Valentina F. Domingues

REQUIMTE/LAQV, ISEP, Polytechnic of Porto

Jointly elaborated by: Aix Marseilhe Université

AGRIMA

ECO-FRIENDLY BIOPLASTICS AND BIOCOMPOSITES

PART 2 | Biocomposites from chestnut industry waste: production and characterization

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Agri-food Waste Management for Sustainable bio-economy through Higher Education curricula and upskilling

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Goals

AGRIMA aims to foster universities' **capacity building** for the **green transition** through **innovative practices and higher education curricula updating in agri-food waste management** for the **circular bioeconomy**.

AGRIMA addresses:

1. **Advancing pedagogical methods** for industrial agri-food waste valorisation **based on business-academia synergies**.
2. **Integrating citizen science** in bio-economy-enhanced waste valorisation as a means of **civic engagement and environmental advocacy**.

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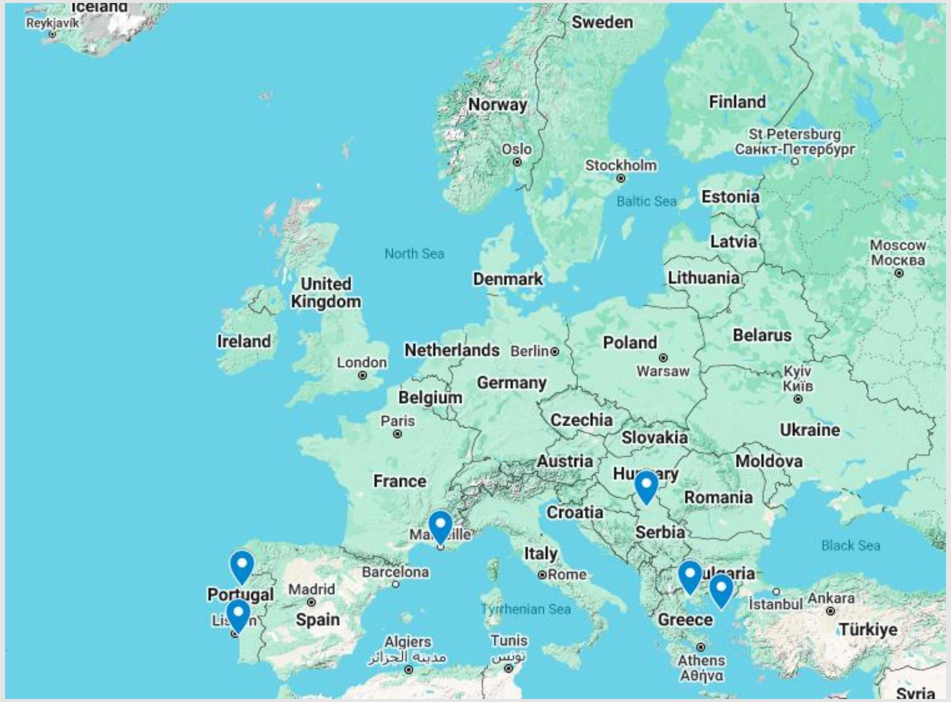
Partners



Aix Marseille université – Socialement engagée

AVIPE – Associação de Viticultores do Concelho de Palmela

P.PORTO

University of the Aegean

University of Novi Sad

Web2Learn – Open, social learning

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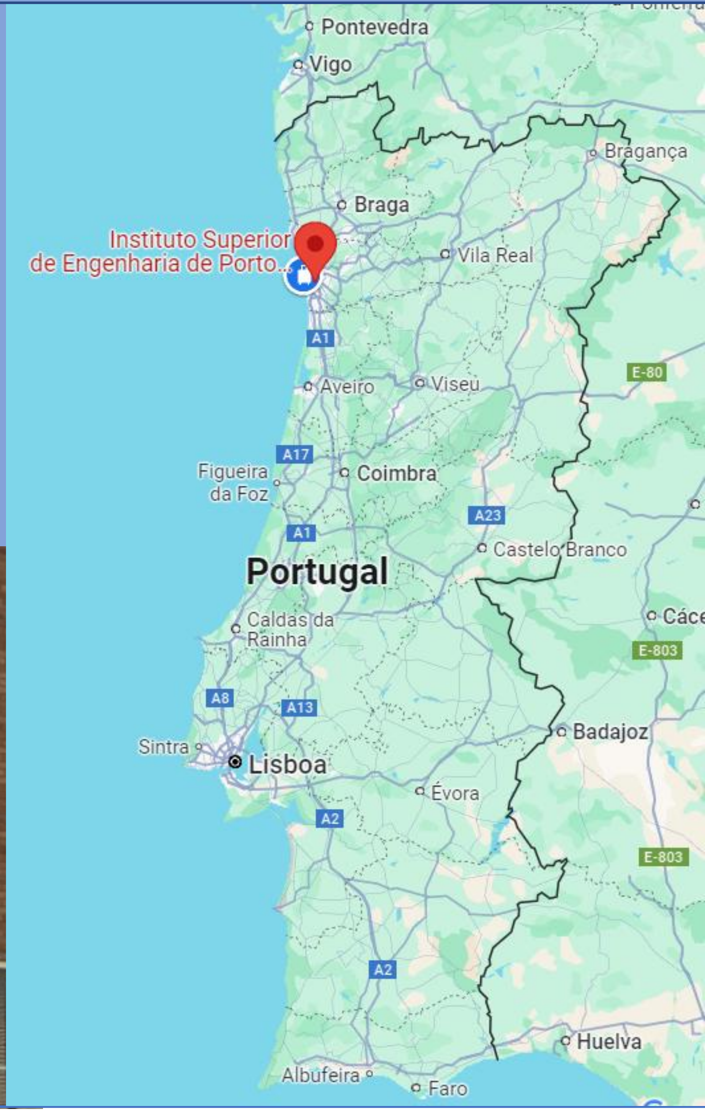
Cádis

ISEP INSTITUTO SUPERIOR DE ENGENHARIA DO PORTO

P.PORTO

One of the top schools of technology in Portugal, **ISEP** has been pioneering education and research in Engineering since **1852** and offers a wide range of programs in different fields of Engineering, with more than **6000 students**.



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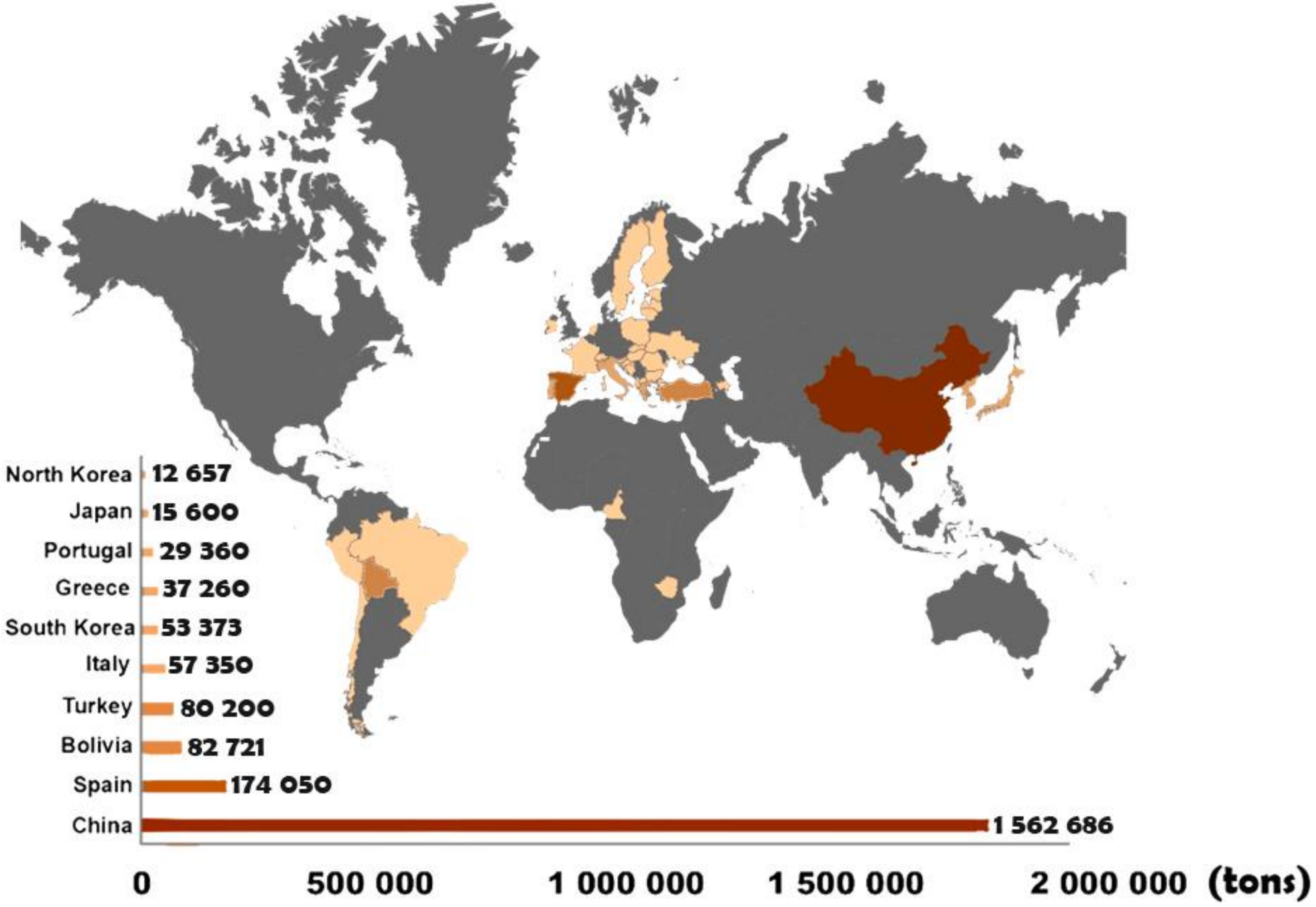
Research group
Environmental Chemistry

- **Contaminants**
- **Biocomposites**
- **Biomonitoring**
- **Food**
- **Phytotoxicity**

Chestnut Production

The production area has more than **doubled** in the last **20 years**, according to the latest FAO data.

China stands out in 1st place, while **Portugal** is in **8th place**.



FAO - Food and Agriculture Organization

Consumption

Asia

2.5 kg per person

Europe

0.5 kg per person

EUA

0.05 kg per person

Consumption

Production Chain

1
Farming

2
Processing

3
Distribution

4
Retailing

5
Home

40-50%

Chestnuts are lost to infestations, rot and processing residues.

