New biodegradable compound intended for forest industry

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Contents

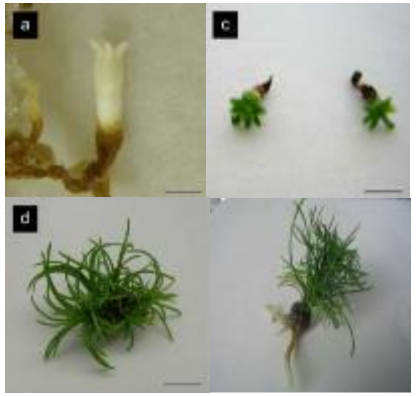
Introduction

- ✓ The most planted specie in the forest industry in Chile is Radiata Pine, which is cultivate 50.000 ha per year.
- ✓ The plant production in nurseries at industrial scale, requires large extensions of land and use of high amount of plastic to keep the plants at nursery.
- ✓ To reduce the accumulation of plastic in environment, more attention has been paid to development of biobased and biodegradable materials, that can be directly placed to the fields and degrade with the time.
- ✓ The use of these biodegradable material for capsules, to generate artificial seeds for the forest industry, emerges as a possible optimization of their clonal multiplication process, reducing the time and costs of production of plants.
- ✓ The aim of this work is development of biodegradable materials for potential application in clonal multiplication process of plants in forest industry.
- ✓ This work deals with optimization of blend formulations, in order to obtain biodegradable and non-phytotoxic materials, maintaining good mechanical properties, in terms of mechanical resistance to break and elasticity.

