

Autex 2018 Conference

INCORPORATION OF TANNINS IN POLYPROPYLENE BY MELTING PROCESS FOR ANTIBACTERIAL AND SUSTAINABLE TEXTILES

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gemtex
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With the support of the European fund for regional development

Context

Increase of textile functionality



Antimicrobial Textile

- Durable
- Odor
- Hygienic

Medical

Clothing

Building



Silver particles



Environmental concerns

REACH regulation



*



Replacement of silver particles by sustainable fillers

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CETI PRODUCT INSIGHT
SOLUTIONS FOR TEXTILE SOLUTIONS

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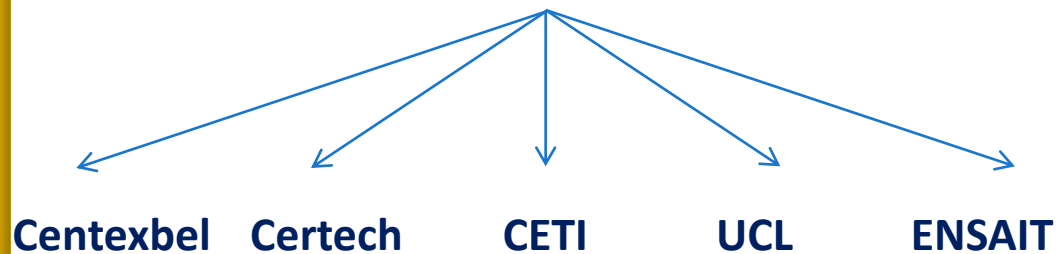
UCL
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de Louvain

Context of Duratex project

Interreg Project

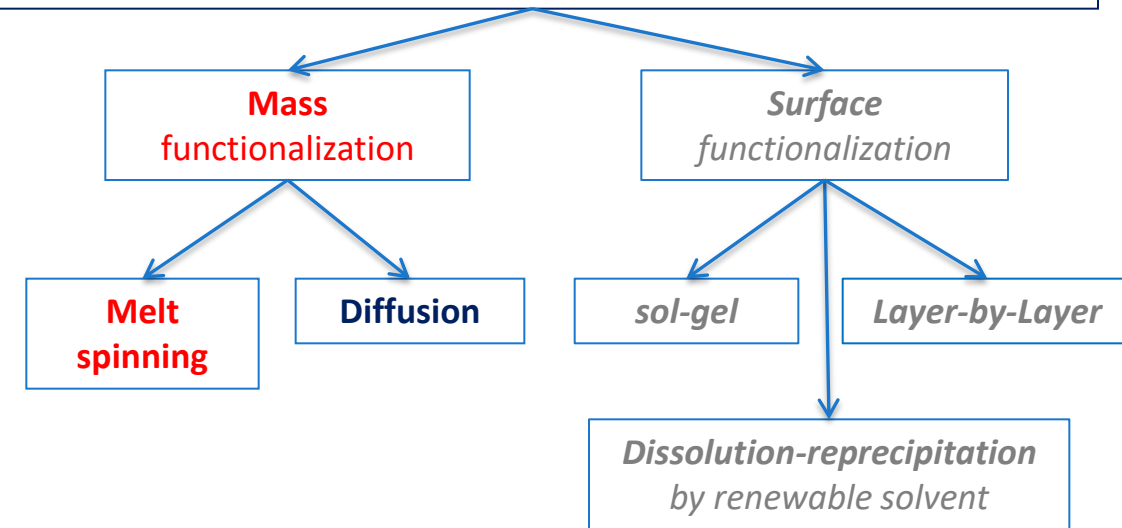

 GoToS3
DURATEX

5 Partners





Antifouling and anti-microbial textiles in the field of construction and architecture



Eco-friendly

No fluorocarbon compounds
 No problematic biocides for the environment
 (ex: silver nanoparticles)

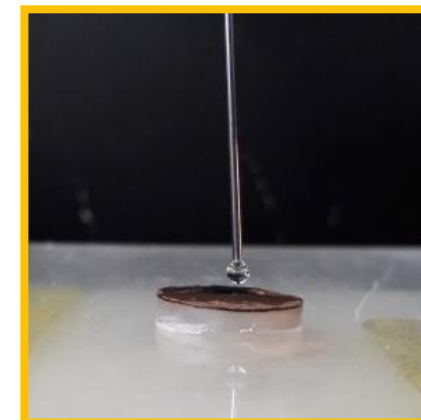
Sustainable

Improvement of the sustainability of treatments made with respect to environmental factors

