



Emerging technologies to develop active packagings

September, 7th, 2017

Budapest, ActInPak Meeting

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Packaging is important in our society

- Way of life indicator:

- Europe: 116 kg/person/year
- USA: 200 kg/person/year
- Third World: 25% of food lost due to lack of packaging



- Packaging:

- 5 to 10 % of product value
- **Protects** 10 x its weight
- Represents 40% of Homewaste
- 1h/day less of Housekeeping thanks to packaging



SOCIETY IS CHANGING

. « From eating more quantity to eating best quality »

diversity, safety, functional food, nutrition

⇒ food tracability, tamper proof packaging

. *Social & economical evolution*

- ↗ level of life,

- **New marketing target** : children, old person

- ↗ **women's employment**: from 40% to 80% between 1965 & 1999,

- **New family** : mono-parental, single (1 foyer sur 3)

- **Less time for shopping**: from 1h30 in 1980 to 30 min en 2001

- **less time for dinner** : 1/3 of population eat « stand up » their lunch during week

⇒ Precooked dish, frozen food, fast food, higher shelf life food

⇒ New packaging systems are expected

Active & Intelligent Packaging



Standardized field [DeJong, 2005; DeKruif, 2002, Veermeiren, 1999]

- **Active Packaging**: **change packaging conditions** to increase shelf life & safety of food by keeping quality

3 types:

1. *Barrier & MAP*,
2. *Scavenger (O₂, humidity, ...)*,
3. *Anti-microbial*

- **Intelligent Packaging**: **monitor the quality** of the food product or its surrounding environment to predict or measure the safe shelf life better than a best before date

Ex: ITT, leakage Indicator, freshness indicator



TOXDTECT



SusFoFlex



BIOACTIVELAYER



acticospack

FLHEF



FRESHFILM

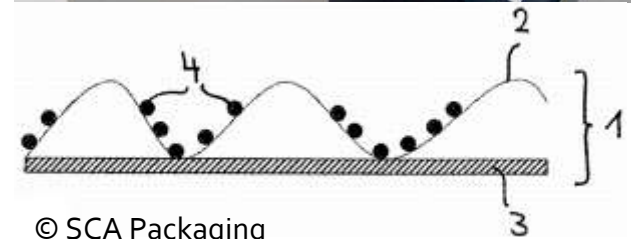
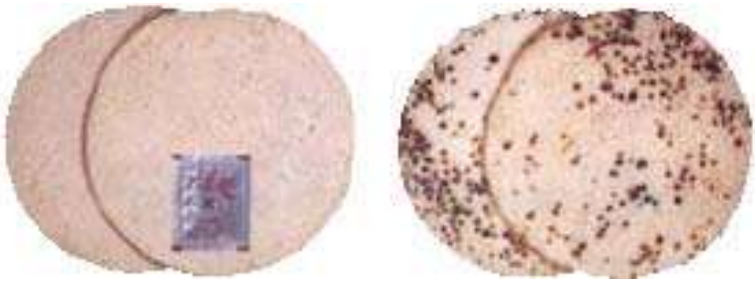
ECOBIOCAP



hortibiQpack



Introduction to active packaging



??

New active packaging systems = Need New Technologies

- ❑ **NEW SOLUTIONS IN MATERIALS**
 - ❑ Antimicrobial Peptides for Bioactive Packaging
 - ❑ Cellulose nanofibers (CNF) as a new opportunity

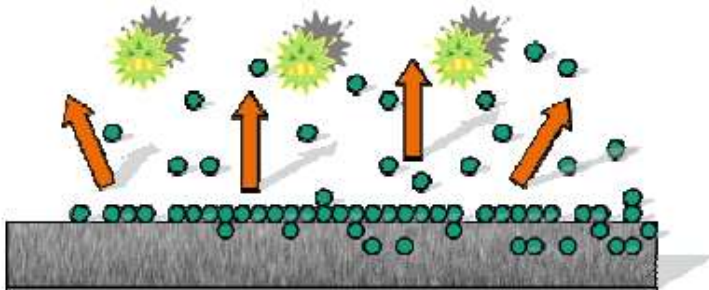
- ❑ **NEW SOLUTIONS IN PROCESSES**
 - ❑ Encapsulation systems in the packaging material
 - ❑ Electrospinning for active materials

- ❑ **NEW CONCEPTS IN ACTIVE PACKAGING**
 - ❑ Bionanocomposites for active packaging

Antimicrobial Surfaces and Coatings

Release-Mechanism

- Diffusion
- Decreasing effect over time

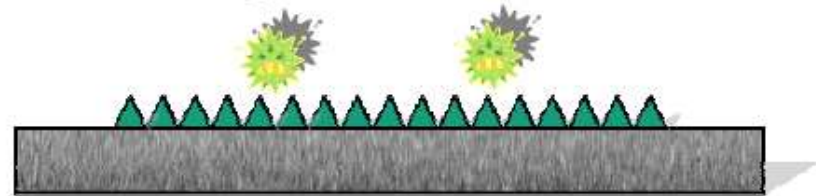


Examples:

- Triclosan (Textiles, Microban®)
- Enzymes (e.g. Lysozyme)
- Organic acids (e.g. sorbic acid)
- Bacteriocines (e.g. Nisin)

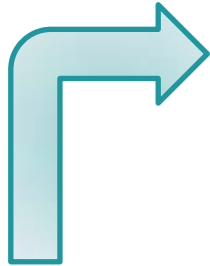
Contact-Mechanism

- Surface immobilized substances
- Antimicrobial effect by direct contact
- Deposits influences effect
- Structures (e.g. Lotus effect)



Examples:

- Chitosan (Textiles)
- Silver (consumer articles, AgION™)
- Quaternary ammonium compound

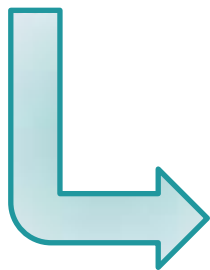


Antimicrobial peptides

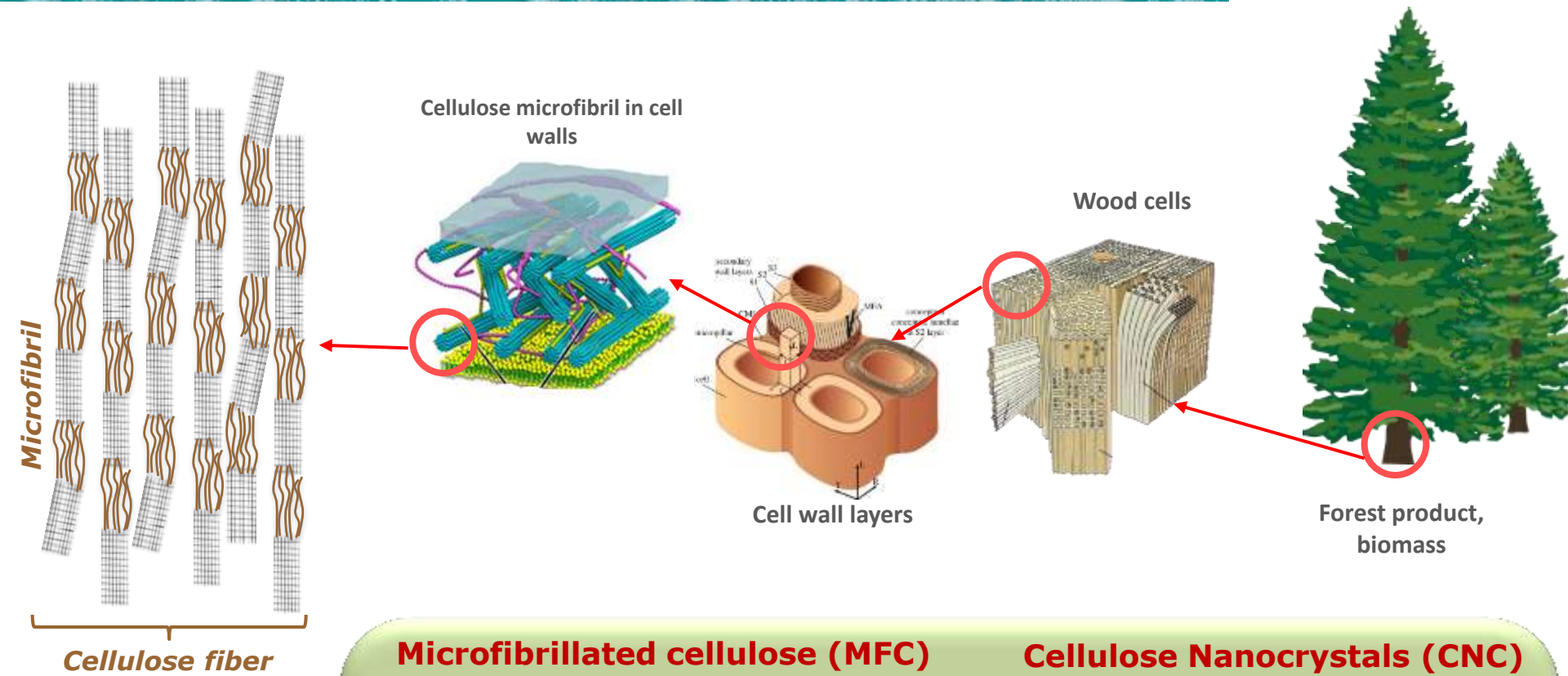
New Materials
For Active packaging



Nanocellulose



New Oxygen Scavenger



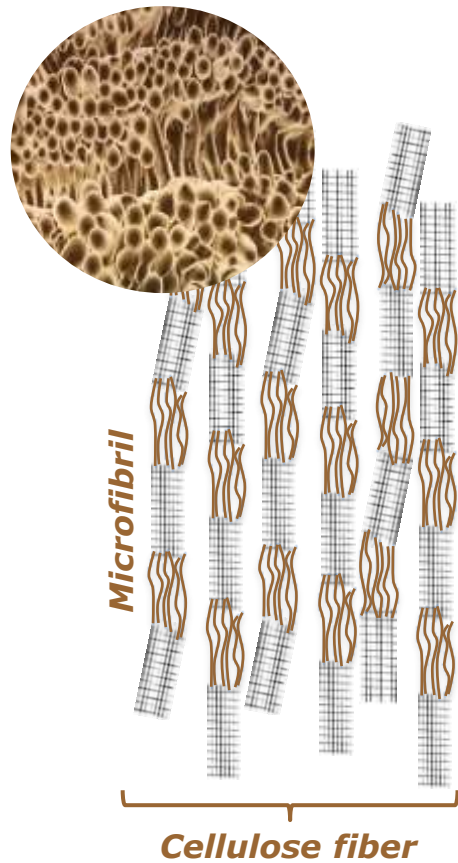
Microfibrillated cellulose (MFC)

D 2-20 nm
L > 1000 nm

Cellulose Nanocrystals (CNC)

D 2-20 nm
L 200-1000 nm