Technical Solution

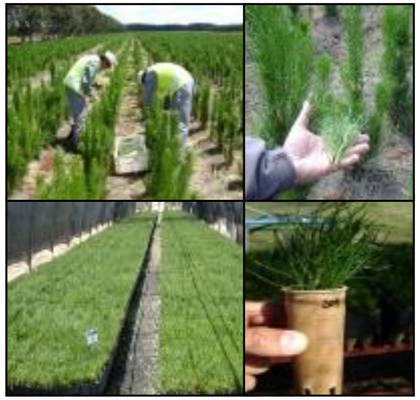
without proyect

Biotechnology Laboratory



Macro propagation

Hedge Garden



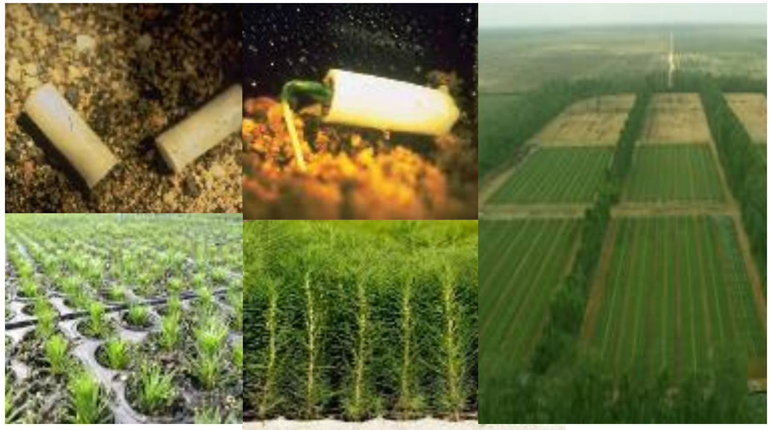
Nursery

Field

With proyect

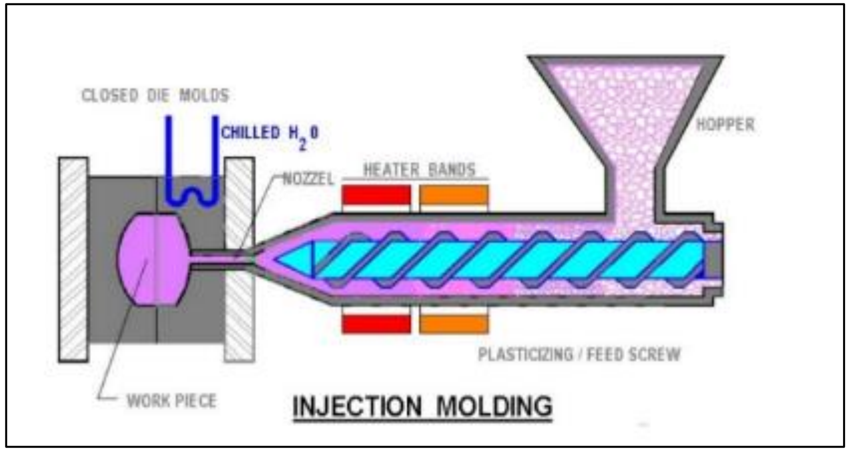
Biotechnology Laboratory

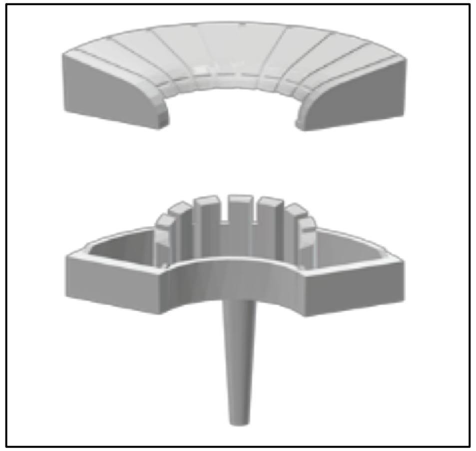
Nursery



Field

✓ The aim of the work, is to develop capsules for artificial seed: clonar propagation.





✓ Design and material are protected with a patent register number N°56.842

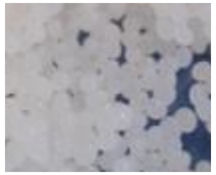
Methodology

Blends preparation

Materials

- PLA 3251 D (Ingeo)
- PBAT (Basf)
- Corn starch Buffalo

PLA / PBAT



Mixer 50 EHT, Brabender.
160°C; 10 min ; 60 rpm.

Cool mixer
Labtech
50°C
2800 rpm.

<u>Thermo-plasticized starch</u>

Glycerin

Biodegradable compound

Hydraulic press,
Labtech LP-20B .
pre-heated for 10 min at 170 ° C.
31 bar 10 min at 170 ° C
cooling 1 min

Castillo C., Nesic A., Urra N., Maldonado A. International Journal of Biological Macromolecules 122 (2019) 924–929

Blends characterization

Samples → Thermal Analysis → Mechanical Analysis

Formulation of PLA/PBAT/Starch blends

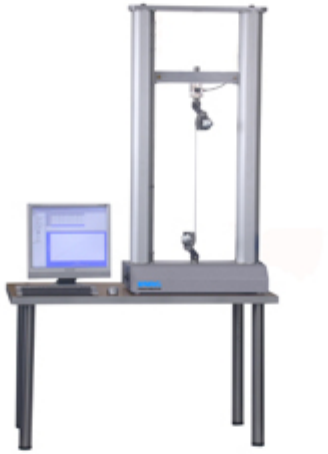
Samples	PLA 3251 D (%)	PBAT (%)	TPS (%)
P1	100	0	0
P2	60	40	0
P2-S10	54	36	10
P2-S20	48	32	20
P2-S30	42	28	30