12.000 ton

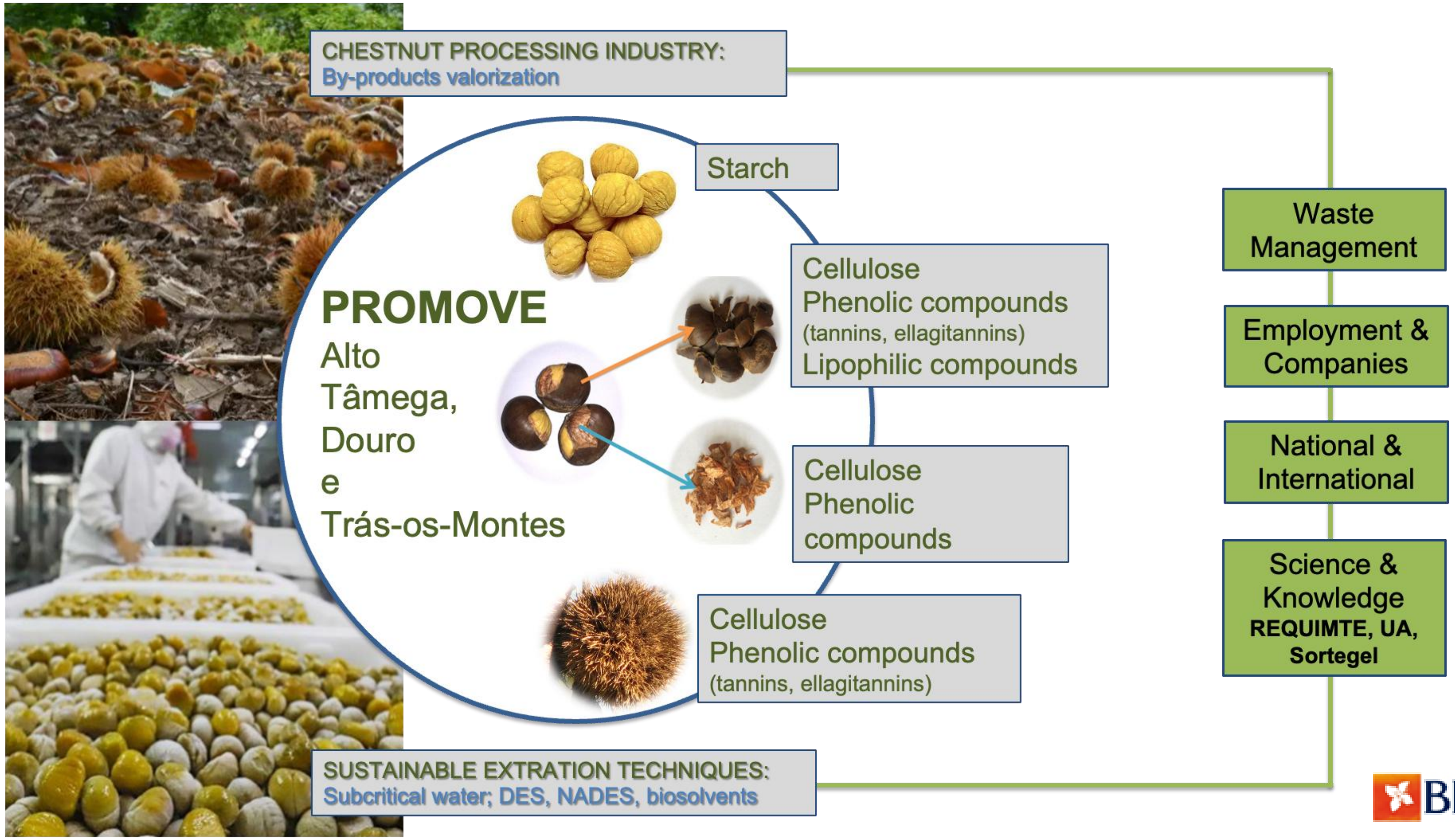
Estimated annual weight loss

24 M €

Estimated annual economic loss

Chestfilm

Valorization of solid residues from processing of chestnuts



Chestfilm project

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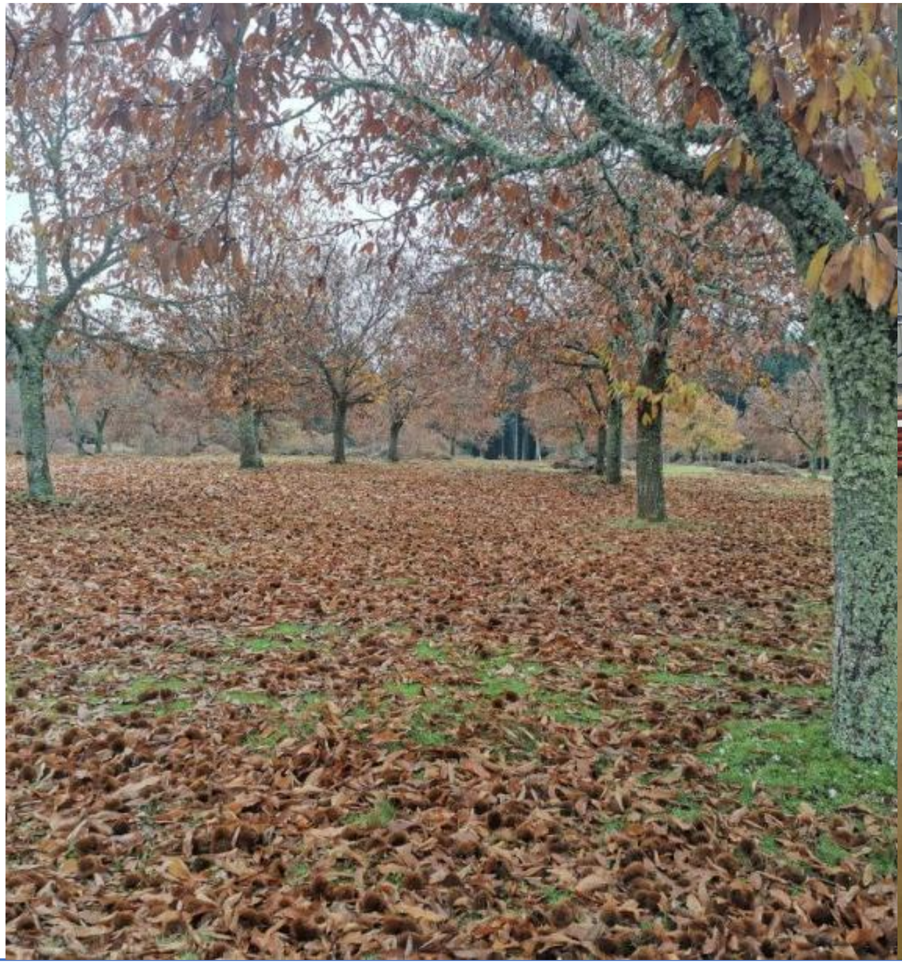
Industrial Processing

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Industrial Processing

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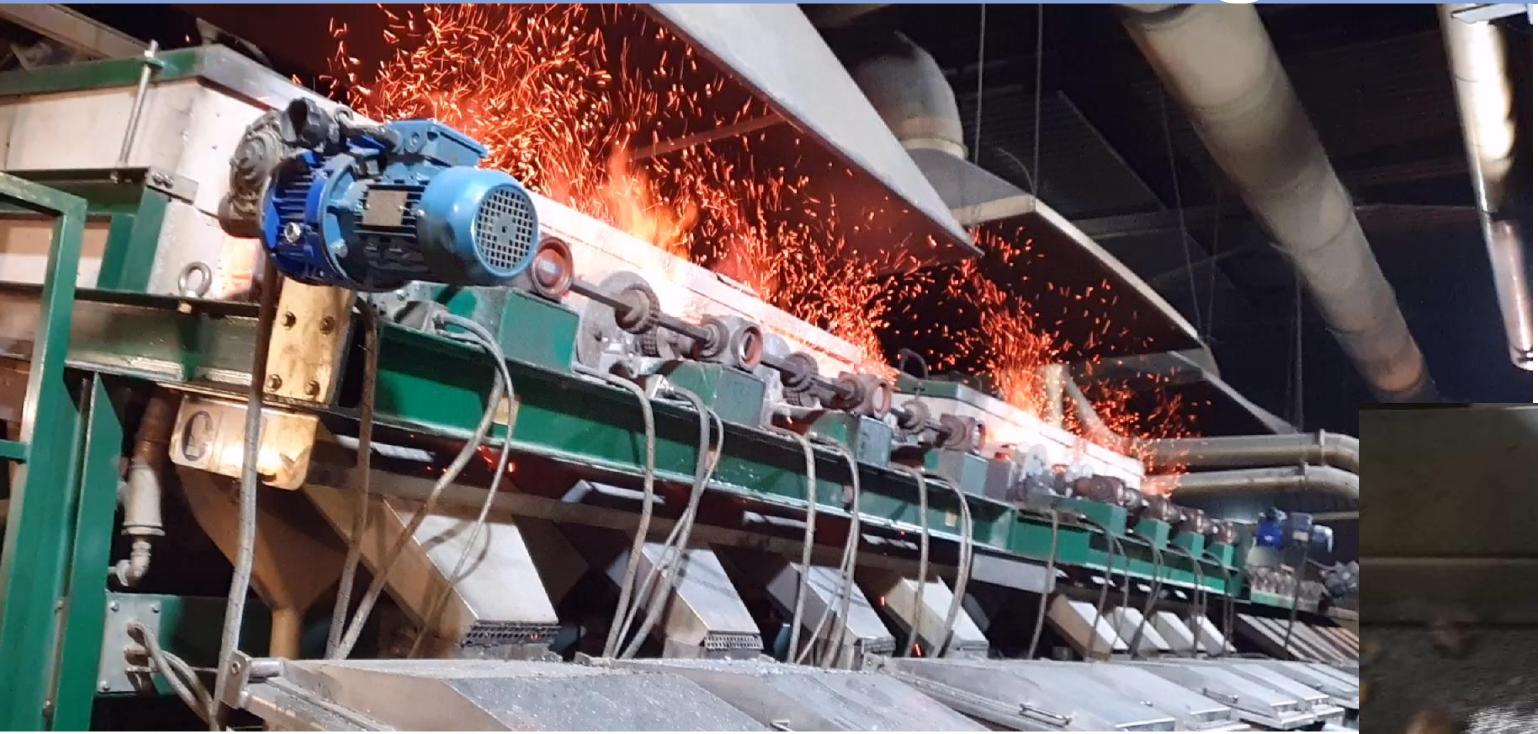
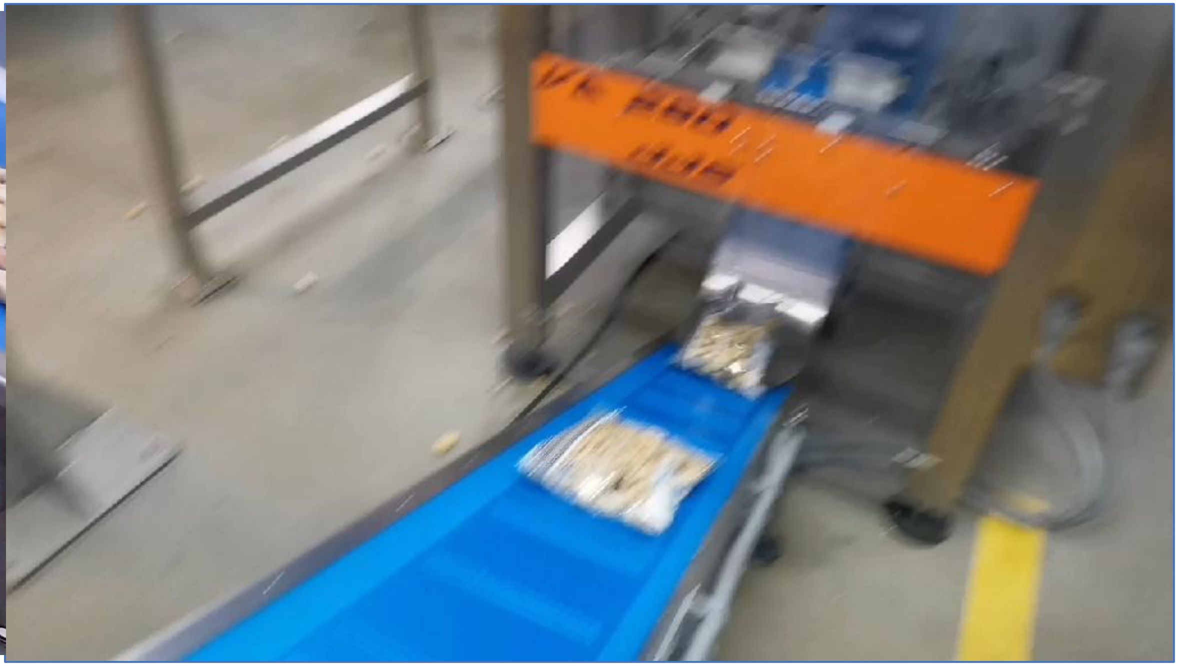