The bottom section of the slide is a green rounded rectangle containing two panels. The left panel is for Microfibrillated cellulose (MFC). It features a diagram of three long, thin microfibrils on the left. To the right is a scanning electron microscope (SEM) image showing a dense network of these microfibrils. A circular inset provides a magnified view of the microfibrils. The right panel is for Cellulose Nanocrystals (CNC). It features a diagram of several short, rod-like nanocrystals on the left. To the right is an SEM image showing a dense network of these nanocrystals. A circular inset provides a magnified view of the nanocrystals.



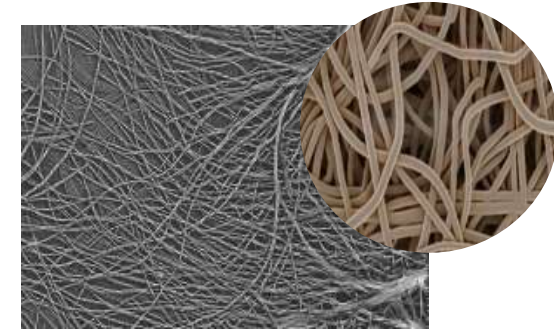
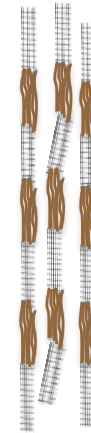
2. Mechanical Homogenization

1. Defibrillation

1. Hydrolysis

2. Dialysis /UF

Microfibrillated cellulose (MFC)



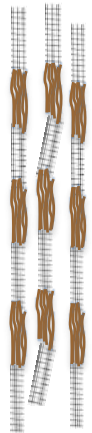
D 2-20 nm
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Cellulose Nanocrystals (CNC)