DSC NETZSCH 204 F1 Phoenix®

N_2 flow (10 ml/min.)
Heating 25 to 250 °C
H. Rate 10°C/min

TGA NETZSCH TG209 F3 Tarsus

N_2 flow (10 ml/min.)
Heating 30 to 600 °C
H. Rate 10°C/min



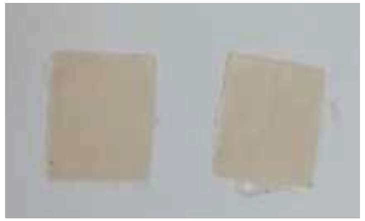
KARG Industrie technik Smartens 005

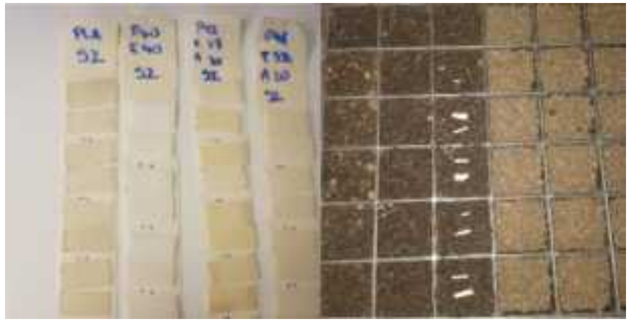
ASTM D-638(2010)
Load cell 5 kN
T°: 23 ± 2 °C
RH: 45 ± 5%
Dumbbell-shaped films:
5 mm x 25 mm x 1mm
Speed : 2 mm/min.

Methodology

Plates characterization

Plate Samples → Biodegradation → Phytotoxicity





Assays conditions:

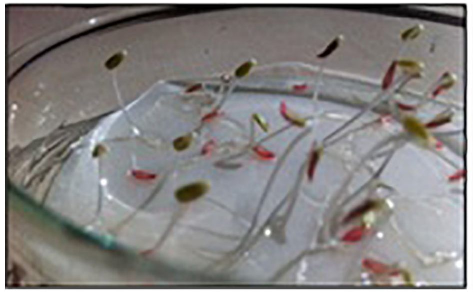
ASTM D5338
Factorial test 6x3
Control: Celullose
T° substrate: 25°C
RH: 75%
Samples:
2cm x 1cm x 0.8mm

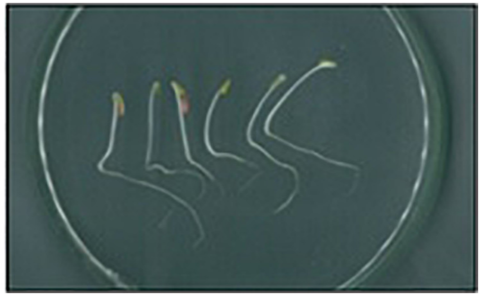
Characteristic of compost substrate:

Organic matter > 20%
Humidity: 30-45%
C/N ratio: < 50
Elec. Conduc.: 3 ds/m
App. Density: 0.5-0.7 kg/dm3
Ph: 5- 8.5
Origin: vegetable material-residue from agro and forest indutry.
Inet material: 2880 NCh

Indicator specie: *Lactuca sativa* (lettuce).
5 g of compost with 25 ml of distilled water for 3 h.
supernatant used for phytotoxicity test.

OECD 208 and ISO 11269-2.





$$PGR = \frac{N^{\circ}\text{ of seeds germinated in the extract} \times 100}{N^{\circ}\text{ of seeds germinated in the control}}$$

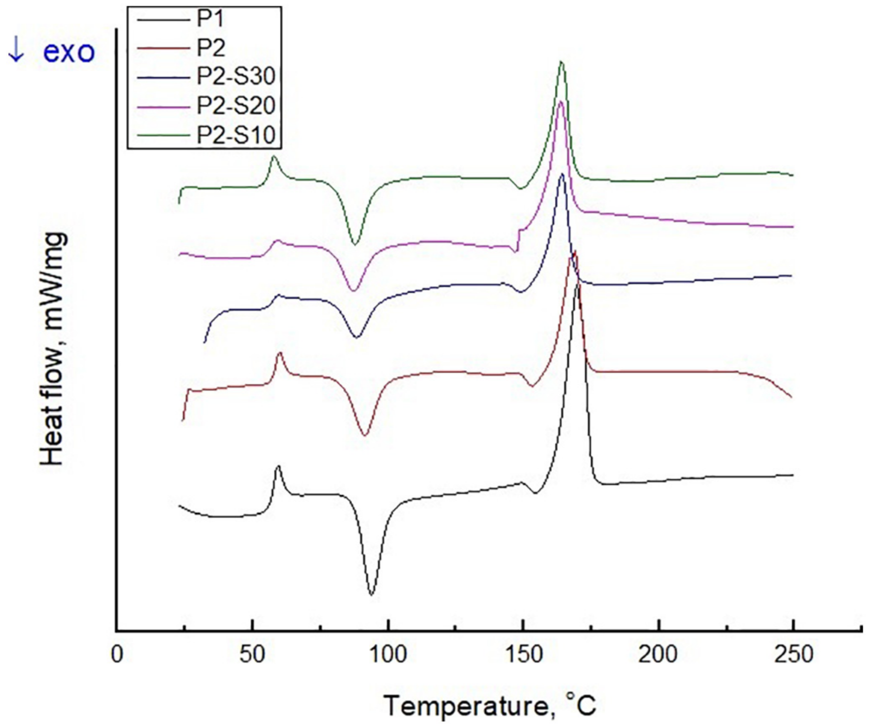
$$CRR = \frac{\textit{Elongation of radicles in the extract} \times 100}{\textit{Elongation of radicles in the control}}$$