Objective: To develop anti-microbial textiles with biobased and eco-friendly fillers in filament by melt spinning

Plan

- Context
- Biocides choice : Tannins
- Melting process
- Results and discussion
 - Interface properties
 - Mechanical properties
 - Thermal properties
 - Antibacterial properties
- Conclusions and perspectives



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Biocides choice

Significants parameters

- Degradation temperature (210°C)
 - Good dispersion in the PP
- Resistant to climatic conditions and UV radiations
 - Efficient in small amount

Unstable at high temperature

- Chitosan
- PHMB (polybiguanide)

Environmentally harmful

- Triclosan
- Titanium dioxide
- Zinc oxide
- Copper oxide

Choice

✓ Tannic acid

What is tannin ?

- Contained in some beverages

- Tea
- Coffee
- Wine

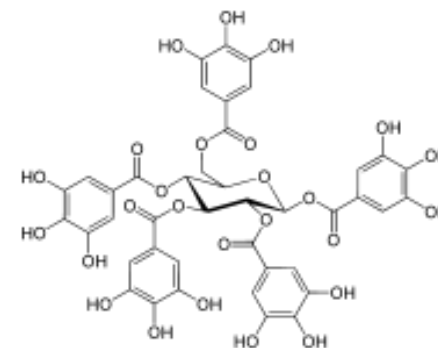


- Occurred in large quantity in

- trees
- leaves
- fruits



→ Vegetable origin



- Complex phenolic compounds
- Extreme diversity : Variable chemical structure

Biocides choice : Tannins used

Three kinds of commercial tannin tested

- 100% natural material
- Application : Brewery and food
 - From Ajinomoto OmniChem

> TANAL 01 : TA01

> BREWTAN F : BT

> TANAL 04 : TA04

	TA01	BT	TA04
pH (1% in water)	3.0 – 4.0	3.4 - 4.5	2.5 – 3.5
molecular weight	high	high	low
purity	> 98%	> 98%	>96%
Gallic acid percentage	<1%	<0.1%	

Thermal stability : TGA

From 20°C to 700°C

10 °C/min

Under air

→ $T_d(^{\circ}\text{C})$: 5 wt.% of loss weight

Fillers	TA01	BT	TA04
$T_d(^{\circ}\text{C})$	252.7	244.3	258.0



No significant weight loss at PP processing temperature (210°C)

Antibacterial Tests

■ Qualitative test : Agar Diffusion Test

■ Bacteria :

■ Staphylococcus Epidermidis

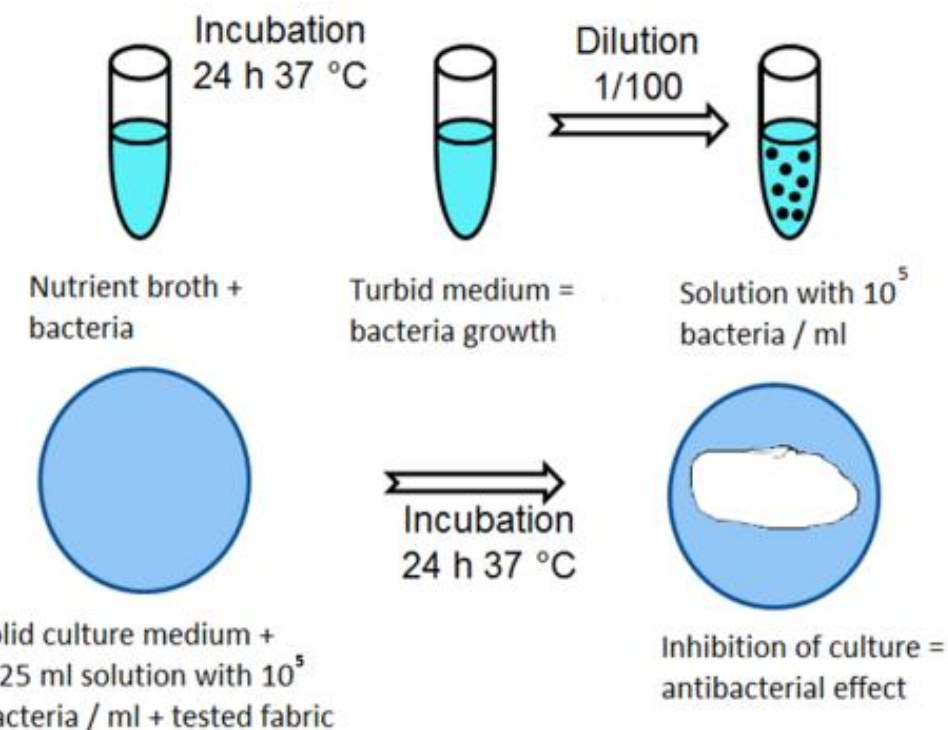


Present in large amounts on the human skin constantly in contact with clothing textiles