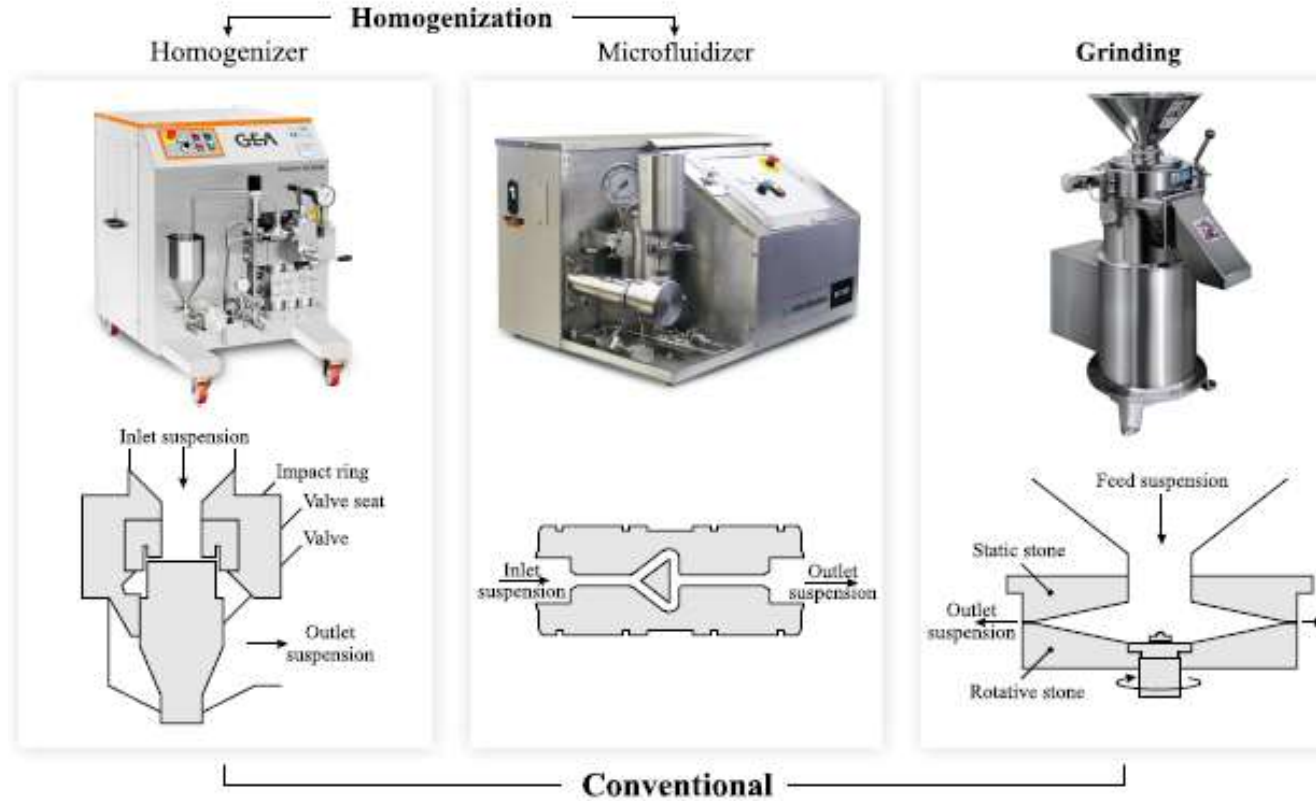
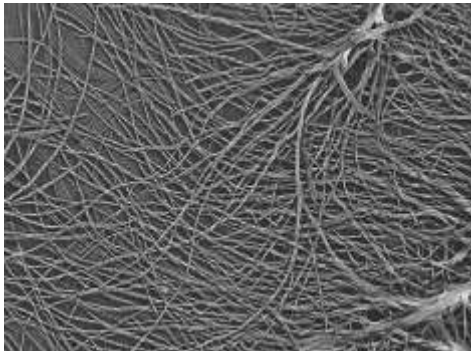
D 2-20 nm
L 200-1000 nm

Nanofibrils & process

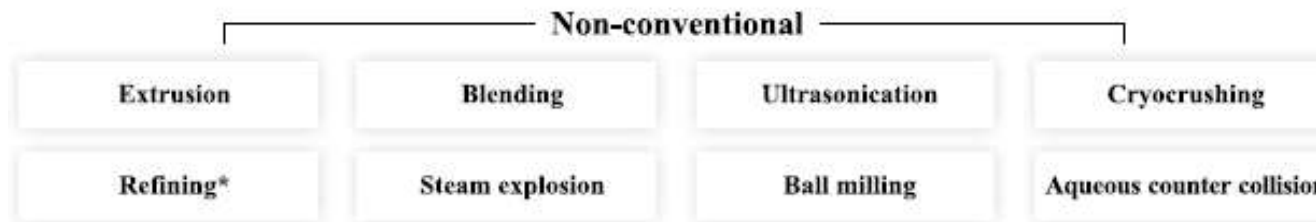


D 2-20 nm
L > 1000 nm

Microfibrillated cellulose (MFC)