Industrial Processing

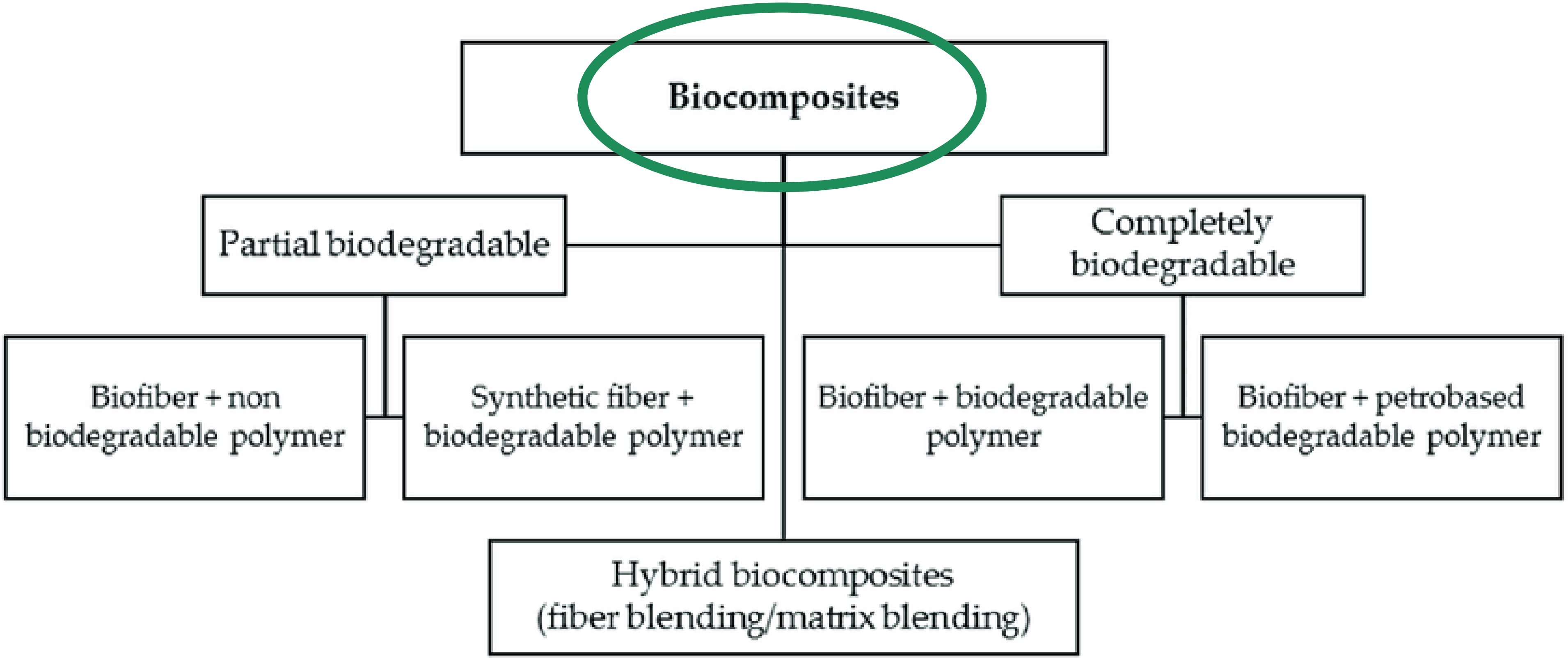
Industrial Processing



Biocomposites

Material made from **two or more distinct components**, **with at least one being naturally sourced**, combined to create a new material that offers **enhanced performance** compared to its individual components.

Biocomposites



Bahrami, M.; Abenojar, J.; Martínez, M.Á. *Materials* 2020, 13, 5145.

Biodegradable polymers

- **Biomass products** (from agro-resources)
  - **Polysaccharides**
    - Starches (wheat, potatoes, maize…)
    - Ligno-cellulosic products (wood, straws…)
    - Others (pectin, chitosan/chitin, gums…)
  - **Proteins, lipids**
    - Animals (casein, whey, collagen/gelatin…)
    - Plants (zein, soya, gluten…)
- **From Microorganisms** (obtained by extraction)
  - **Polyhydroxyalkanoates (PHA)**
    - Poly(hydroxybutyrate) (PHB)
    - Poly(hydroxybutyrate) co-hydroxyvalerate (PHBV)
- **From biotechnology** (conventional synthesis from bio-derived monomers)
  - **Polylactides**
    - Poly(lactic acid) (PLA)
- **From petrochemical products** (Conventional synthesis from synthetic monomers)
  - **Polycaprolactones (PCL)**
  - **Polyesteramides (PEA)**
  - **Aliphatic co-polyesters** (eg PBSA)
  - **Aromatic co-polyesters** (eg PBAT)

Chestnut composition

Fruit

Starch	38 – 63 g/100 g
Proteins	4 – 11 g/100 g
Sugars	7 – 31 g/100 g

Burr

Lignin	24 g/100 g
Cellulose	33 g/100 g

Shell

Internal

Outside

Lignin	42 – 47 g/100g
Cellulose	21 – 29 g/100g

STEPS

Production

- Pretreatment
- Preliminary tests
- Optimization (Box-Behnken design)

Characterization

- Mechanical properties
- Wettability
- Water absorption
- Thermal conductivity
- TGA and DSC
- SEM
- Phytotoxicity

Application

- Customized press
- Production of a candle holder

Production | Pretreatment

Chestnut with no commercial value

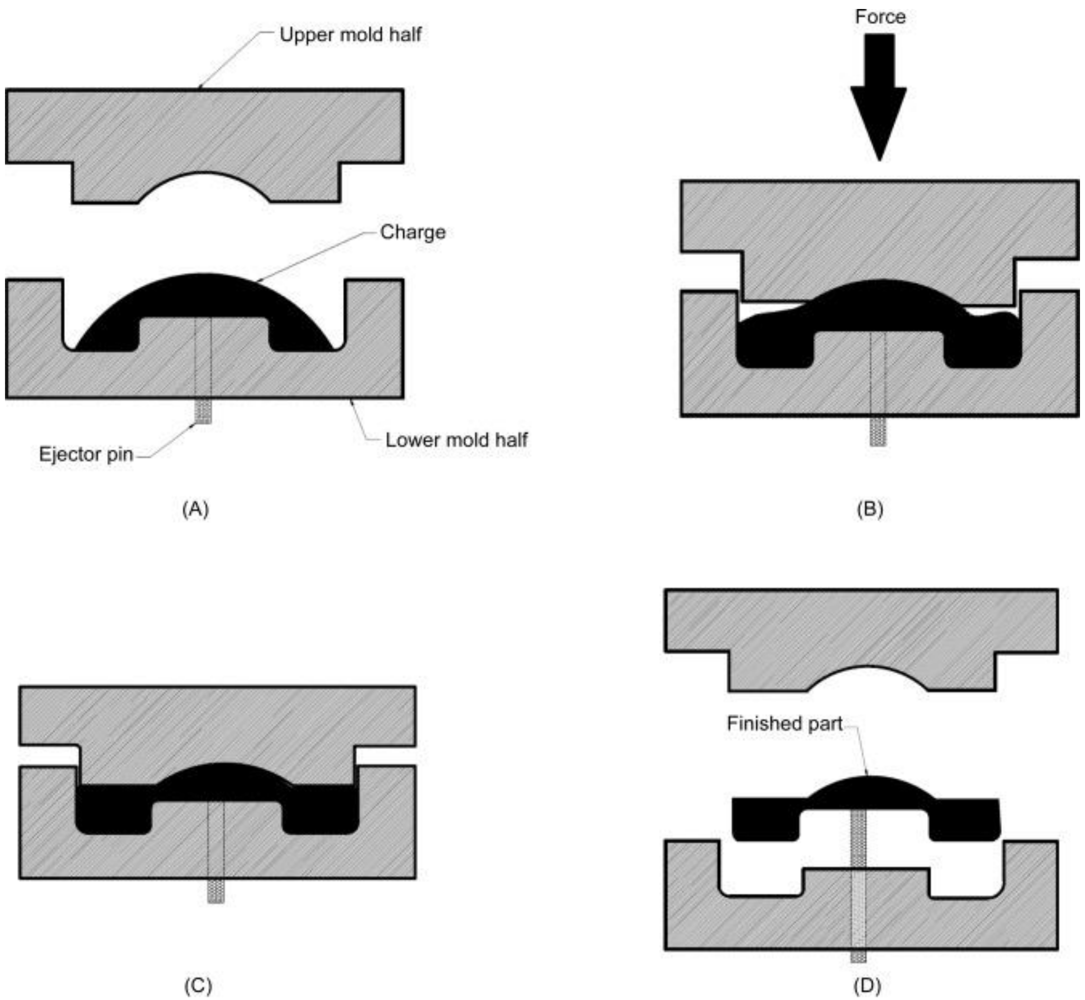
Ø < 20 mm hollow nut rotten nut

Dehydrator

Ground into 2 mm particles

Production

Compression molding is a simple process of pressing or squeezing a deformable material charge between two halves of a heated mold and its subsequent transformation into a molded part after cooling or curing.



Robert A. Tatara, 14 - Compression Molding, Editor: Myer Kutz, In Plastics Design Library, Applied Plastics Engineering Handbook (2nd Edition), William Andrew Publishing (2017), 291-320

Production | Preliminary tests

Silva, S.B.; Freitas, O.M.; Vieira, E.F.; Gomes, A.; Carreiras, A.R.; Moreira, D.C.; Esfandiari, P.; Silva, J.F.; Delerue-Matos, C.; Domingues, V.F. Polymers 2025, 17, 616.

Preliminary Tests

- ✓ Hot compression moulding
- ✓ Disc-shaped specimens (Ø 160 mm)
- ✓ Pressure of 6 tonnes for 30 min

Qualitative Tests

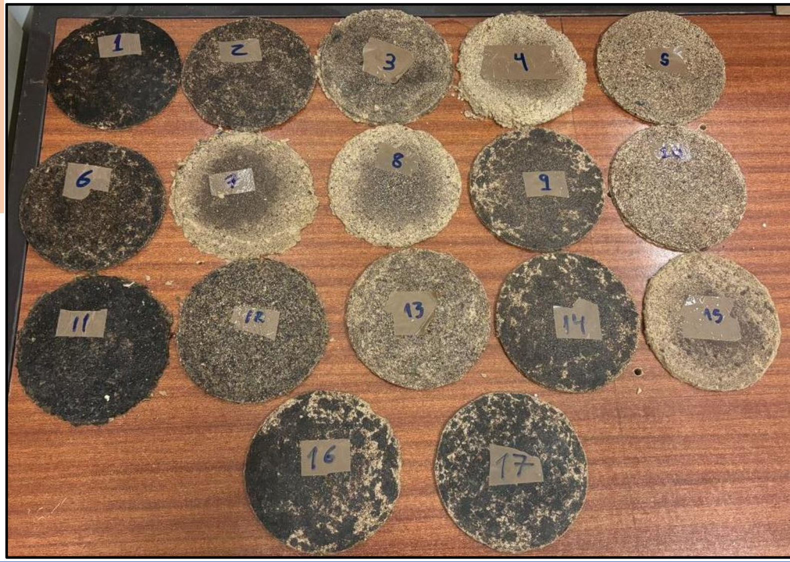
- ✓ Chestut (70-90%) |Glycerol (0-30%)
- ✓ Ideal temperature defined: 80-120 °C
- ✓ Flexural strength and modulus of elasticity evaluated

Response Surface Methodology (RSM)

Box-Behnken Design (BBD)

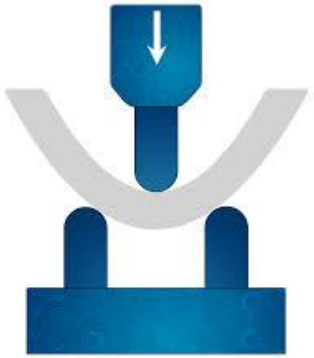
Symbol	Independent Variable	Actual levels at coded factor levels		
		-1	0	1
X1	Chestnut (%)	65	75	85
X2	Glycerol (%)	0	15	30
X3	Temperature (ºC)	80	100	120