$$IG = \frac{PGR \times CRR}{100}$$

Results

Thermal analysis - DSC

- ✓ The Tg has not been significantly changed with addition of PBAT and plasticized starch.
- ✓ The incorporation of TPS into PLA/PBAT system, induce the decrease of Tm in 5° C.
- ✓ The decrease of Tm in polymeric blends can be due to morphological effects (decrease in lamellar thickness) and to thermodynamics factors (polymer-polymer interaction).
- ✓ The PLA and PLA/PBAT blend as semi-crystalline components are partially miscible with plasticized starch.
- ✓ The glycerol used for plasticization of starch might have important role in organization of PLA chains which is evidenced by increase in cold crystallization enthalpy ΔHc value and lower Tc.
- ✓ A decrease of Tc value with an increase of TPS content in blends implies slower nucleation, thus having less nuclei with more space to grow. Hence, PLA chains have more time to be ordered and attain higher degree of crystallization.



DSC parameters for neat polymers and PLA/PBAT/Starch materials.

Sample	Tg(°C)	Tc(°C)	ΔHc(J/g)	Tm(°C)	ΔHm(J/g)
P1	59.3	93.7	27.3	169.6	55.0
P2	59.8	91.3	33.8	169.0	50.2
P2-S10	58.0	87.7	38.3	164.0	54.7
P2-S20	59.1	87.3	38.3	163.7	59.8
P2-S30	59.4	88.3	38.6	164.2	68.6