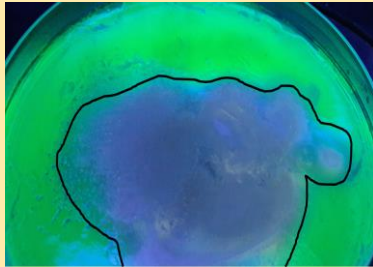
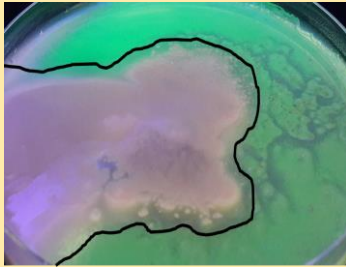
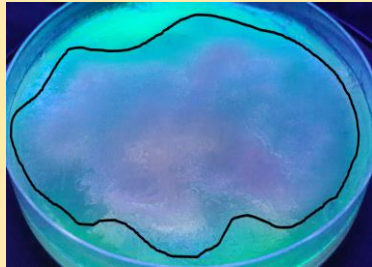
■ Escherichia Coli



Present mainly in the intestines of mammals



Antibacterial activity of tannin powder

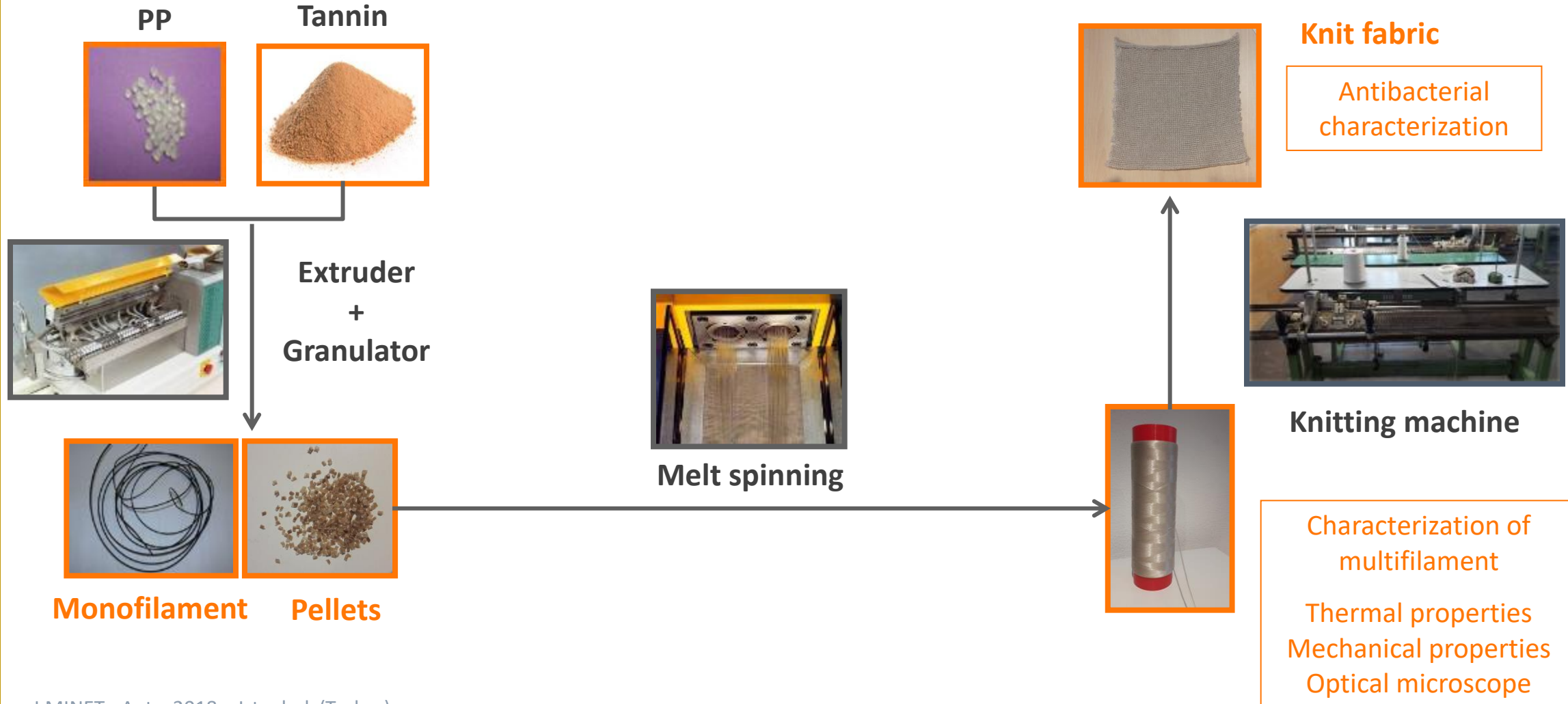
Powder	TA01	BT	TA04
Gram-positive bacteria <i>S.Epidermidis</i>			
Gram-negative bacteria <i>E.Coli</i>			