Run	X1 (%)	X2 (%)	X3 (ºC)
1	65	15	120
2	65	30	100
3	75	15	100
4	75	0	80
5	75	30	80
6	85	15	120
7	75	0	120
8	65	0	100
9	75	15	100
10	65	15	80
11	75	30	120
12	85	30	100
13	85	15	80
14	75	15	100
15	85	0	100
16	75	15	100
17	75	15	100

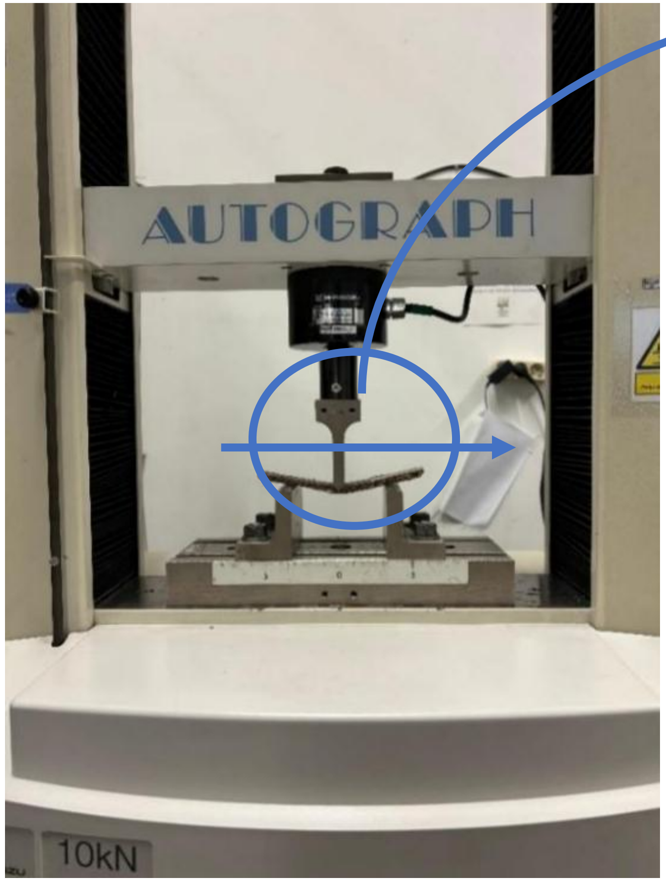


Silva, S.B.; Freitas, O.M.; Vieira, E.F.; Gomes, A.; Carreiras, A.R.; Moreira, D.C.; Esfandiari, P.; Silva, J.F.; Delerue-Matos, C.; Domingues, V.F. Polymers 2025, 17, 616.

Characterization | Flexural strength and Flexural modulus

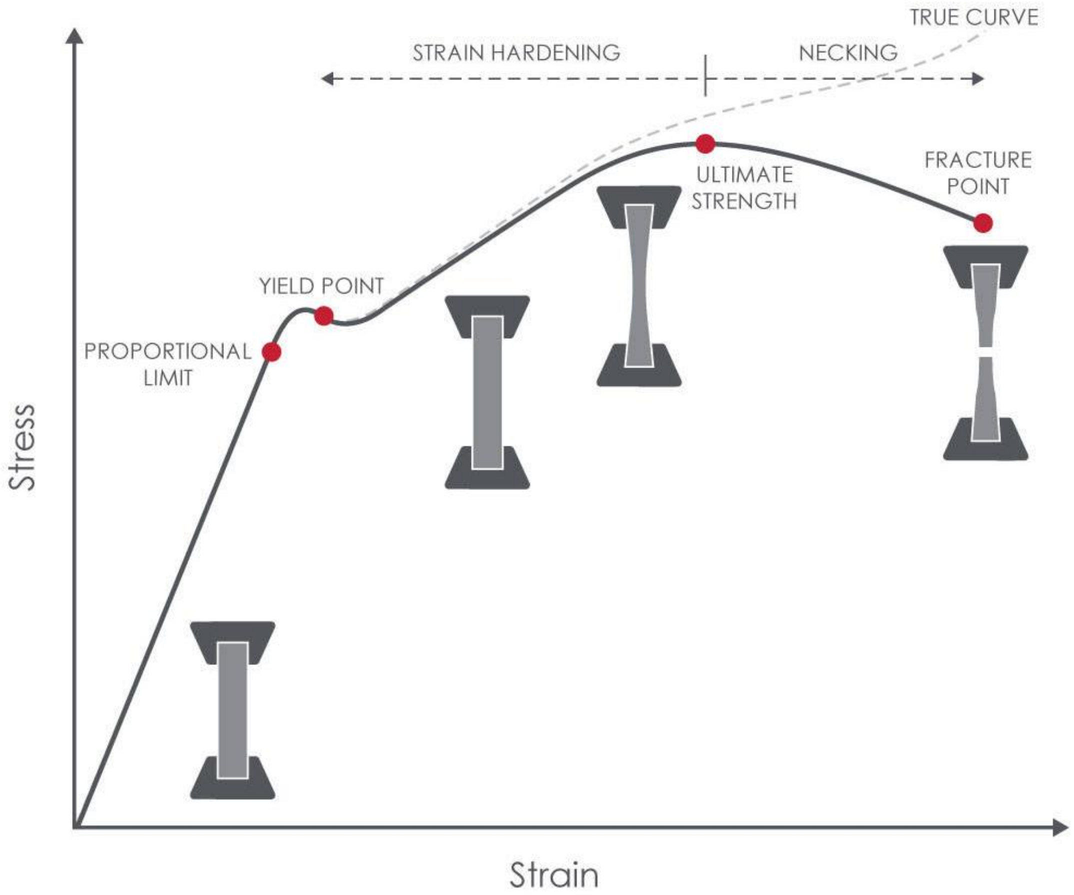


ISO 14125



SHIMADZU AG-I 10KN

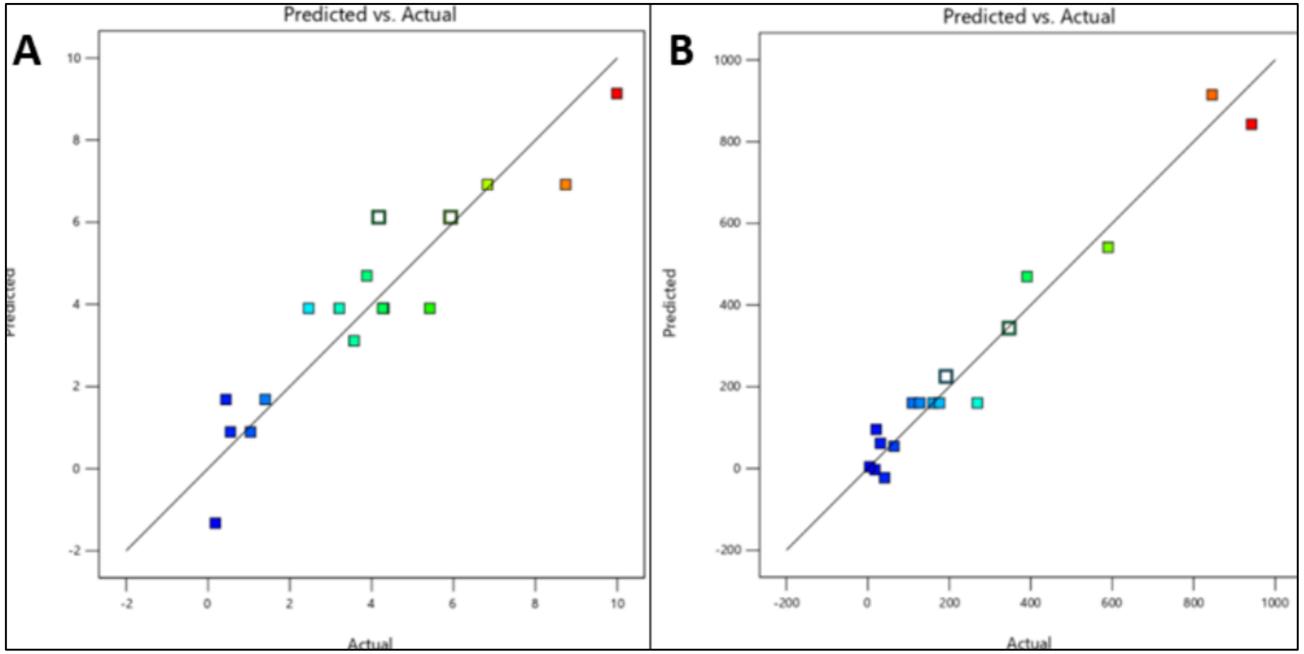
Maximum flexural point
(flexural strength)



Production | Optimization

Run	Dependent Variables			
	Flexural Strength (MPa)		Flexural Modulus (MPa)	
	Exp	Pred	Exp	Pred
1	5.9 ± 1.3	6.1	350 ± 120	343
2	0.55 ± 0.14	0.9	17.3 ± 3.8	−3
3	3.21 ± 0.51	3.9	160 ± 17	160
4	3.9 ± 1.7	4.7	390 ± 120	470
5	0.177 ± 0.020	−1.3	4.31 ± 0.65	3.8
6	4.17 ± 0.36	6.1	192 ± 17	225
7	9.90 ± 0.14	9.1	850 ± 280	910
8	8.7 ± 1.0	6.9	940 ± 230	840
9	4.3 ± 2.4	3.9	130 ± 20	160
10	0.44 ± 0.15	1.8	20 ± 11	90
11	3.6 ± 2.9	3.1	64 ± 18	54
12	1.04 ± 0.20	0.9	30.8 ± 5.3	61
13	1.40 ± 0.46	1.7	41 ± 11	−22
14	2.46 ± 0.39	3.9	110 ± 19	160
15	6.83 ± 0.78	6.9	590 ± 130	540
16	5.42 ± 0.53	3.9	270 ± 73	160
17	4.27 ± 0.17	3.9	180 ± 41	160

Actual vs. Predicted (based on BBD evaluation) values of Flexural Strength **(A)** and Elastic Modulus **(B)**



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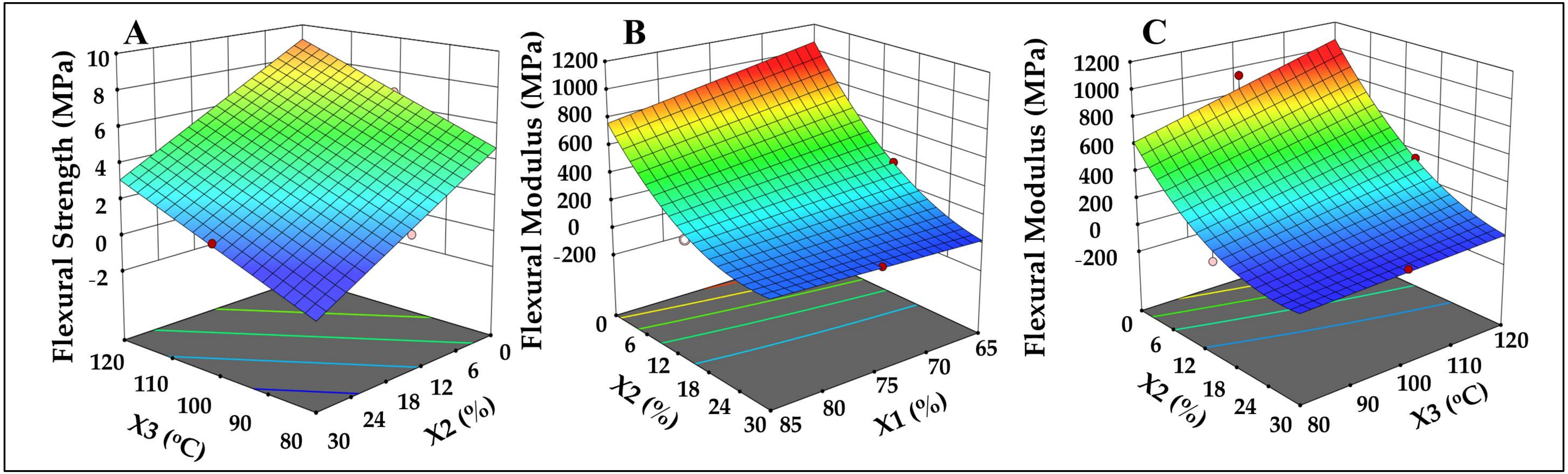
Production | Optimization



Response surface 3D plots for BBD:

(A) glycerol (X2) and temperature (X3) for Flexural Strength (MPa)

(B) chestnut (X1) against glycerol (X2) for Flexural Modulus (MPa)

(C) glycerol (X2) against temperature (X3) for Flexural Modulus (MPa).