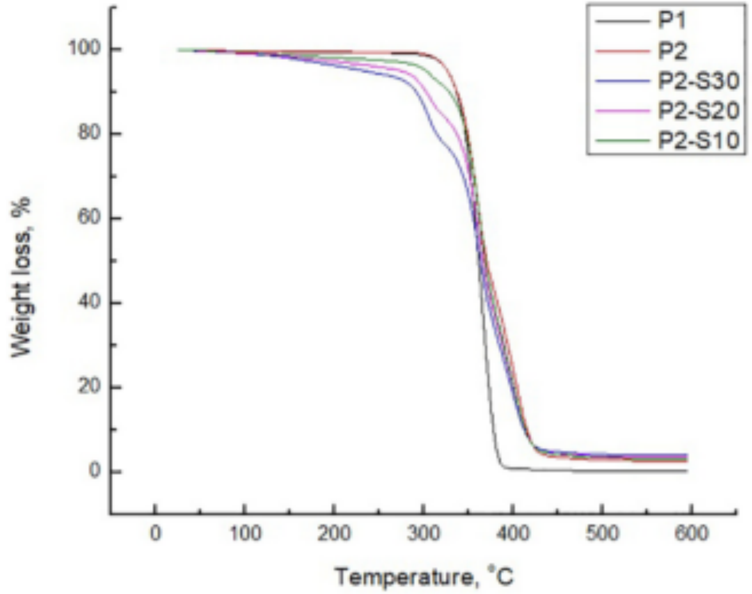
Results

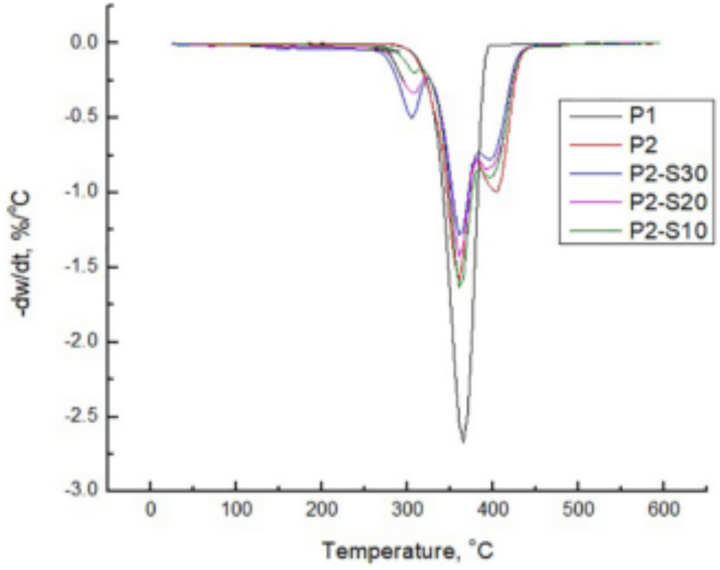
Thermal analysis - TGA

- ✓ Descomposition steps (see Table):
  1. Decomposition of amylose and amylopectin chains contained in TPS.
  2. The degradation temperatures of PLA chains at 361° C.
  3. Degradation of PBAT chains at 397° C.
- ✓ The main degradation peaks of PLA and PBAT are shifted to slightly lower temperatures with incorporation of TPS.
- ✓ The presence of TPS causes the chain segments mobility of these two polymers and that is only partially miscible with PLA and PBAT.

TGA parameters of neat polymers and their blends.

Sample	T_{onset}, °C	T_{deg1}, °C	T_{deg2}, °C	T_{deg3}, °C	Char residue, °C
P1	308	–	365	–	0.35
P2	305	–	360	404	2.81
P2-S10	281	307	361	397	4.1
P2-S20	276	306	361	397	3.9
P2-S30	275	305	361	397	3.4