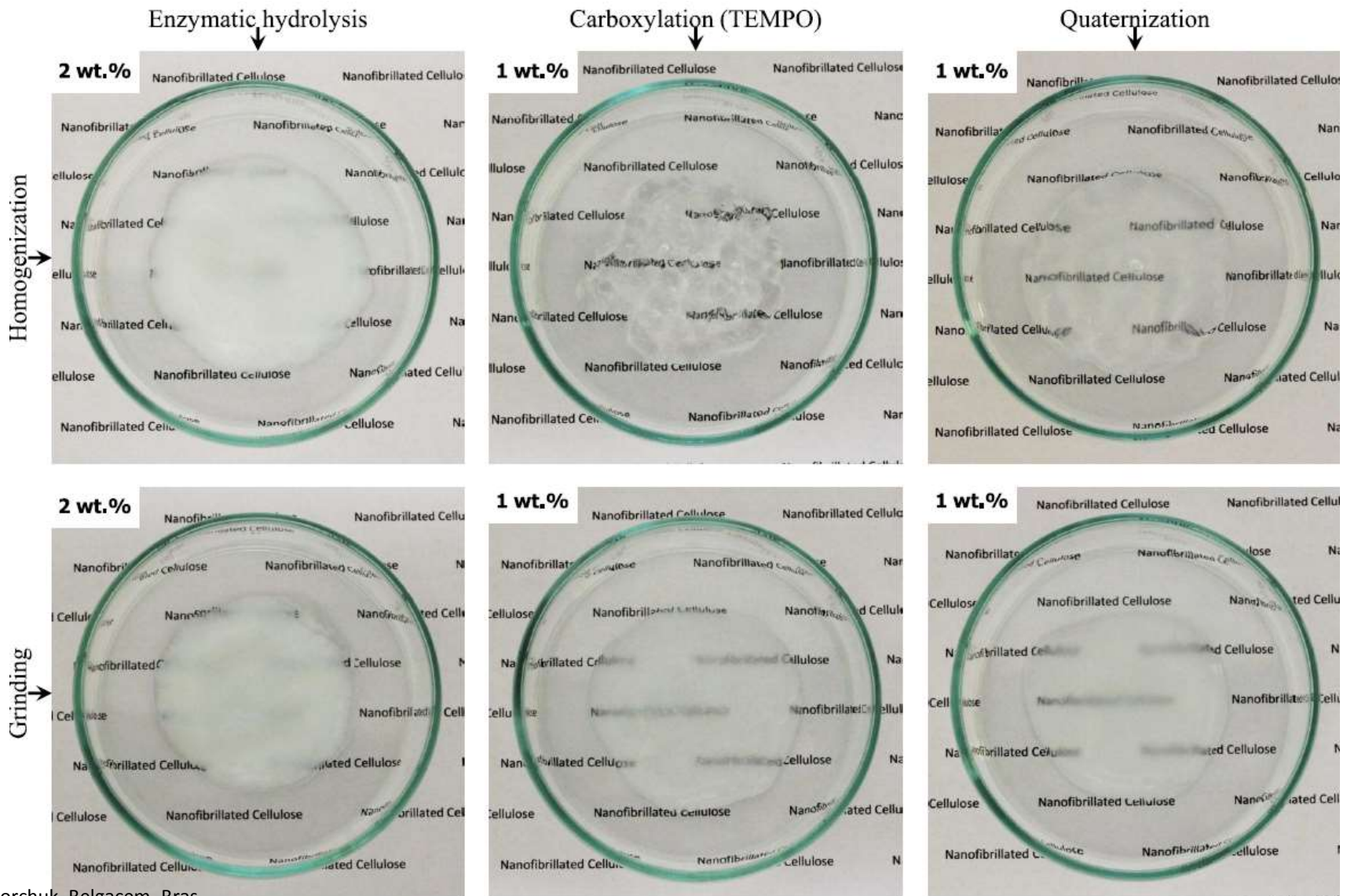


Mechanical processes for CNF production



Nechporchuk, Belgacem, Bras
Production of cellulose nanofibrils: a review of recent advances, Ind crops (2016), in press

* as a principal mechanical process



Nanocelluloses

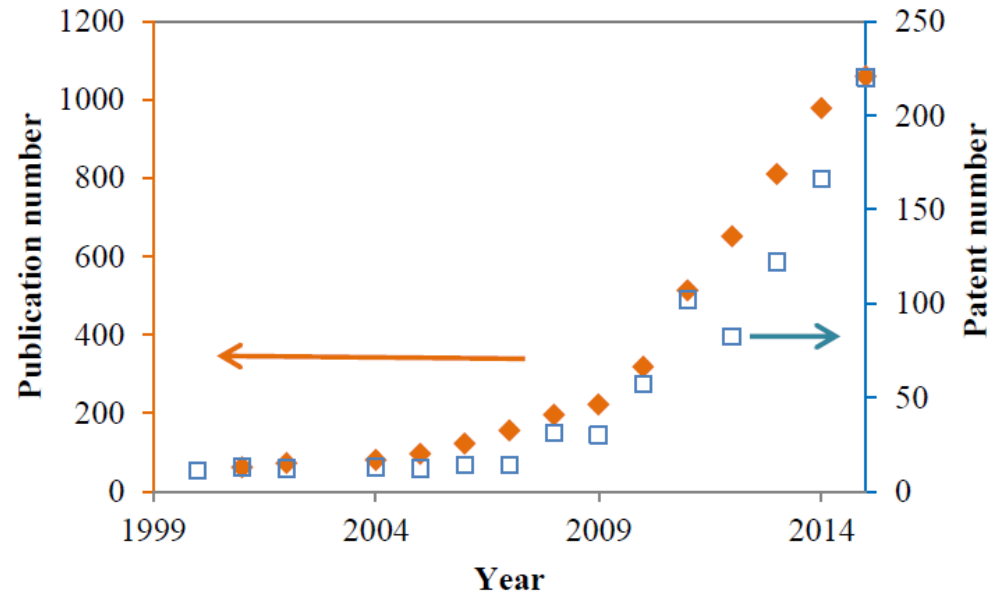
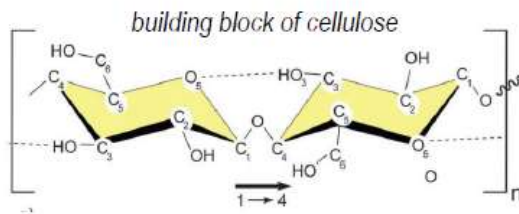
Nanomaterials

