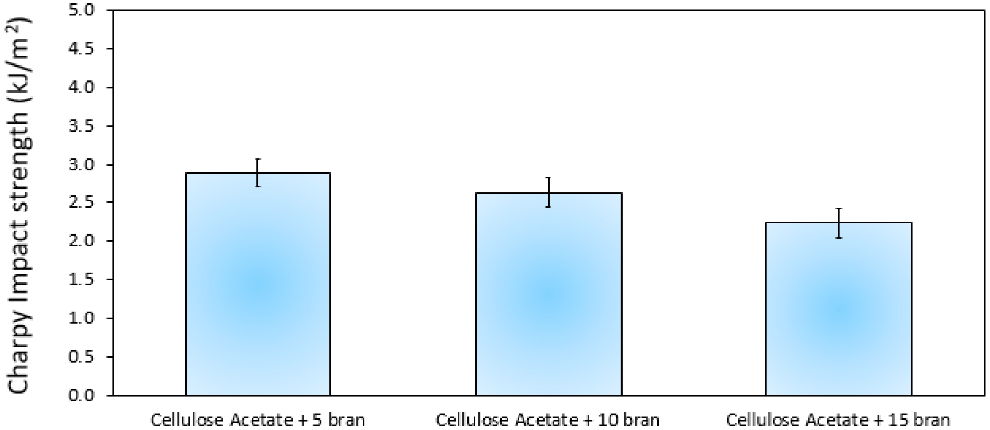
Cellulose di acetate (CDA) with depleted bran from Chiesa (after extraction of ferulic acid)





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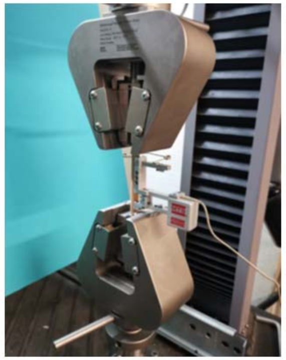
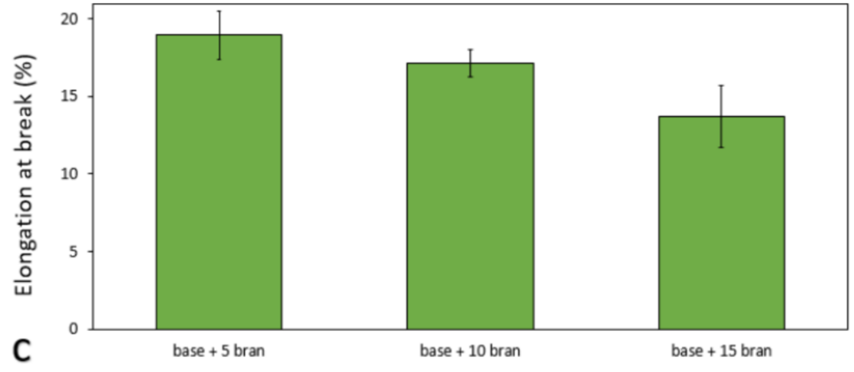
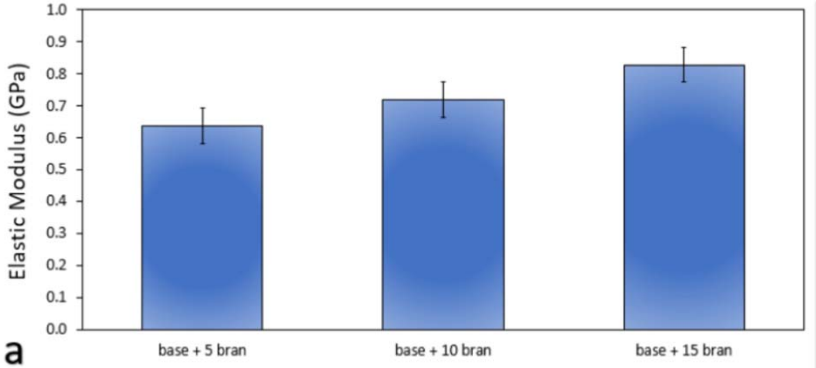
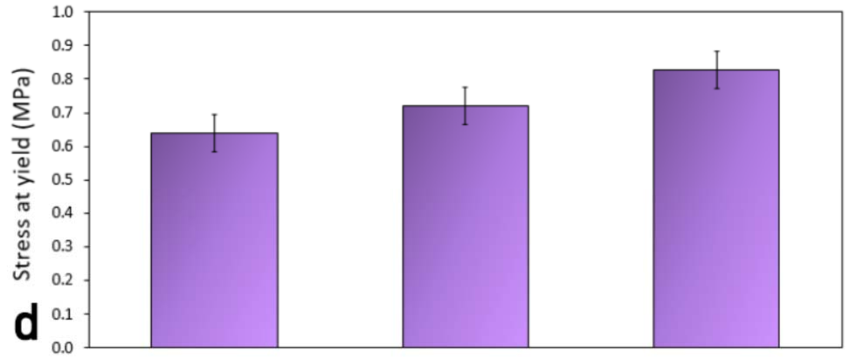
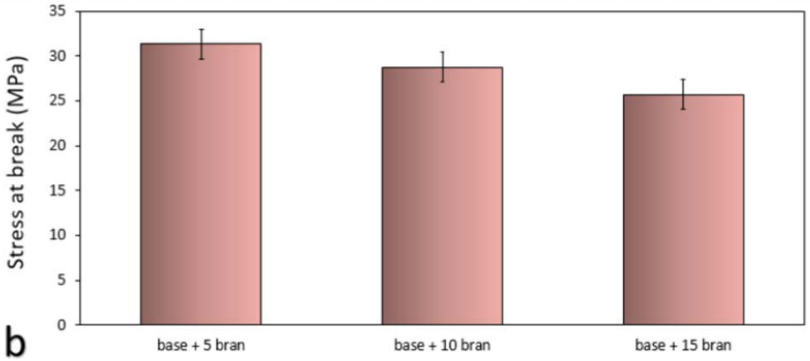