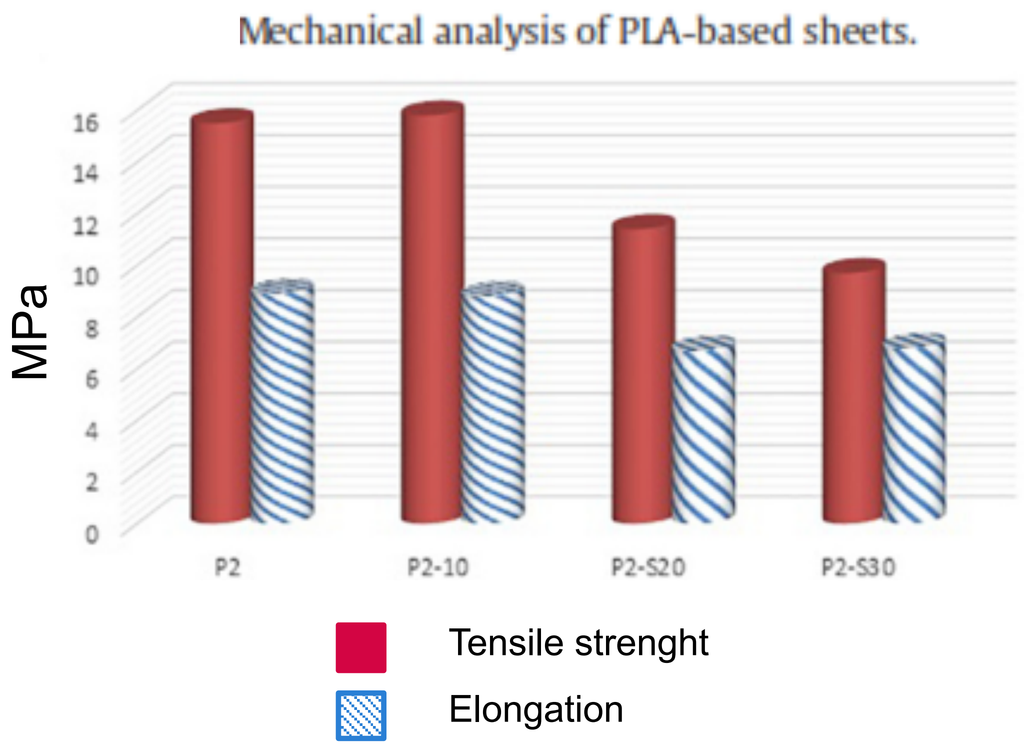




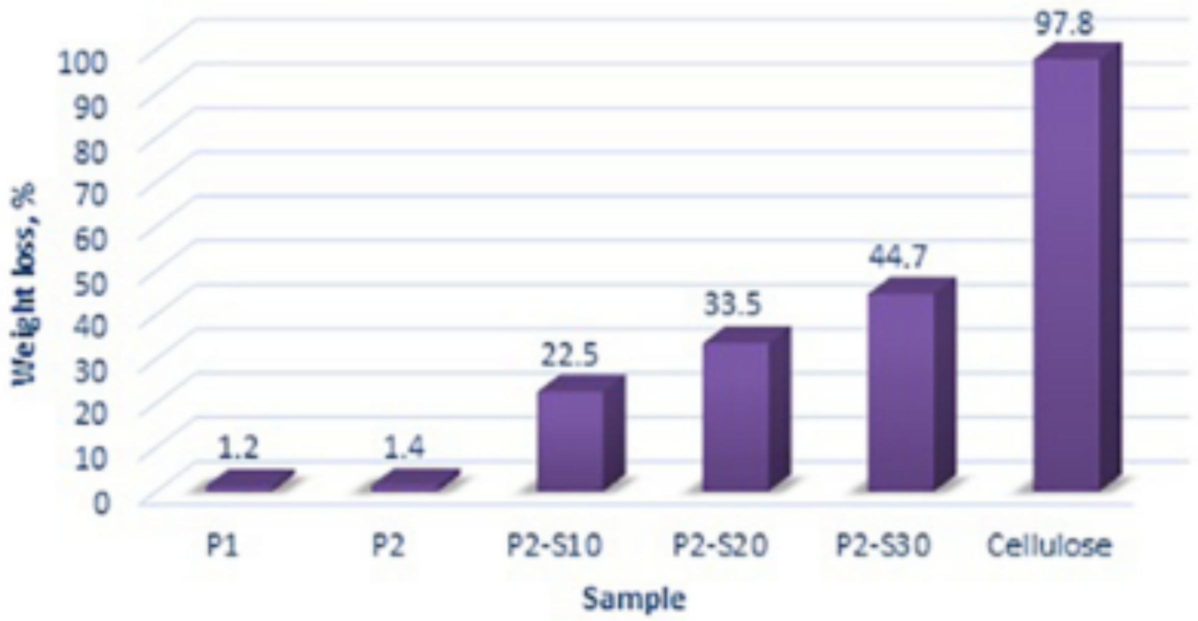
Results

Mechanical analysis

- ✓ Control PLA (P1) sheet shows to be very fragile and it was not possible to measure mechanical parameters.
- ✓ The addition of PBAT enhances the ductility of PLA-based sheets due to its elastomeric behavior.
- ✓ The incorporation of 10% of TPS does not have significant influence on tensile strength and elongation at break of final materials.
- ✓ Higher concentration of TPS leads to decrease in tensile strength for 37% (P2-S30) and elongation at break for 25% (P2-S30).
- ✓ An increase in TPS content generally lowered the tensile strength of PLA/TPS blends, due to low interfacial adhesion.