Silva, S.B.; Freitas, O.M.; Vieira, E.F.; Gomes, A.; Carreiras, A.R.; Moreira, D.C.; Esfandiari, P.; Silva, J.F.; Delerue-Matos, C.; Domingues, V.F. Polymers 2025, 17, 616.

Production | Optimization

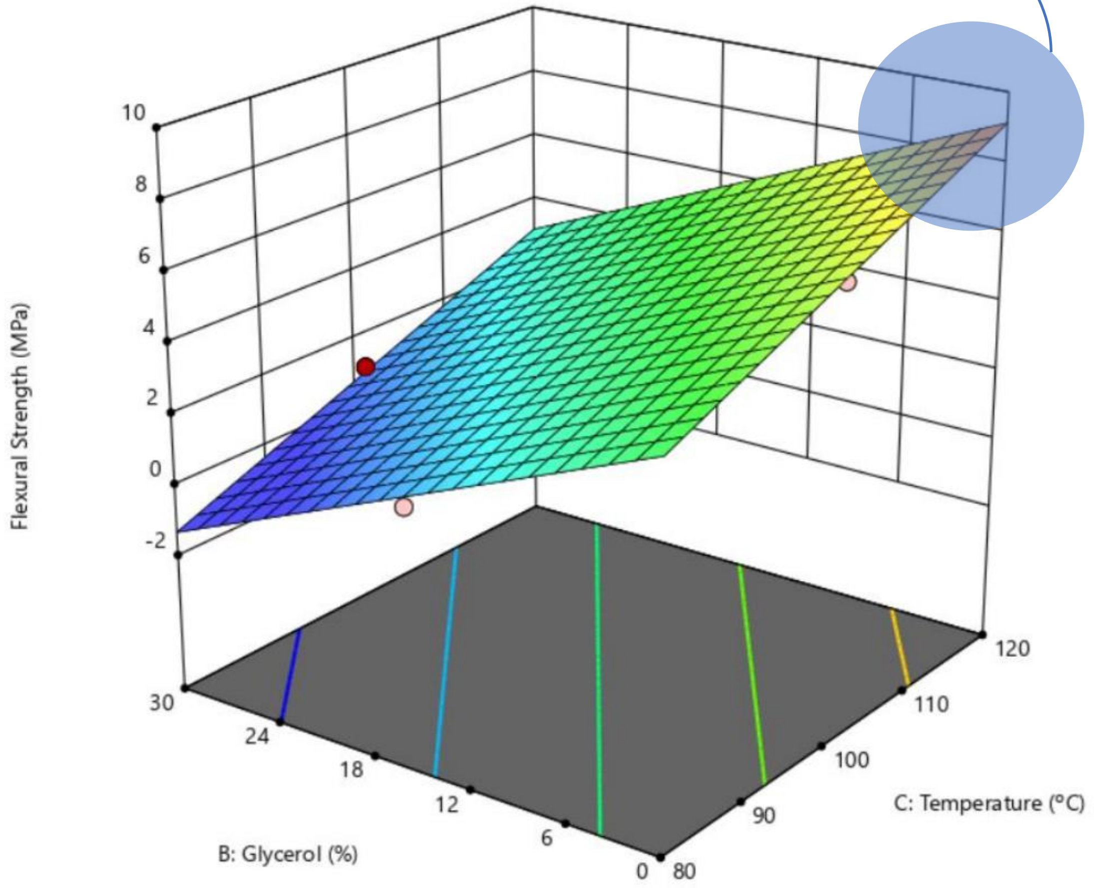
ANOVA for Modified Linear Model of Flexural Strength

Source	Sum of Squares	Mean Square	F value	*p* value
Model	112.03	56.01	43.59	< 0.0001*
X2-Glycerol	72.60	72.60	56.49	< 0.0001*
X3-Temperature	39.43	39.43	30.68	< 0.0001*
Residual	17.99	1.29		
Lack of Fit	12.84	1.28	1.00	0,55
Pure Error	5.15	1.29		
Total	130.02			

* $p < 0.05$.

$$Flexural\ Strength = 3.90 - 3.01\,X2 + 2.22\,X3$$

High temperature
Low glycerol concentration



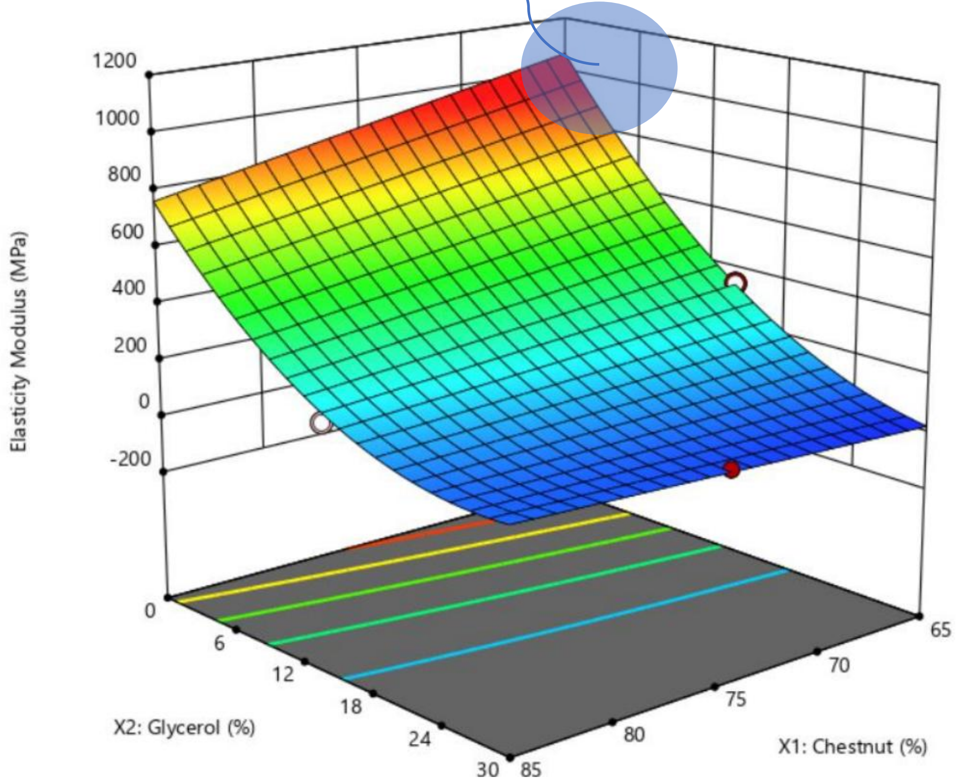
Production | Optimization

ANOVA for Modified Linear Model of elastic modulus

Source	Sum of Squares	Mean Square	F-value	*p* value
Model	1.27×10^6	2.12×10^5	41.47	< 0.0001*
X1-Chestnut	27976.77	27976.77	5.47	0.0414*
X2-Glycerol	8.79×10^5	8.79×10^5	171.91	< 0.0001*
X3-Temperature	1.23×10^5	1.23×10^5	24.05	0.0006*
X1X2	33432.29	33432.29	6.54	0.0285*
X2X3	38911.51	38911.51	7.61	0.0202*
$X2^2$	1.70×10^5	1.70×10^5	33.25	0.0002*
Residual	51127.14	5112.71		
Lack of Fit	35786.32	5964.39	1.56	0.3484
Pure Error	15340.82	3835.2		
Total	1.32×10^6			

*$p < 0.05$.

Low concentration of chestuts
Low concentration of glycerol



$$Elastic\ Modulus = 160.26 - 59.14\,X1 - 331.46\,X2 + 123.97\,X3 + 91.42\,X1X2 - 98.63\,X2X3 + 200.34\,X2^2 \quad (3)$$

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Production | Optimization

Flexural Strength

75% chestnut
0% glycerol
120 °C

Elastic modulus

67% chestnut
0% glycerol
118 °C

Optimal Composition

70% chestnut
0% glycerol
120 °C

Natural resin

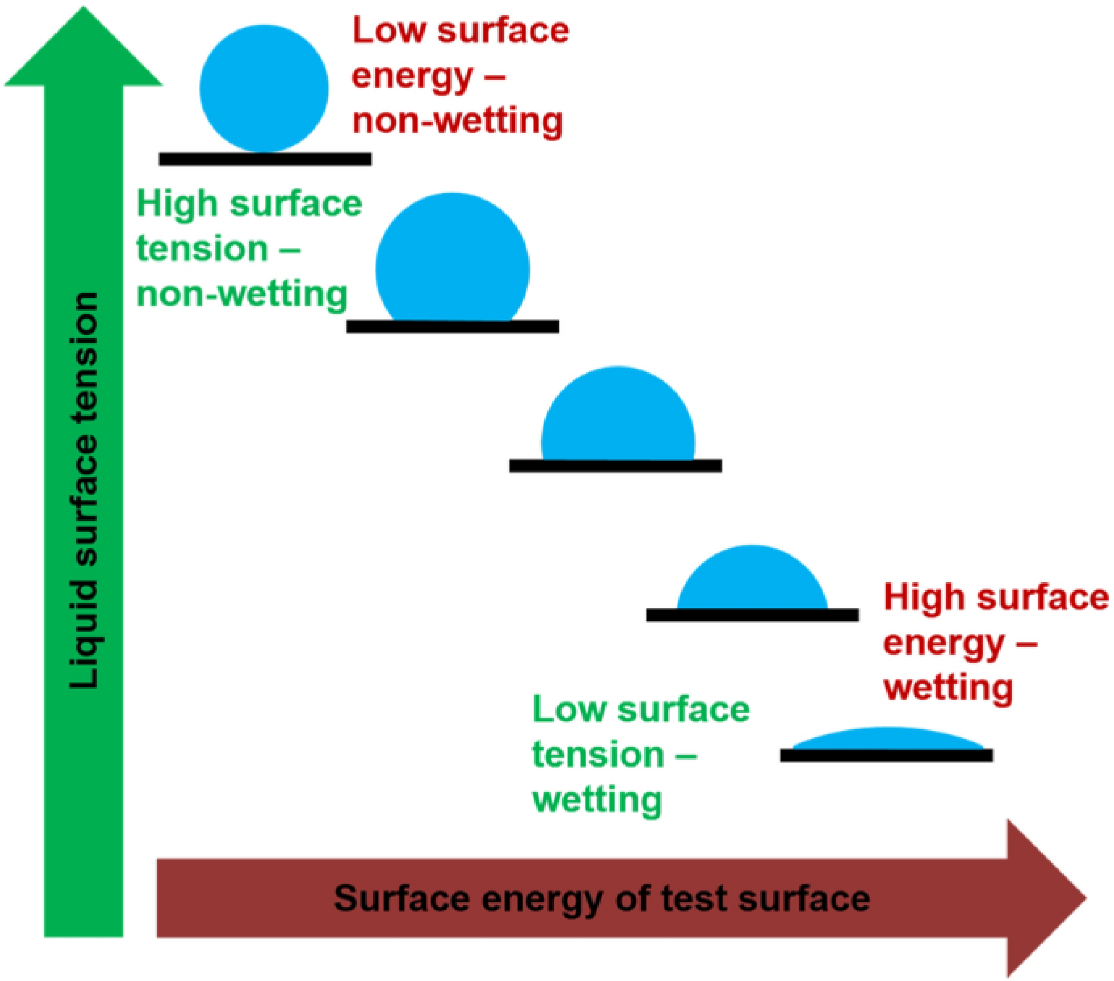
Shellac is obtained from tiny insects from Kerria species, feeding on the sap of specific host trees.