3 min → 200°C

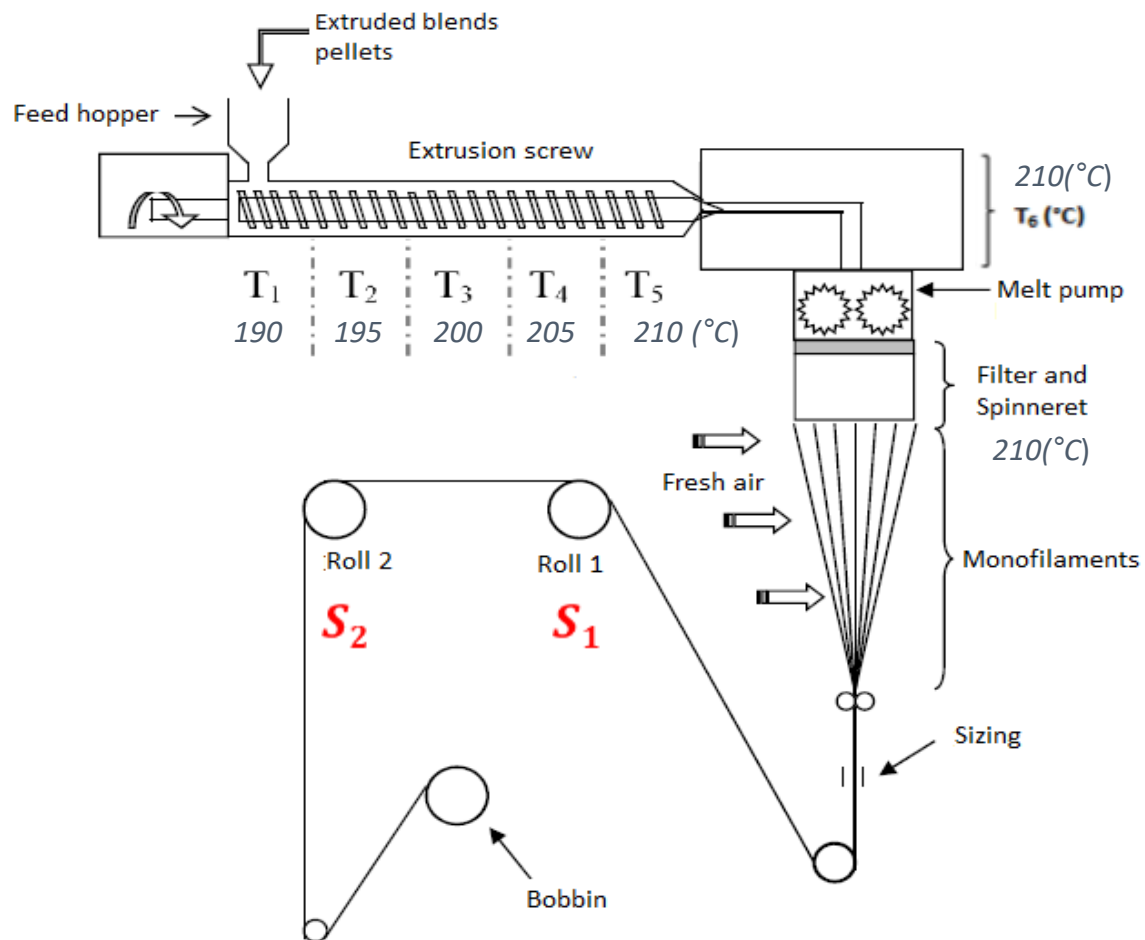
2 min → 210°C

After thermal treatment: same inhibition zone against bacteria

Melt process



Process : Melt spinning



Machine : Spinboy I

- Single screw extruder
- Pump speed: 15 rpm

Draw ratio:

$$DR = \frac{S_2}{S_1}$$

PP + 2 wt.% Tannin		
<i>m/min</i>	S1	S2
DR = 2	150	300
DR = 3	150	450

Thermal properties

DSC (multifilaments)

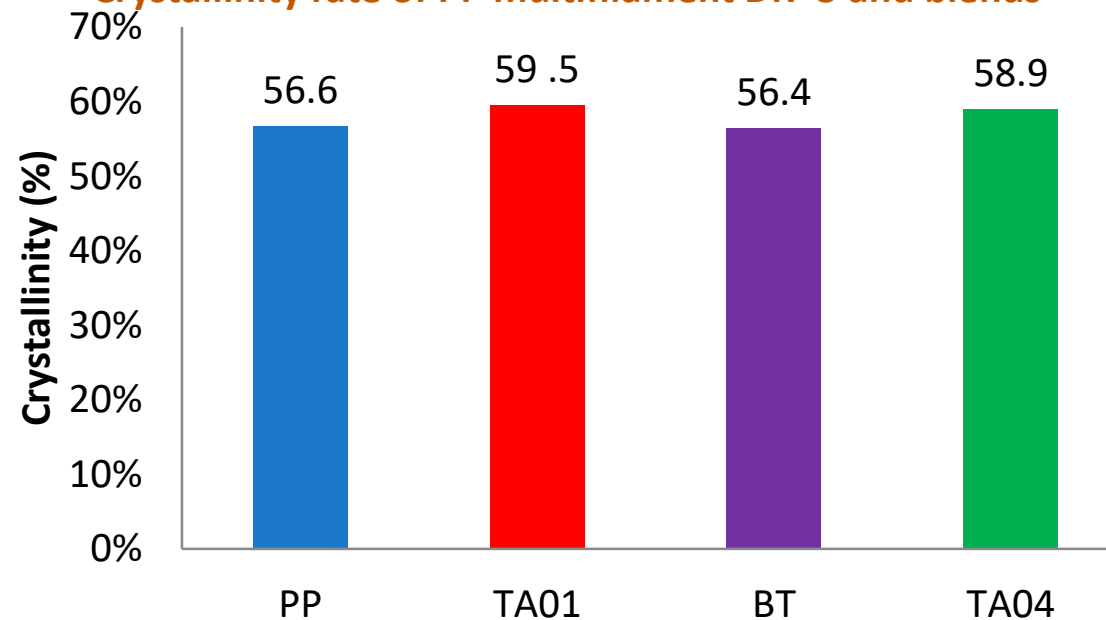
2 cycles 20°C → 200°C ±10°C/min (N₂)