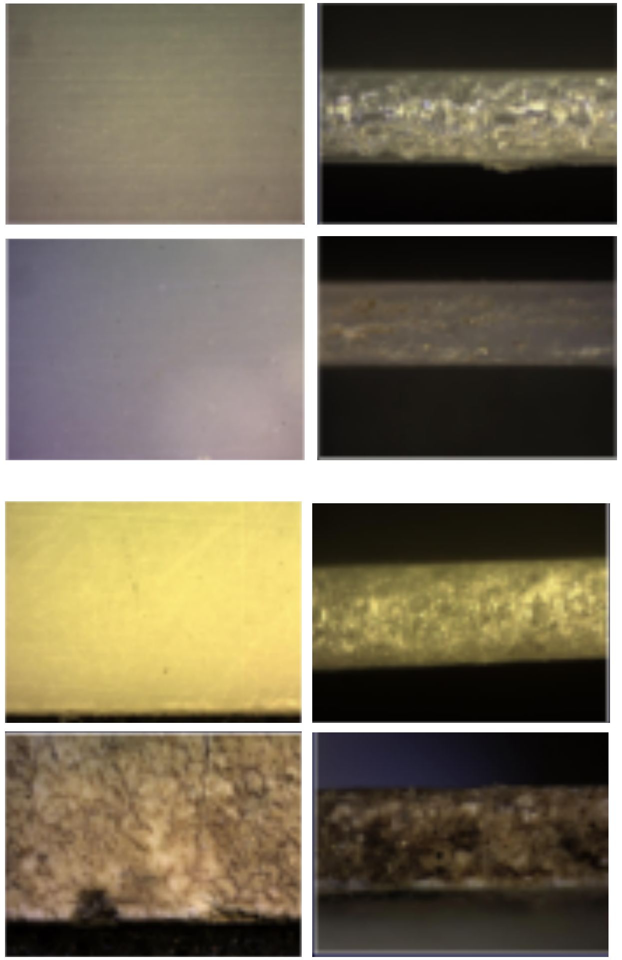


Biodegradation analysis



Biodegradation rate after 90 days.

- ✓ After 90 days samples has changed color, deep cracks appear on the surface, easily subjected to breaking.
- ✓ Softening of materials it is in direct proportion to the amount of TPS contained in the samples.
- ✓ TPS significantly influences the biodegradation rate, which is directly related to the percentage of organic load they contained, facilitating the penetration of microorganism into the blends.