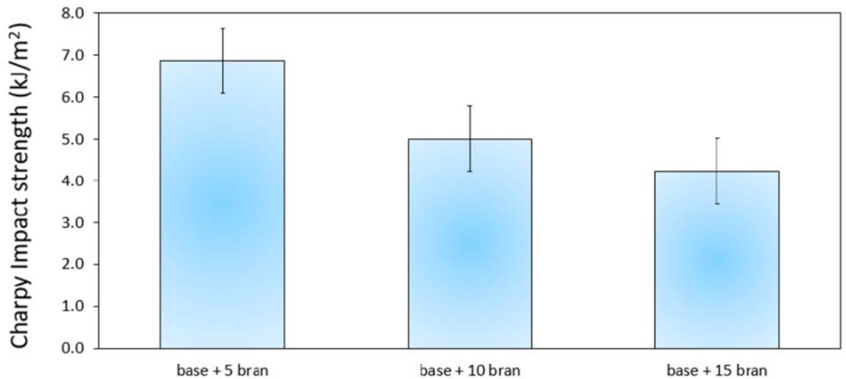
Poly butylene succinate (PBS) with depleted bran from Chiesa (after extraction of ferulic acid)











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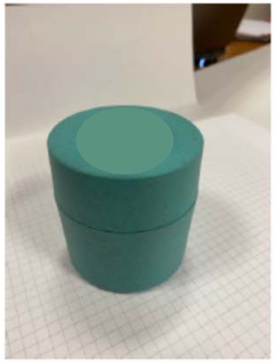
Rigid packaging boxes + 15% bran

BL33050117



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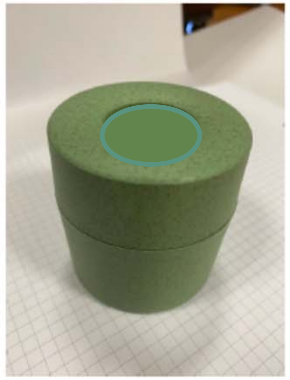

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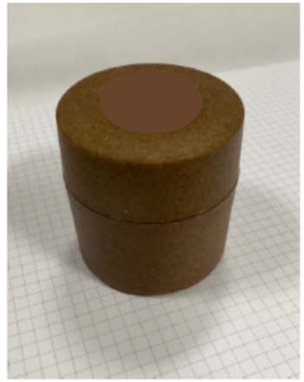
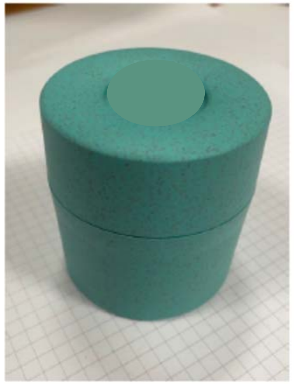


Senza coloranti

PBS based with different Bio-colours (4%)

PHBV based with different Bio-colours (4%)

BL33050117



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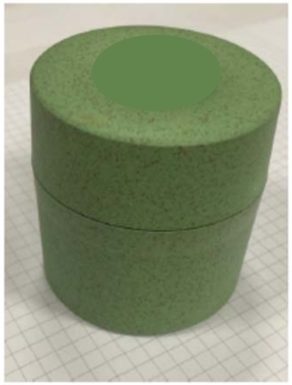

BL63300076

CDA based with different Bio-colours (4%)

BL33050117

BL52300027

BL63300076

Senza coloranti – presenta difetti evidenti in zona iniezione

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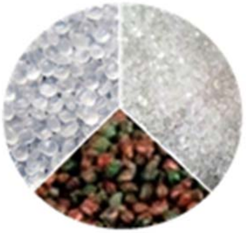
Fibers