- 1 dimension < 100 nm
- high surface area (>100 m² /g)
- novel characteristics



Cellulose

- most available biopolymer on earth
- 200 billion tons / year (3 % used)
- many sources available



Evolution of annual non-cumulative number of publications and patents on nanocellulose

(Source: SciFinder, July 2016 – descriptors : cellulose nanofibrils, cellulose microfibrils, cellulose nanocrystals, cellulose nanowhiskers, microfibrillated cellulose)

De-risking scale-up

GOVERNMENT & INDUSTRY CO-INVESTMENT

World's first **Cellulose Nano-Crystals** plant

1 ton/day - Joint Venture (CelluForce) with Domtar

- Capital: \$45 M
- R&D to Demo: \$10.5 M



World's first **Cellulose Filament** plant

5 ton/day - Strategic alliance with Kruger

- Capital: \$43 M
- R&D to Demo: \$21.5 M



Canada's first **Lignin Separation** plant

30 ton/day - Licensing to West Fraser (>\$10M)

- Capital: >\$10 M
- R&D to Demo: \$12.5 M





2015

Europe's first pilot facility for nanocrystalline cellulose will be based on technology developed by Melodea by the end of 2016

Europe's first pilot facility for nanocrystalline cellulose MoRe Research, alongside Holmen and SP Technical Research Institute of Sweden, is investing in nanocrystalline cellulose by building a pilot facility in Örnsköldsvik.



The facility will be the first of its kind in Europe and represents an important step, allowing interested companies to develop nanocrystalline cellulose from cellulose-based material on a large scale.

Nanocrystalline cellulose has many interesting material properties, and can be used for example as a building material, in biocomposites, printed electronics and dye additives. The operation in the pilot plant is based on technology developed by an Israeli start-up company Melodea.



Melodea
Bio Based Solutions



Flagships in Call 2016

Objective: deployment of a technology, which has been already demonstrated leading to a system, which is complete and qualified (TRL8) for successful commercial operation (large scale production facility in Europe)

TOPIC	FOCUS
BBI.VC1.F1	BIOETHANOL : second generation bioethanol production built on lignocellulosic non-food feedstock (straw)
BBI.VC2.F2	Microfibrillar Cellulose (MFC) : large-scale supply and market creation of MFC to demonstrate an industrial symbiosis between the biomass and the forest industry
BBI.VC1.F1	CELLULOSIC ETHANOL : Cellulosic ethanol from unused crop residues and crops grown on marginal lands



irregaard / News / EUR 25 million in EU funding for Exilva

Borregaard Receives EUR 25 million for Commercialization of Microfibrillar Cellulose from the EU



Borregaard Receives EUR 25 million for Commercialization of Microfibrillar Cellulose from the EU

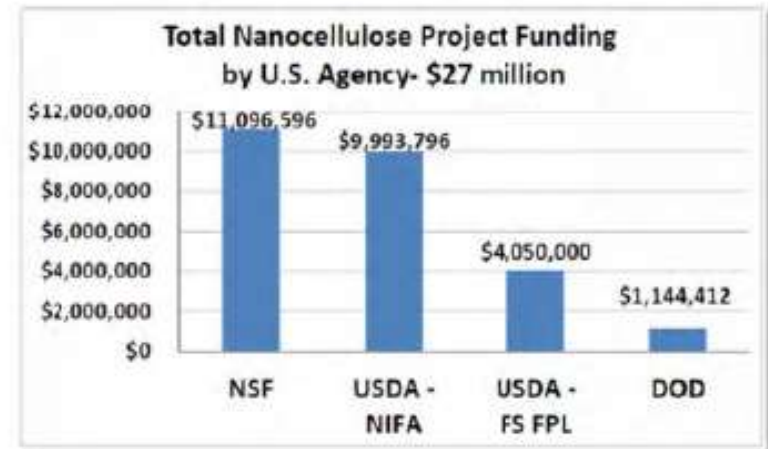
Borregaard has received a funding commitment of EUR 25 million (NOK 232 million), for the development and commercialization of

=> Nanocellulose = 2nd **priority** of european Bioeconomy

=> Not only fashionable but also **sustainable**

Governments throughout the world have recognized the potential economic and societal benefits of nanocellulose and have made significant R&D investments:

- CANADA: ~\$71 million
- EUROPE: ~\$25 million
- USA: ~\$27 million
- JAPAN: ~\$38 million *annually*



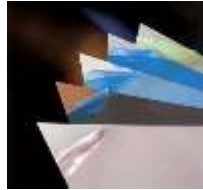
Growing interest among US Federal Agencies in nanocellulose research. RFPs on *specific* nanocellulose topics issued in 2017 by DOE and USDA.