P2 Day 1

P2 Day 90

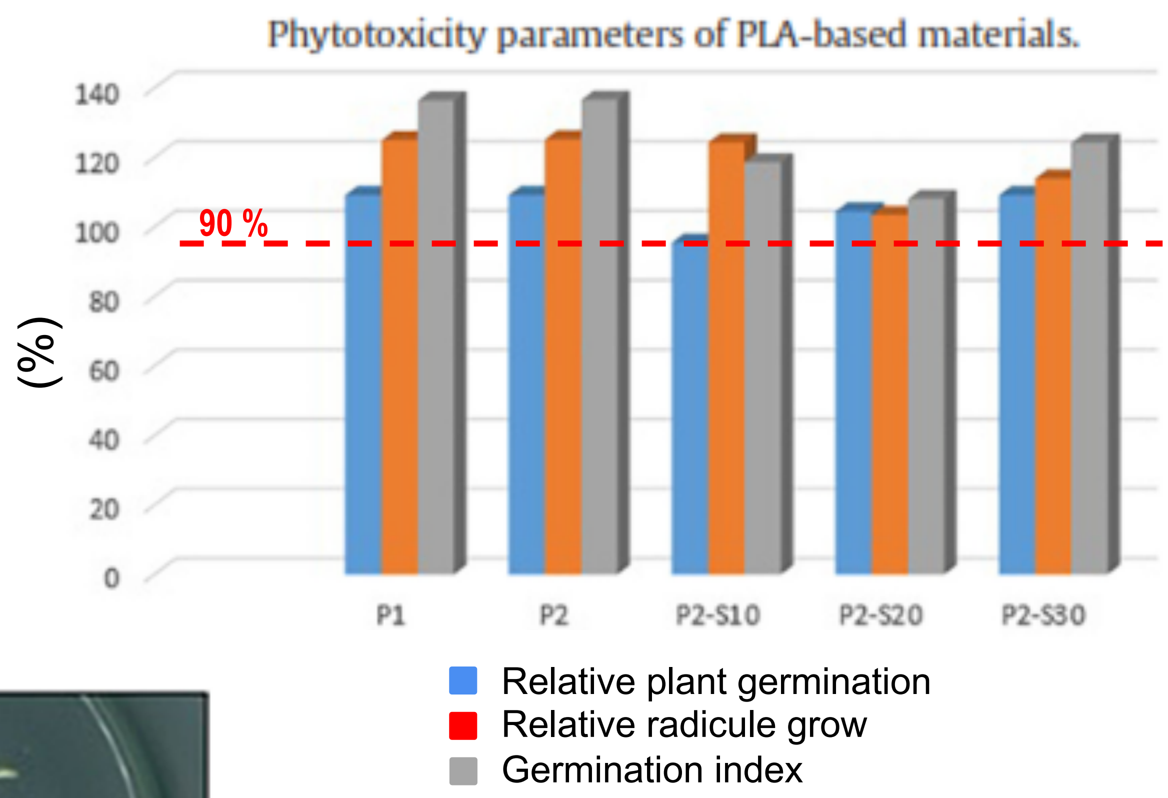
P2-S30 Day 1

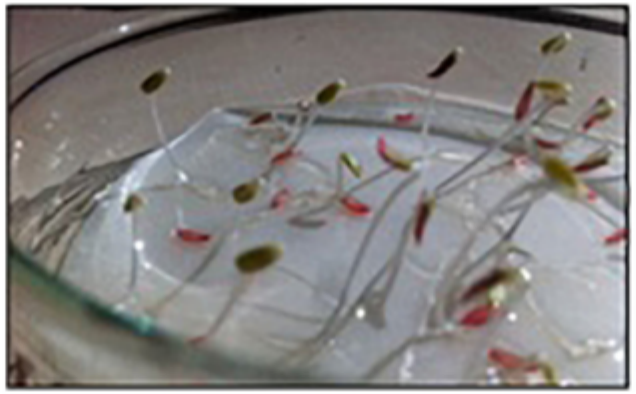
P2-S30 Day 90

Plate sample

Phytotoxicity and germination analysis

- ✓ According to OECD 203 and ISO 11269-2, the compost is non-toxic when the germination rate or the plant seedling weights are higher than 90%.
- ✓ The germination and the root growth are significantly higher than 90%.
- ✓ The degradation of all tested samples in the compost doesn't contaminante the soil, neither represents harmfull effects for the plants.
- ✓ The three parameters analyzed for PLA/PBAT/TPS samples are slightly lower than PLA and PLA/PBAT samples (see Figure).





Conclusions

- ✓ Biodegradable blends made of PLA, PBAT and TPS were prepared and characterized by different techniques.
- ✓ Although the incorporation of TPS into the PLA/PBAT blends lowered thermal and mechanical resistance, still fit well into the scope of forestal application and can be competitive with synthetic-based materials.
- ✓ PLA/PBAT/TPS blends showed remarkable biodegradation rate and no phytotoxic effect on germination of tested seeds.
- ✓ P2-S30 showed the highest biodegradation rate maintaining the good mechanical, thermal and non-phytotoxic properties.
- ✓ In order to validate the real potential of PLA-based materials in forest industry, P2-S30 formulation was chosen for further experiments.
- ✓ To prove the real potential of these blends in forest industry as a biodegradable carrier for multiplication of plants, the forest industry should made tree growing trials using biodegrables capsules (lab → field).
- ✓ The design of biodegradable artificial seed and further results related to forest industrial application are protected by Patent CL201603373 register number N°56.842 “A biodegradable system for clonar multiplication of wood plants, forest species or fruit trees for masive production”.

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Thanks for your attention!

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