DR=3	Virgin PP	TA01	BT	TA04
T _m (C°)	162	164	163	163
T _c (C°)	118	118	117	116

$$\chi(\%) = \frac{\Delta H_m}{(1 - m_{filler}) * \Delta H_m^0}$$

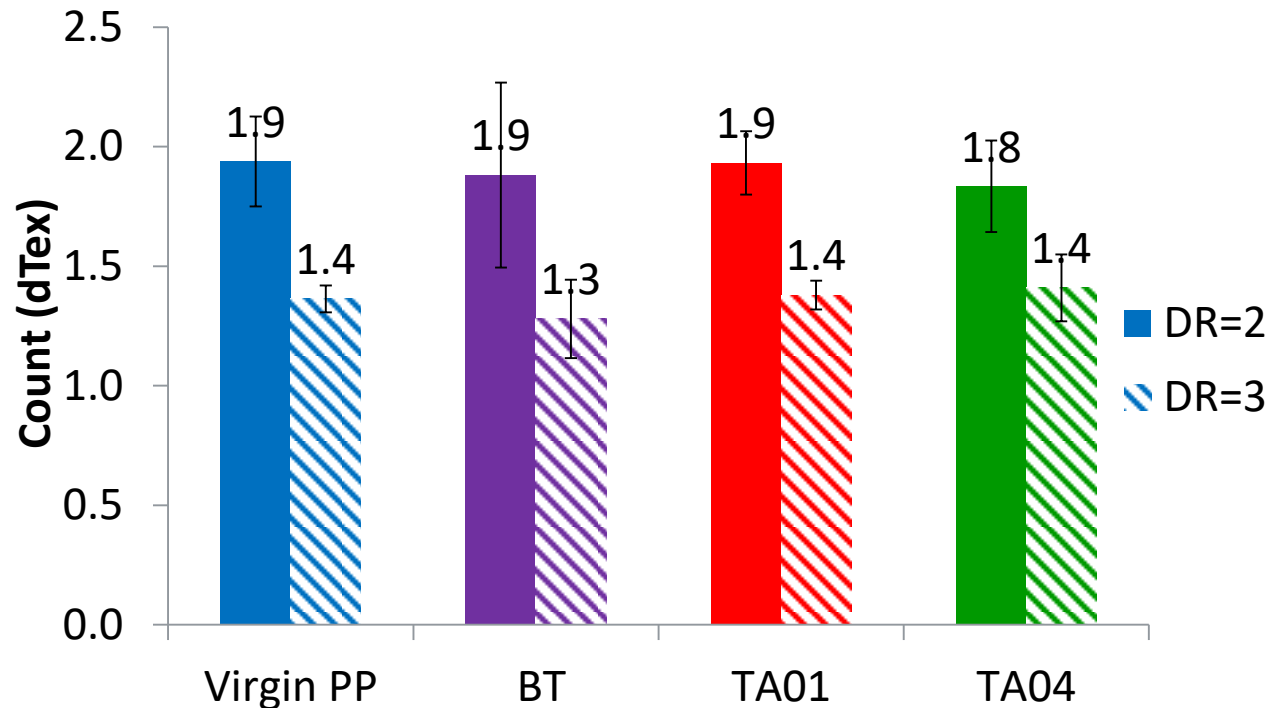


Crystallinity rate of PP multifilament DR=3 and blends



- Introduction of **BT**, **TA04**, and **TA01** into **PP** have no significant impact on melting and crystallinity temperatures
- Slight increase of the crystallinity with **TA04** and especially with **TA01**

Mechanical properties



No significant modification of the monofilaments count

Monofilament count:

- Vibroskop LENZING INSTRUMENTS
- 10 samples

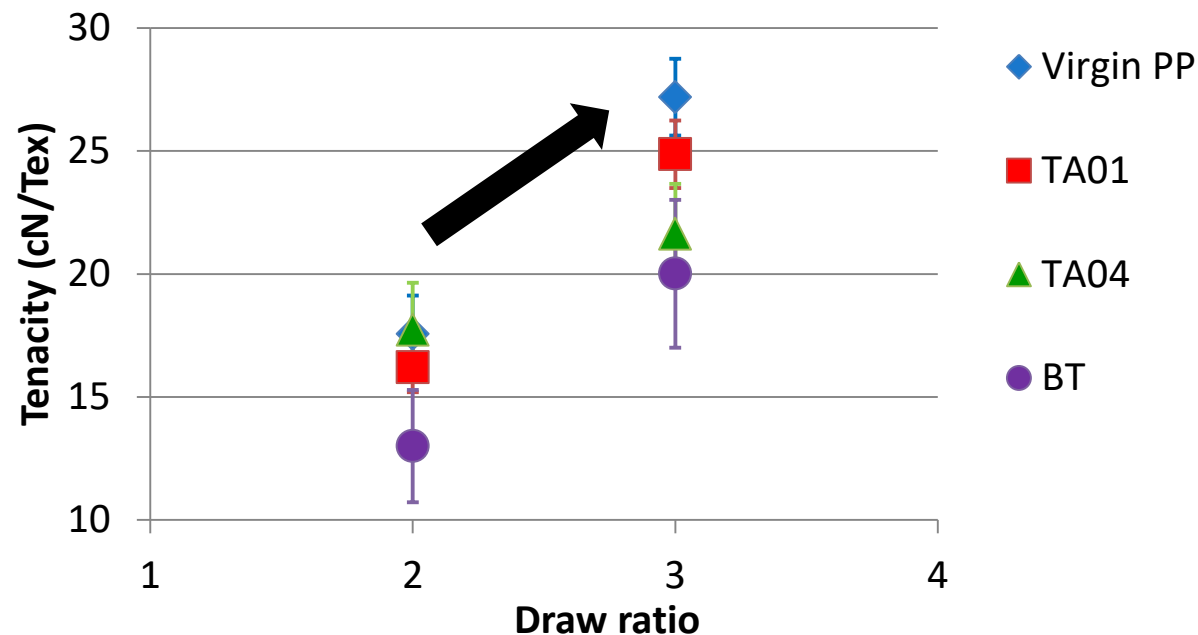
Traction test

- Zwick machine
- Standard NF EN ISO 5079
- Sample size : 20 mm
- Deformation rate : 20 mm/min