Source: Agenda 2020 Cellulose Nanomaterials Working Group, Global Partnerships Workshop, 2016
TAPPI International Nanotechnology Conference, Grenoble, France

CNF – Production status

Different needs in different markets

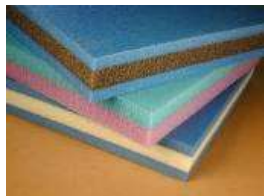




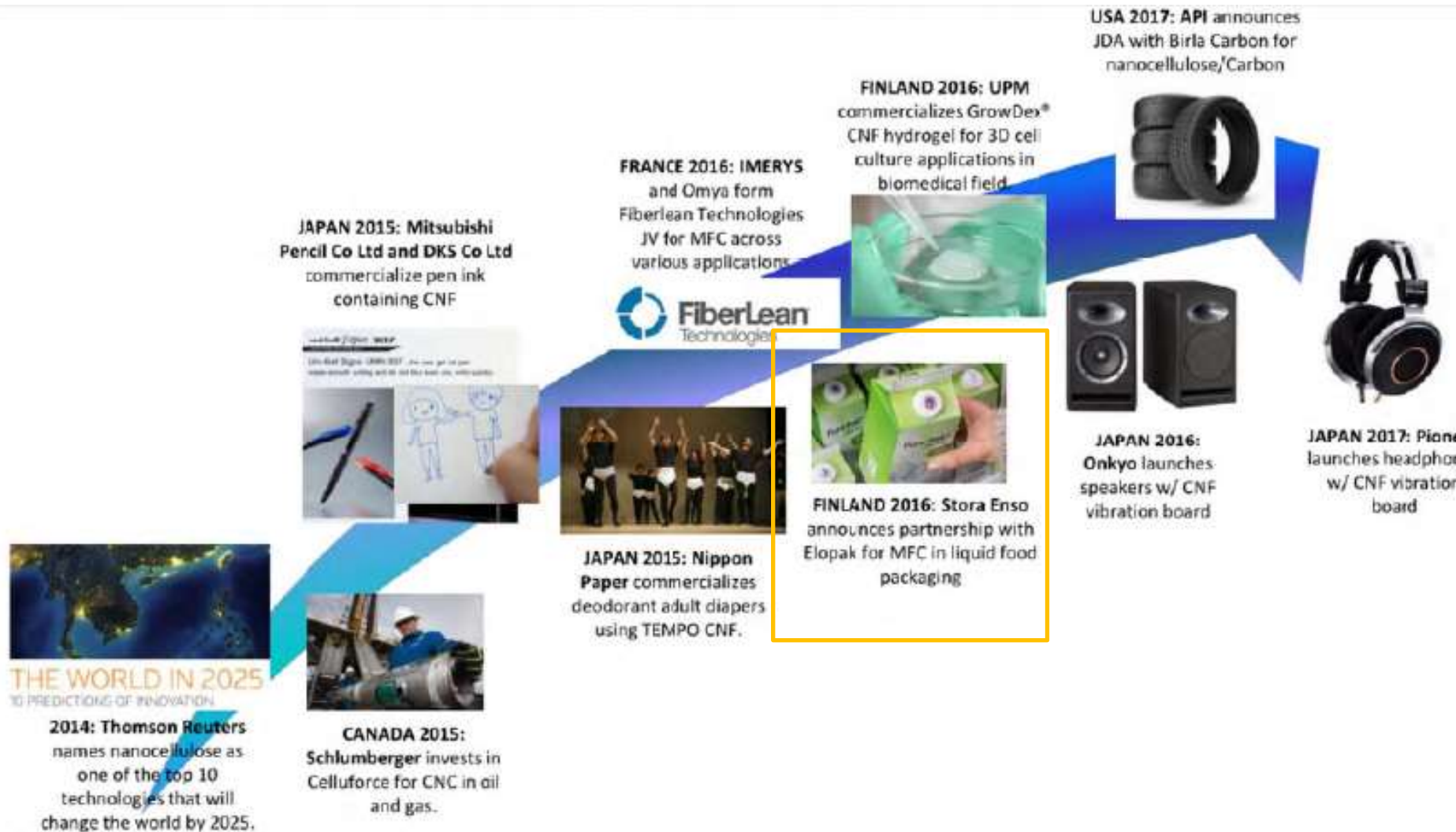
23 million tonne potential (figure in ktons)