In its basic form, **Shellac** is a polyester macromolecule composed of esters of polyhydroxy aliphatic and sesquiterpene acids.

Shellac is a GRAS (Generally Recognized as Safe) natural polymer.

Characterization | Wettability

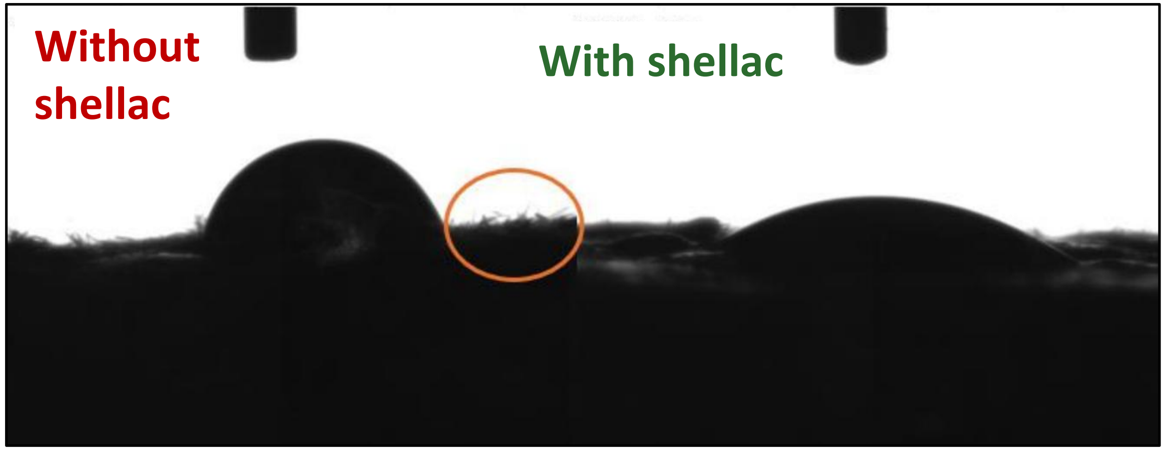
Wettability is the ability of a liquid to maintain contact with a solid surface, and it is controlled by the balance between the intermolecular interactions of adhesive (liquid to surface) and cohesive (liquid to liquid) type interfaces.



Jothi Prakash, C.G., Prasanth, R. Approaches to design a surface with tunable wettability: a review on surface properties. J Mater Sci (2021) 56, 108–135.

Characterization | Wettability

72 ± 14°



43 ± 6°

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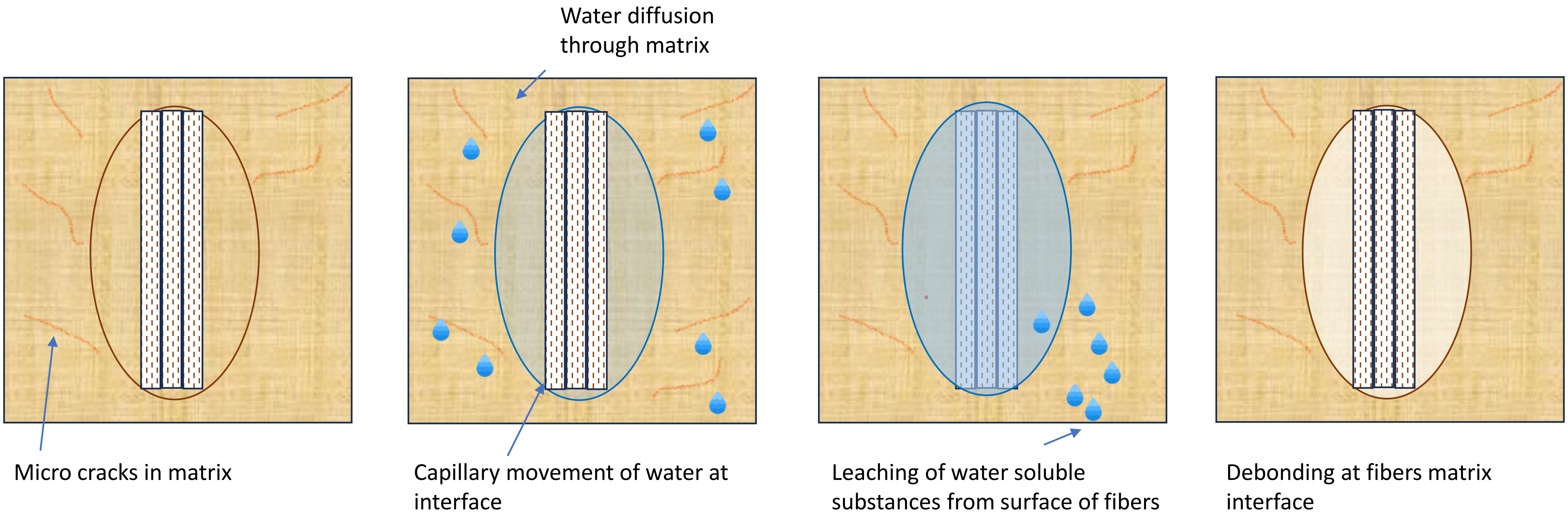
Characterization | Water absorption



Characterization | Water absorption

Water absorption essay of composites with:

- Only chestnut
- Chestnut with glycerol
- Chestnut with shellac (natural hydrophobic coating)

$$Increase\ in\ weight\ (\%) = \frac{Wet\ weight - Conditioned\ weight}{Conditioned\ weight} \times 100$$

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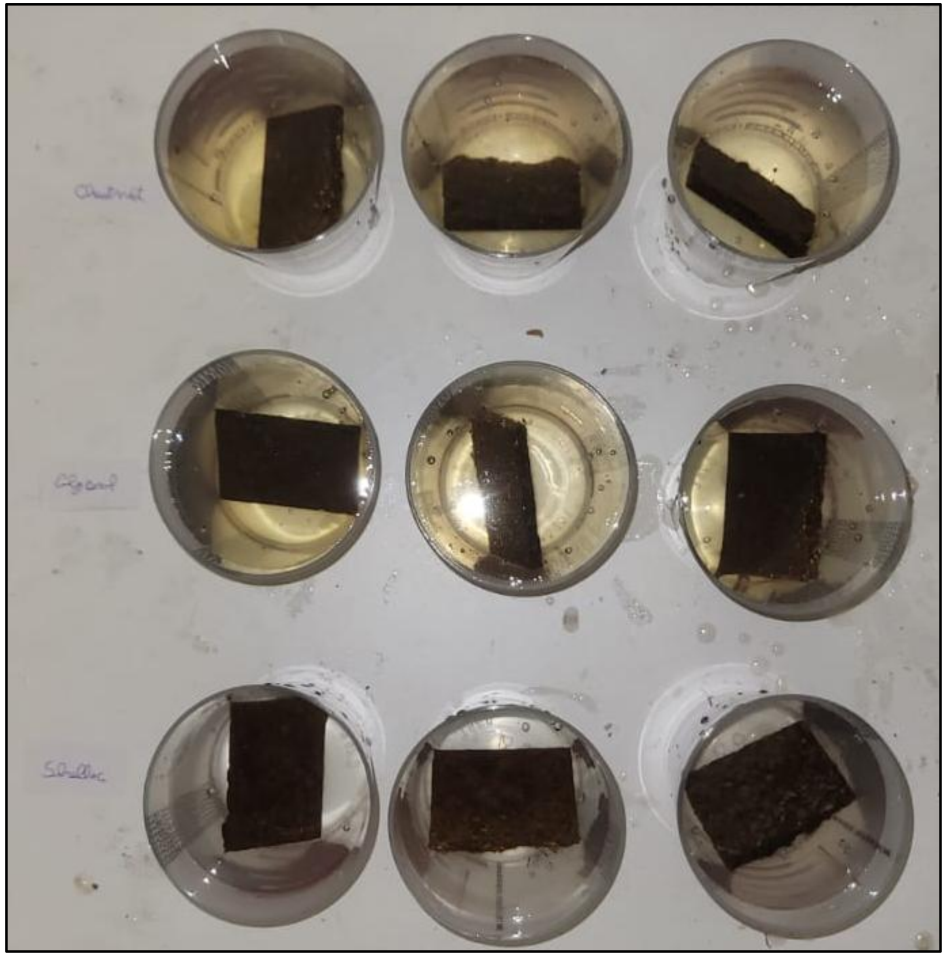

ASTM D570 (2022)

Characterization | Water absorption

	Water absorption (%)		
Time (h)	**2**	**24**	**96**
Only chestnut	10	45	60
Chestnut with glycerol	12	52	65
Biocomposite with shellac	4	18	30

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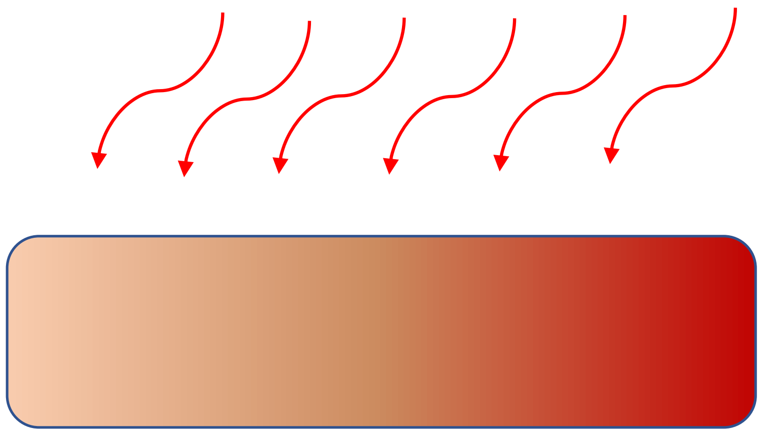
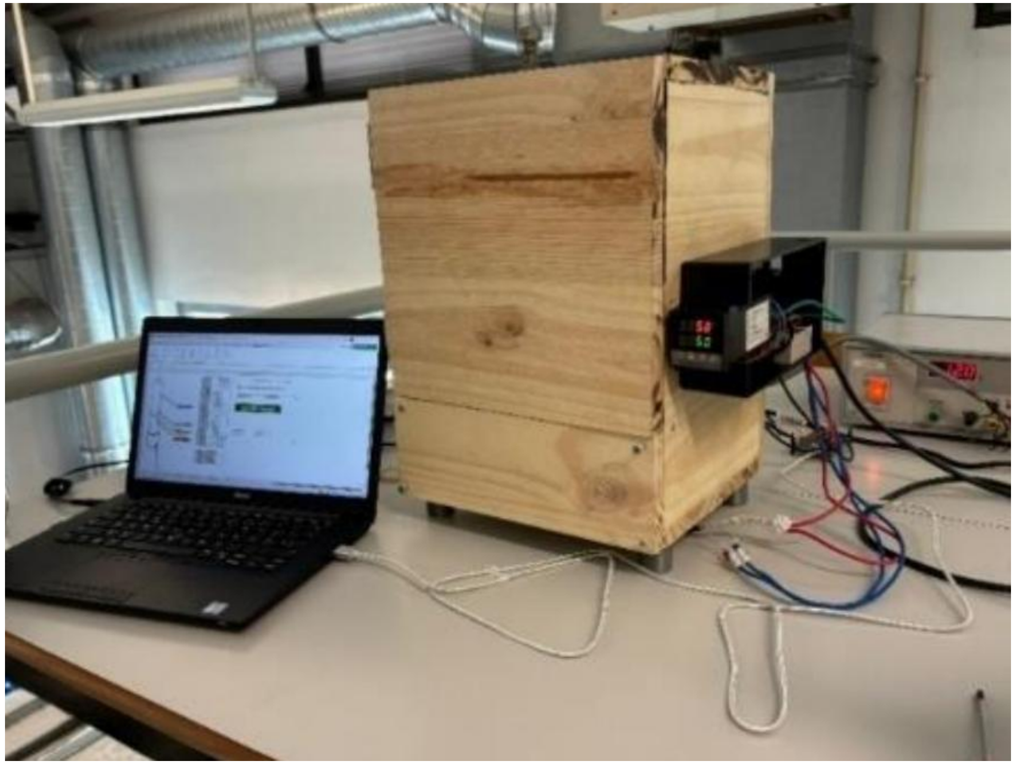
Characterization | Thermal condutivity



Factors influencing **thermal conductivity** include:

- material's density
- moisture content
- type and orientation of reinforcing fibers

Characterization | Thermal condutivity



- Temperature evolution measurement
- 8 mm thick

The chestnut-based composite exhibited a thermal conductivity of 0.79 W/m·K after 53 min at 48 °C, categorizing it as a **thermal insulator**.