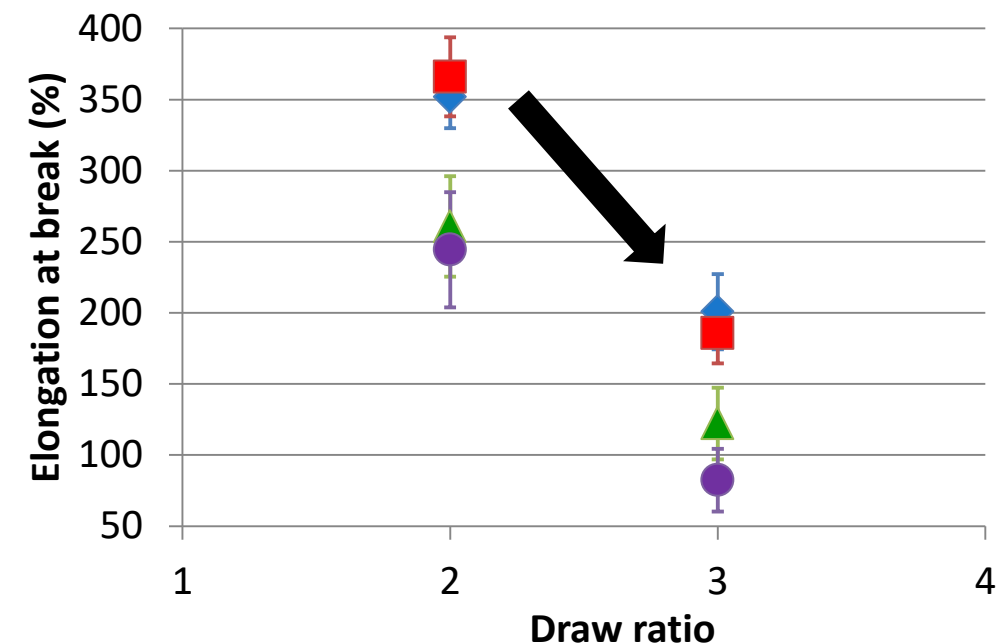


Mechanical properties

Tenacity at break for monofilaments of PP and blends



Elongation at break for monofilaments of PP and blends



- When DR = 2 the material is less resistant than DR=3 but less elongation at break
- Incorporation of **TA01** into **PP** has less impact on mechanical properties than the other fillers **BT** and **TA04**
- **BT** reduces markedly mechanical properties of **PP** and shows the highest standard deviation

Interface properties

Machine : DigiDrop

(mN/m)	Virgin PP	TA01	BT	TA04
γ_s	30.1	60.0	58.0	58.3
γ_s^p	1.9	33.4	28.8	32.8
γ_s^d	28.2	26.7	29.1	25.5

$$\gamma_{x/PET} = \gamma_x + \gamma_{PET} - \frac{4\gamma_x^d \gamma_{PET}^d}{\gamma_x^d + \gamma_{PET}^d} - \frac{4\gamma_x^p \gamma_{PET}^p}{\gamma_x^p + \gamma_{PET}^p}$$

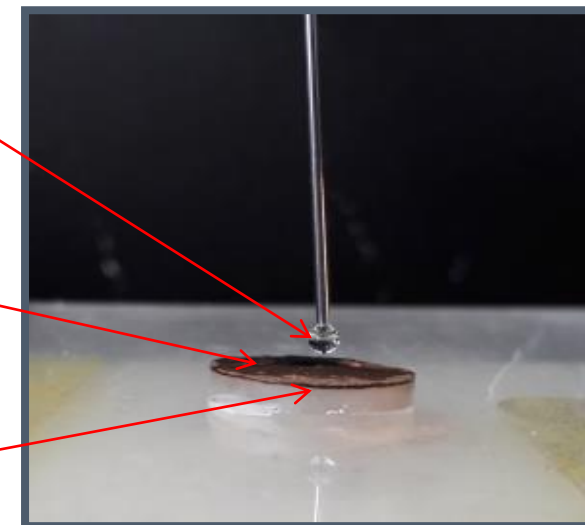
(mN/m)	$\gamma_{X/PET}$
$\gamma_{TA01/PP}$	28.2
$\gamma_{BT/PP}$	23.7
$\gamma_{TA04/PP}$	27.7