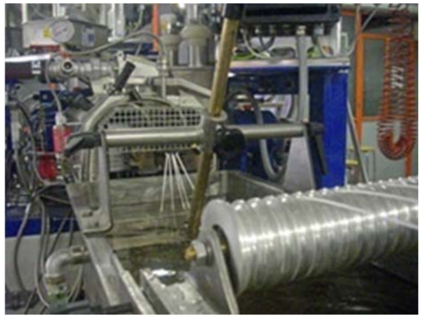
Natural Fibers

\+

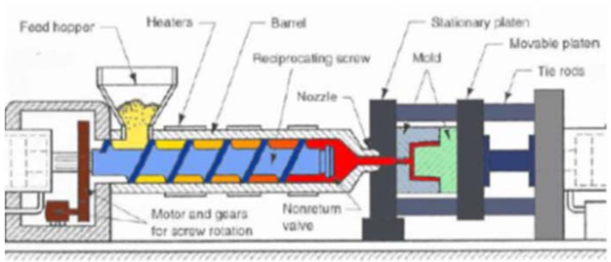


BioPlastics

BIOCOMPOSITES



Injection moulding machine



Injection moulding machine «Sandretto» model HP120

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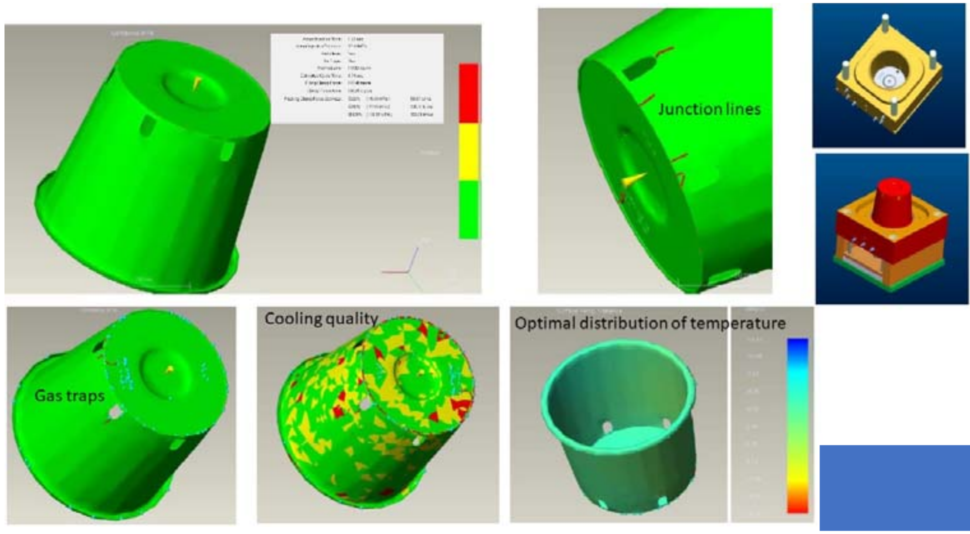

Simulation of the mould filling.

PP

PBSFZ71PB +10% Bran Fibres

PBS FZ71PB + 15% and 20% potato fibres

	Materials	Zone 1 (°C)	Zone2 (°C)	Zone 3 (°C)	Nozzle (°C)
pp	PP	160	165	165	150
PLA matrix	PLA 3251D + 15% potato fibres	189	188	197	190
	PLA 3251D + 20% potato fibres	192	190	200	190
	PLA2003D + 10%Bran+bees wax	170	170	175	170
PBS matrix	PBS FZ71PB + 10% potato fibres	158	160	165	150
	PBS FZ71PB + 15% potato fibres	187	188	198	190
	PBS FZ71PB + 20% potato fibres	190	190	200	190
PHBV matrix	PHBV (PHI002) + 15% Bran	169	171	174	170
Cellulose diacetate	CDA + 30%ATEC + 10% Bran	189	191	198	190

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PLA 3251D + 20% potato fibres

PLA + 11% bran treated with bees wax

PHBV + 15% bran.

CDA +30% ATEC+10% Bran

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Project next steps and implementation

Composites based on PLA, PHBV, CDA, PBS, PP and fibers showed a good processability by injection moulding up to 20% to 30% of fibre content.

The elongation at break decreased significantly with increasing fibers loading, while the Tensile strength decreased slightly. Young's modulus increased with increasing the fibre loading, as well as impact test energy absorbed. Bran behaves as a particulate fillers, adhesion is promoted by treatment of fibres with waxes (bees, carnuba) increasing impact resistance.

Fibres as by product of functional molecules extraction (depleted fibres) can be used as well for bio-composites production

The presence of fibres lowers cost, promotes biodegradation (disintegration) and increases sustainability

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THANKS FOR YOUR ATTENTION

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