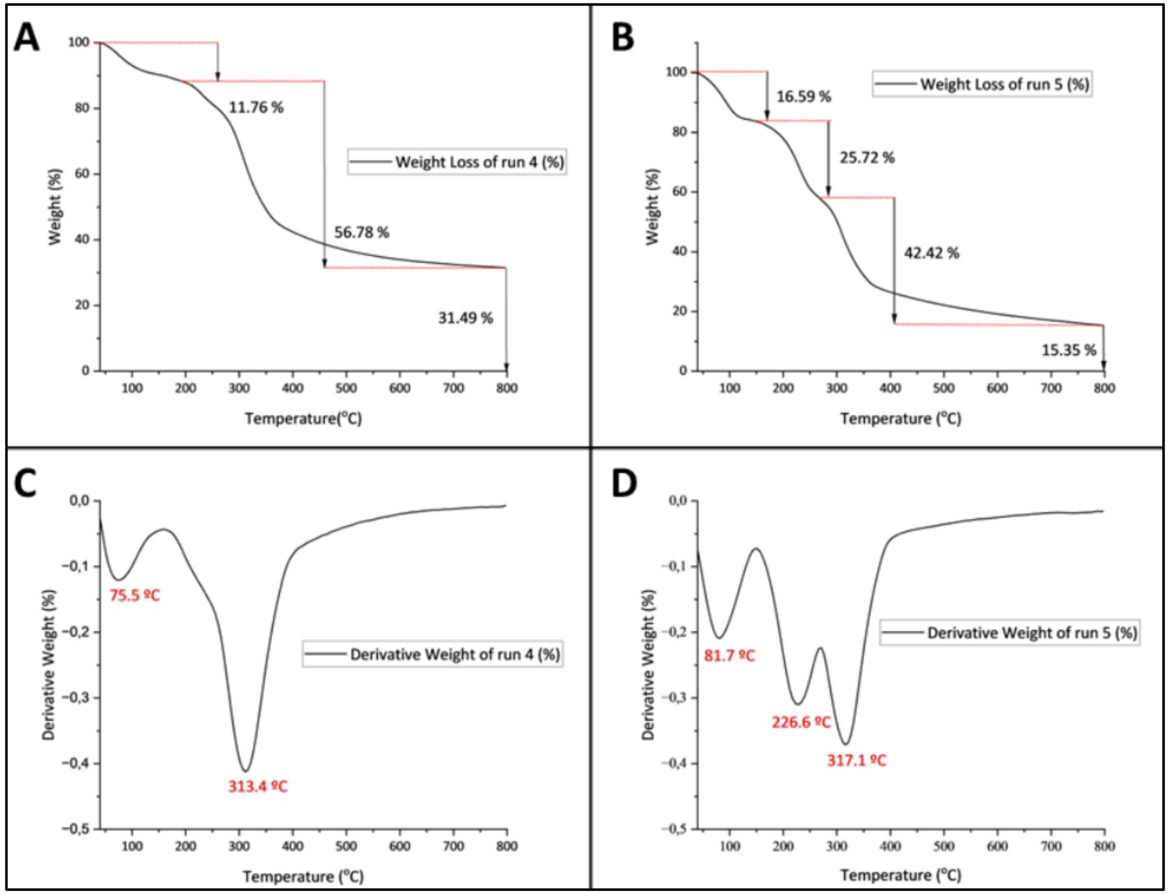
Silva, S.B.; Freitas, O.M.; Vieira, E.F.; Gomes, A.; Carreiras, A.R.; Moreira, D.C.; Esfandiari, P.; Silva, J.F.; Delerue-Matos, C.; Domingues, V.F. *Polymers* **2025**, *17*, 616.

Characterization | TG/DSC

Thermogravimetry (TG)
Differential Scanning Calorimetry (DSC)

Experimental conditions:

- ✓ 10 mg of composite
- ✓ **TGA:** 25 to 800°C, 20°C/min
- ✓ **DSC:** 25 to 350°C, 2°C/min
- ✓ Glass transition was measured by the tangent graphical method.
- ✓ Effect of glycerol



(A) and **(B)** TGA curves **(C)** and **(D)** DTG curves of two composites: without glycerol and with glycerol

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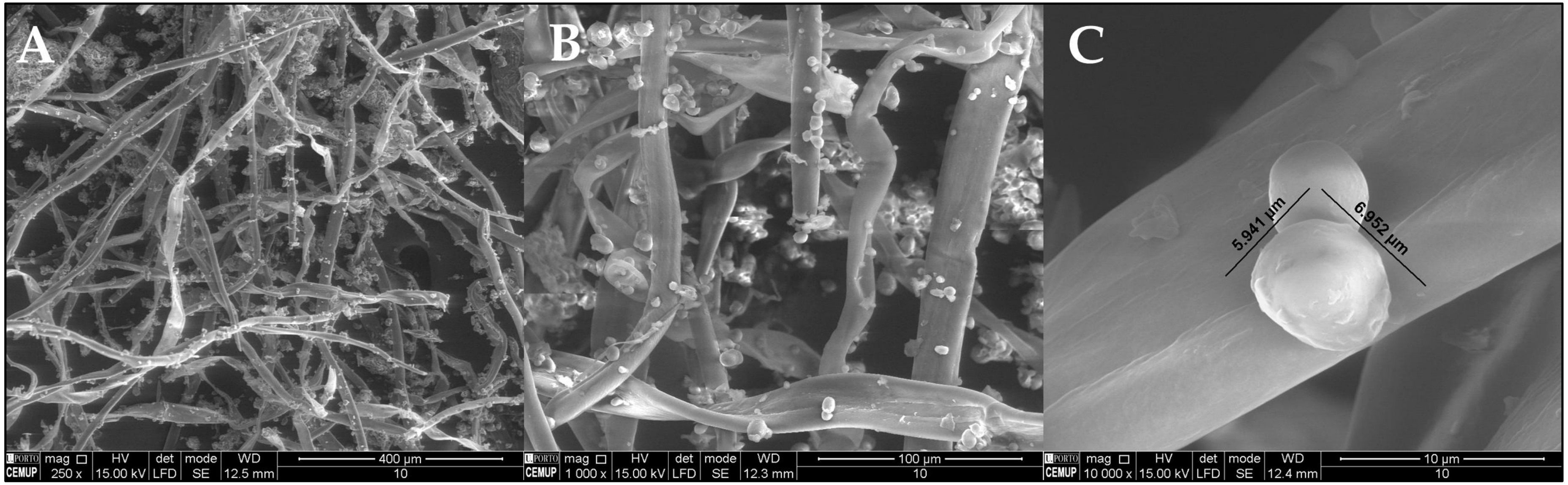
Characterization | SEM

Scanning Electron Microscopy



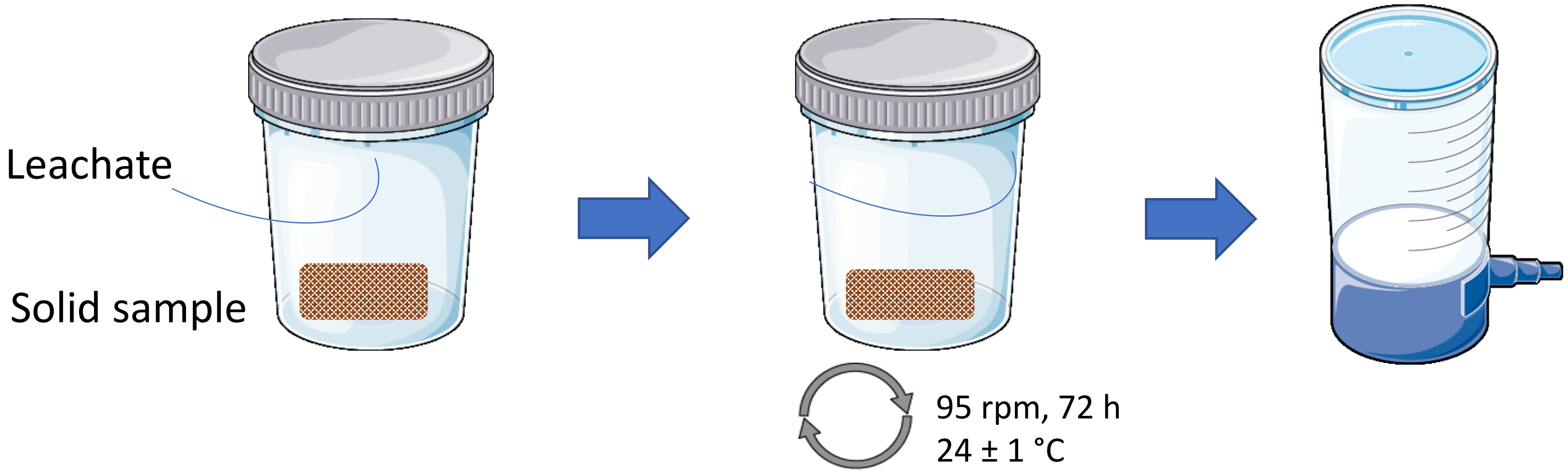
(A) 400 µm, (B) 100 µm (C) 10 µm.

Silva, S.B.; Freitas, O.M.; Vieira, E.F.; Gomes, A.; Carreiras, A.R.; Moreira, D.C.; Esfandiari, P.; Silva, J.F.; Delerue-Matos, C.; Domingues, V.F. *Polymers* **2025**, *17*, 616.