High interfacial energy

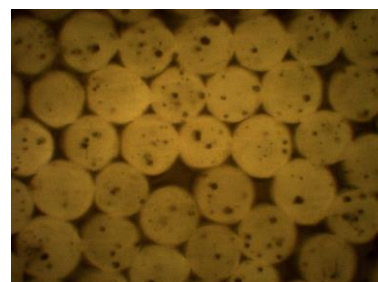
Drop of water

Tannin

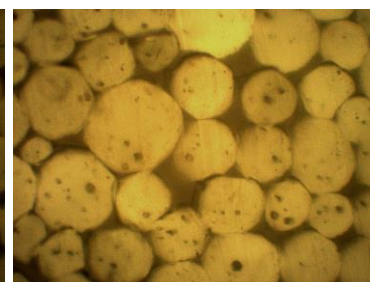
Paraffin



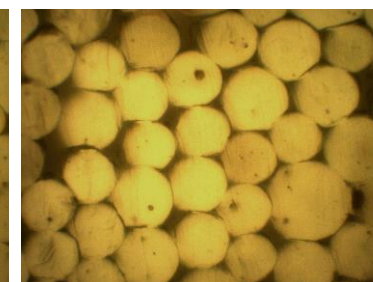
Optical microscope



PP + 2% TA01 E3



PP + 2% BT E3

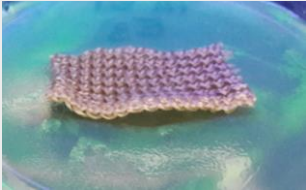


PP + 2% TA04 E3

Cross section:
Homogeneous
sections except for BT

Antibacterial activity

- Desizing : knit fabrics with Soxhlet
- Sterilization : 15-20 min at 120°C
- Qualitative test : Agar Diffusion Test

2 wt.% ; DR = 3	TA01	BT	TA04
Gram-positive bacteria Staphylococcus Epidermidis			
Gram-negative bacteria Escherichia Coli			

- No bacteria development below each knit fabrics
- Antibacterial properties by contact
- Results has to be confirmed : Knit fabrics will be tested with **transfer test** (ISO 20743)



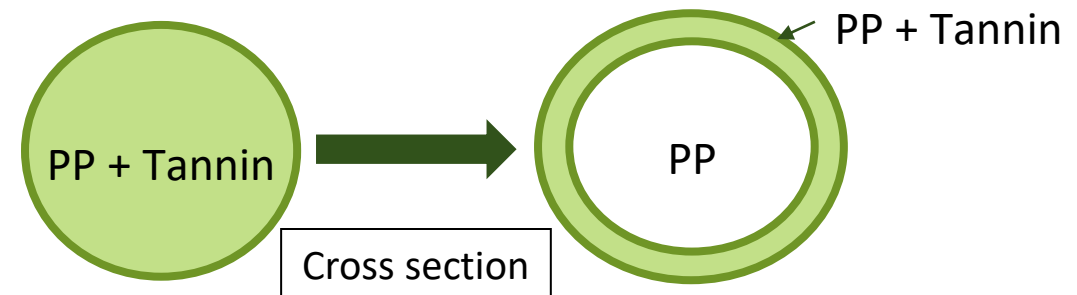
Conclusions & Perspectives

Conclusions:

- Tanal 04, Brewtan F and Tanal 01 have antibacterial activity alone and incorporated into PP
- Crystallinity increases slightly with the incorporation of Tanal 01 into the PP
- PP with 2wt.% Tanal 01 has the best mechanical properties compared to Brewtan F and Tanal 04

Perspectives :

- Testing the durability of anti-bacterial properties
 - > Washing (rain)
 - > Weather conditions (temperature, humidity, UV)
- Bi-composant



Reconnaissance / Erkenning

Projet soutenu par
Project ondersteund door

Interreg
France-Wallonie-Vlaanderen

Recherche et innovation
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Plus d'infos
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