Phytotoxicity

SWEDISH STANDARD · SS-EN 12457-2

Characterization of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 2: One stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 4 mm (without or with size reduction)



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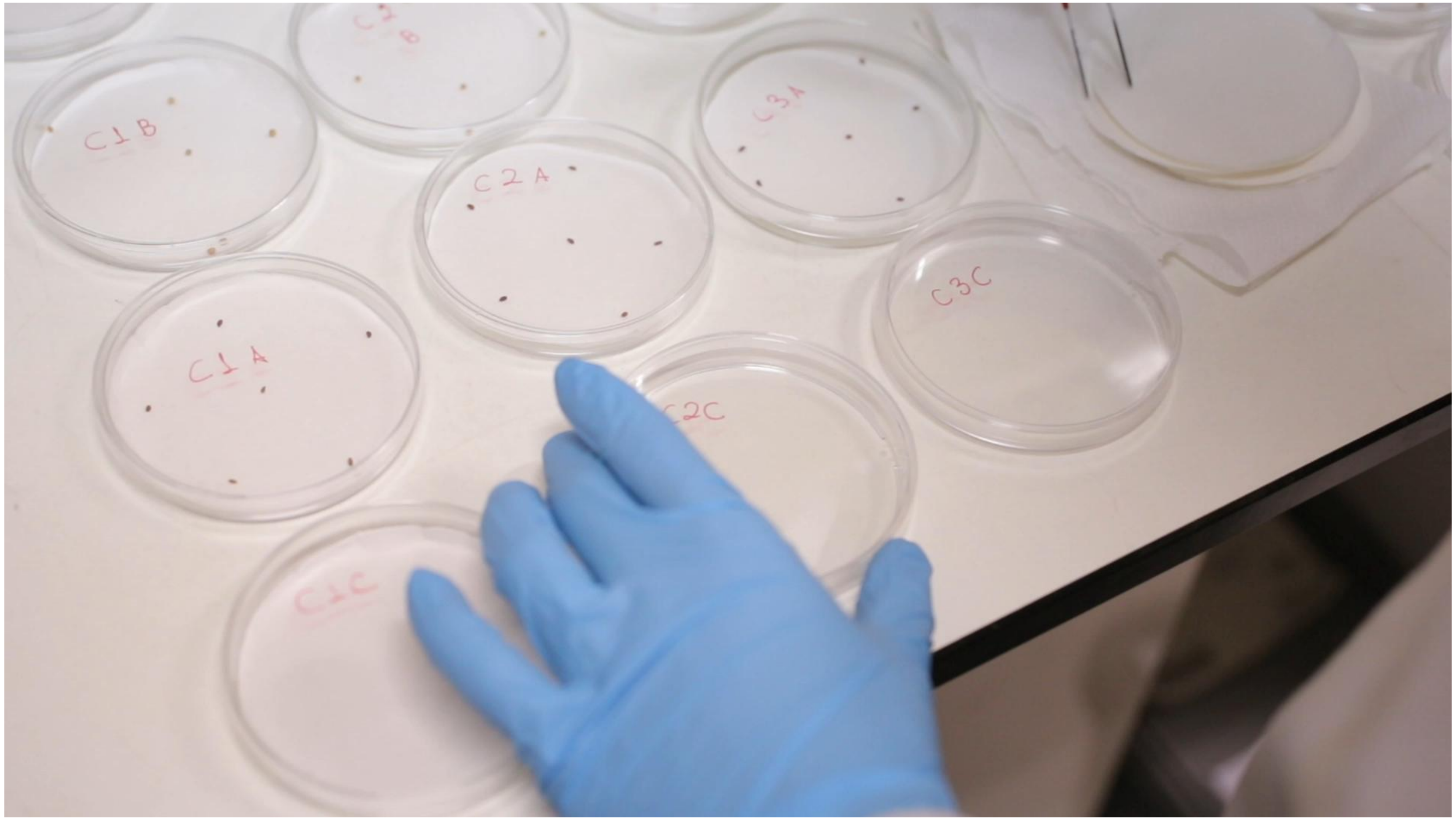
Phytotoxicity



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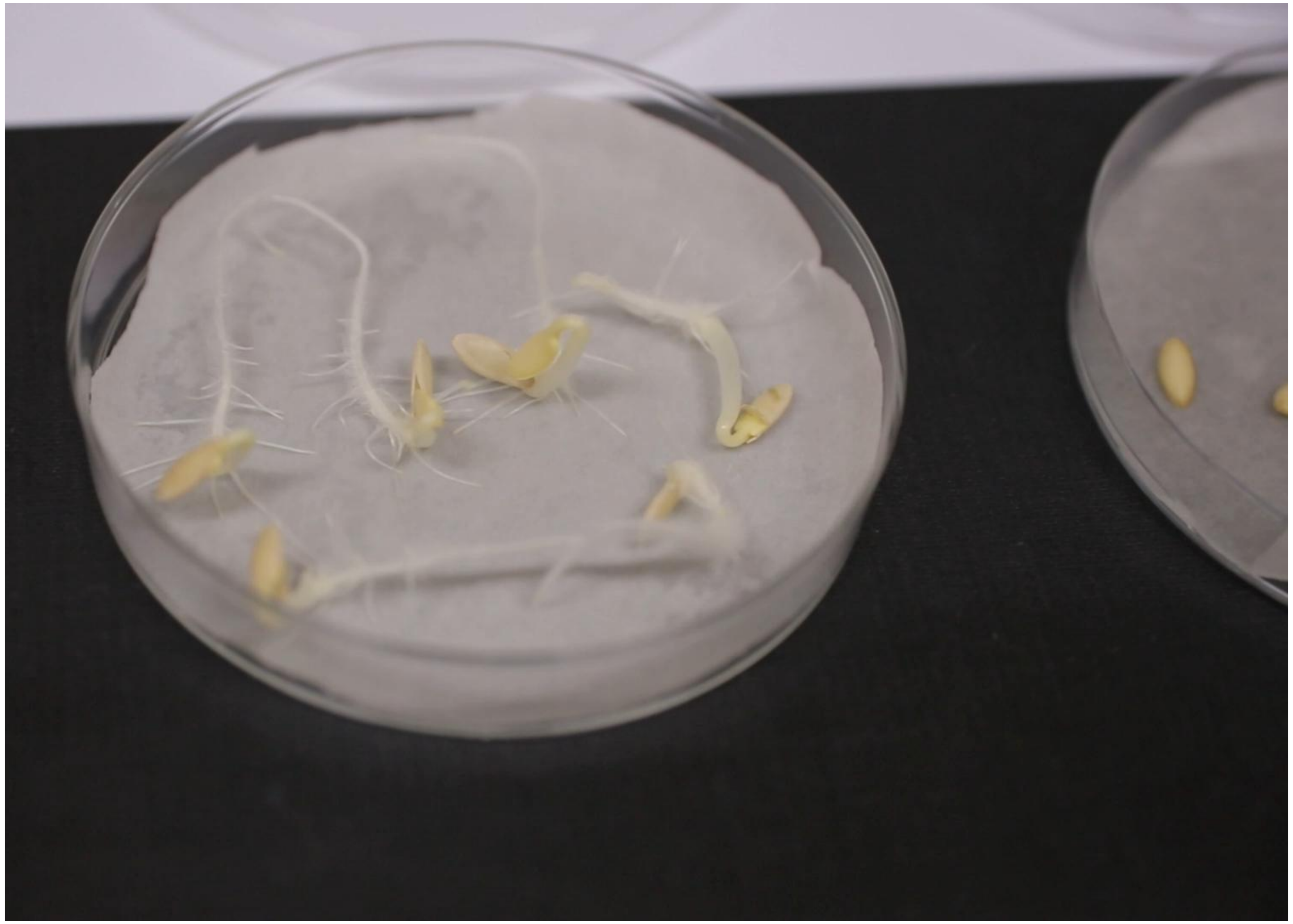
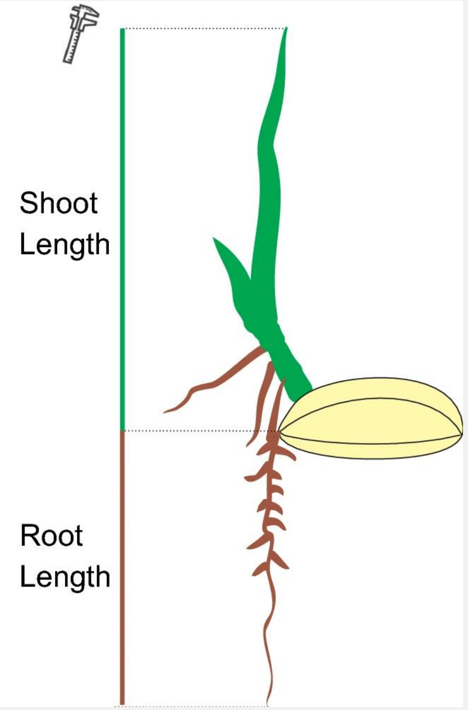
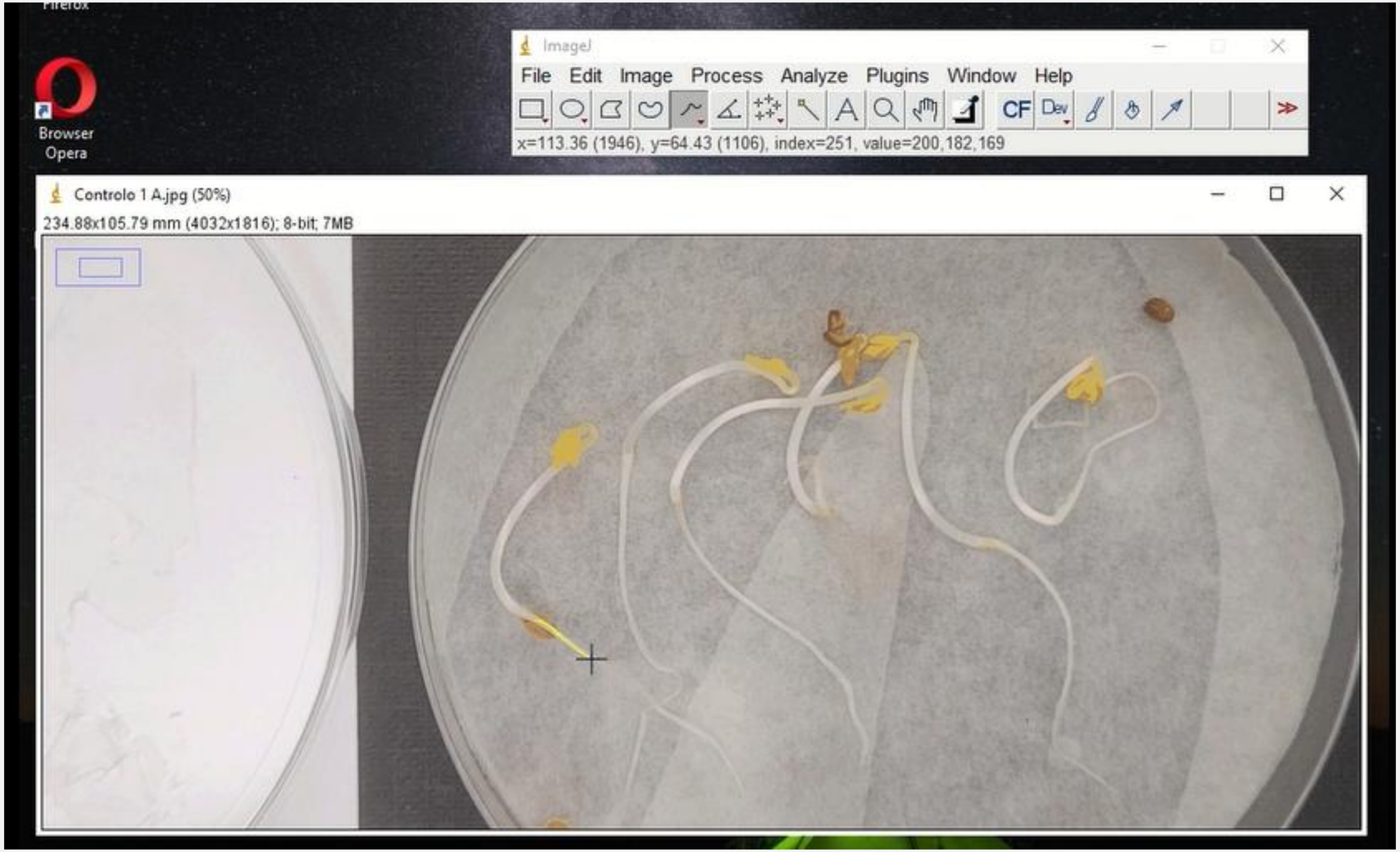
Phytotoxicity



Phytotoxicity

Root elongation
Shoot length





Application

Hydraulic press developed to produce a chestnut-based candle holder

And light was made

Silva, S.B.; Freitas, O.M.; Vieira, E.F.; Gomes, A.; Carreiras, A.R.; Moreira, D.C.; Esfandiari, P.; Silva, J.F.; Delerue-Matos, C.; Domingues, V.F. *Polymers* **2025**, *17*, 616.

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