	Market Size	Potential Loading	Nano Cellulose Potential	Potential @ 5% Market Penetration	CNF Potential	CNC Potential	CNF	CNC
Paper and Paperboard	400,000	5.0%	20,000	1,000	95%	5%	950	10*
Paints and Coatings	40,000	2.0%	800	40	5%	95%	2	38
Composites	9,000	2.0%	180	9	5%	95%	0	9
Films and Barriers	9,670	2.0%	193	10		100%	0	10
Excipients	4,600	2.0%	92	5	10%	90%	0	4
Natural Textiles	34,500	2.0%	690	35		100%	0	35
Manufactured Textiles	56,300	2.0%	1,126	56		100%	0	56
Cement	15,000	0.5%	75	4	5%	95%	0	4
Oil and Gas	17,500	1.0%	175	9	10%	90%	1	8
Nonwovens	7,000	2.0%	140	7		100%	0	7
Adhesives	4,000	2.0%	80	4	5%	95%	0	4
TOTAL			23,551	1,178			954	184

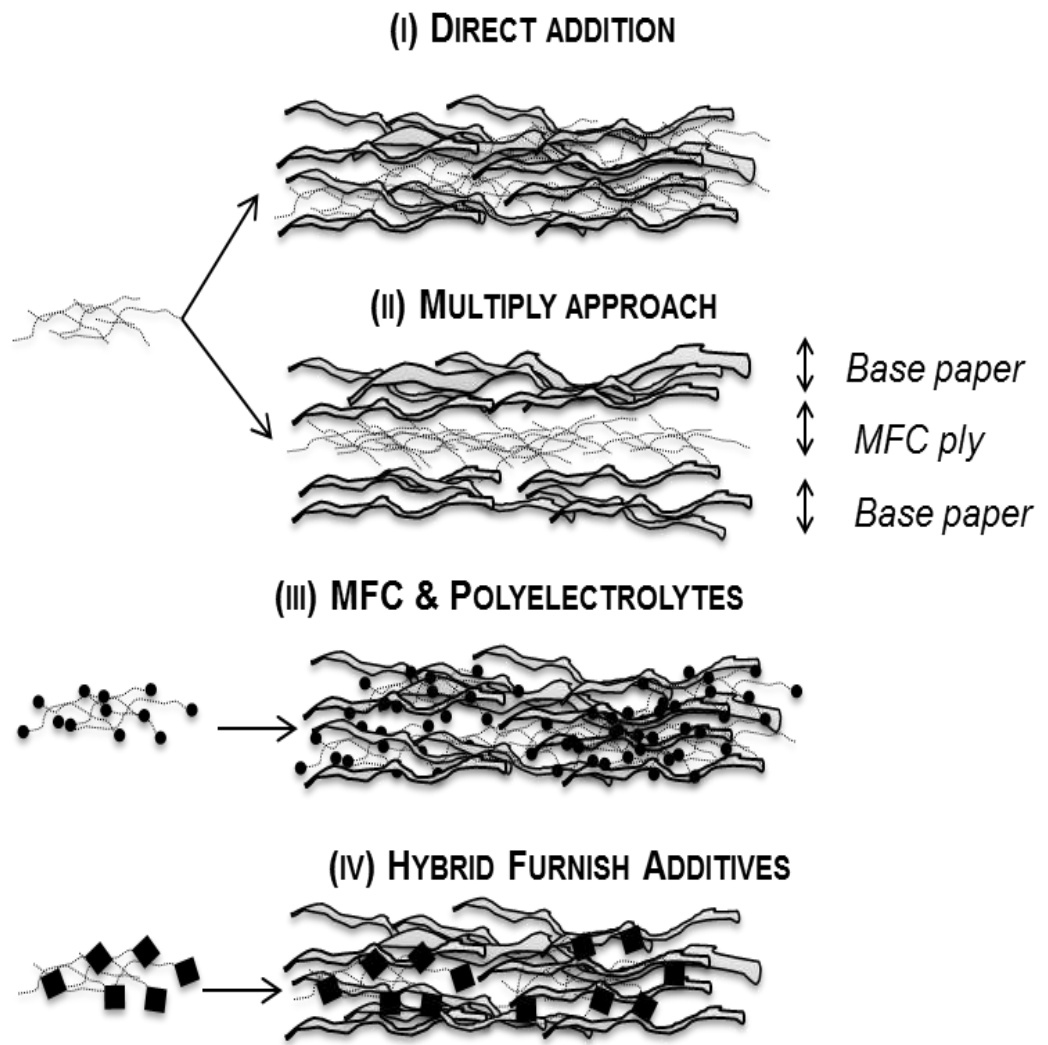
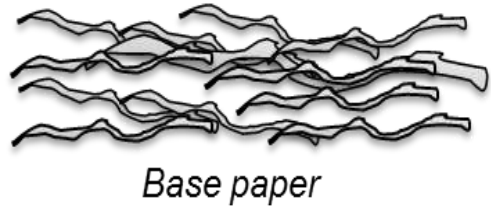


Source: RISI, *Nanocellulose: Technology Applications, and Markets*

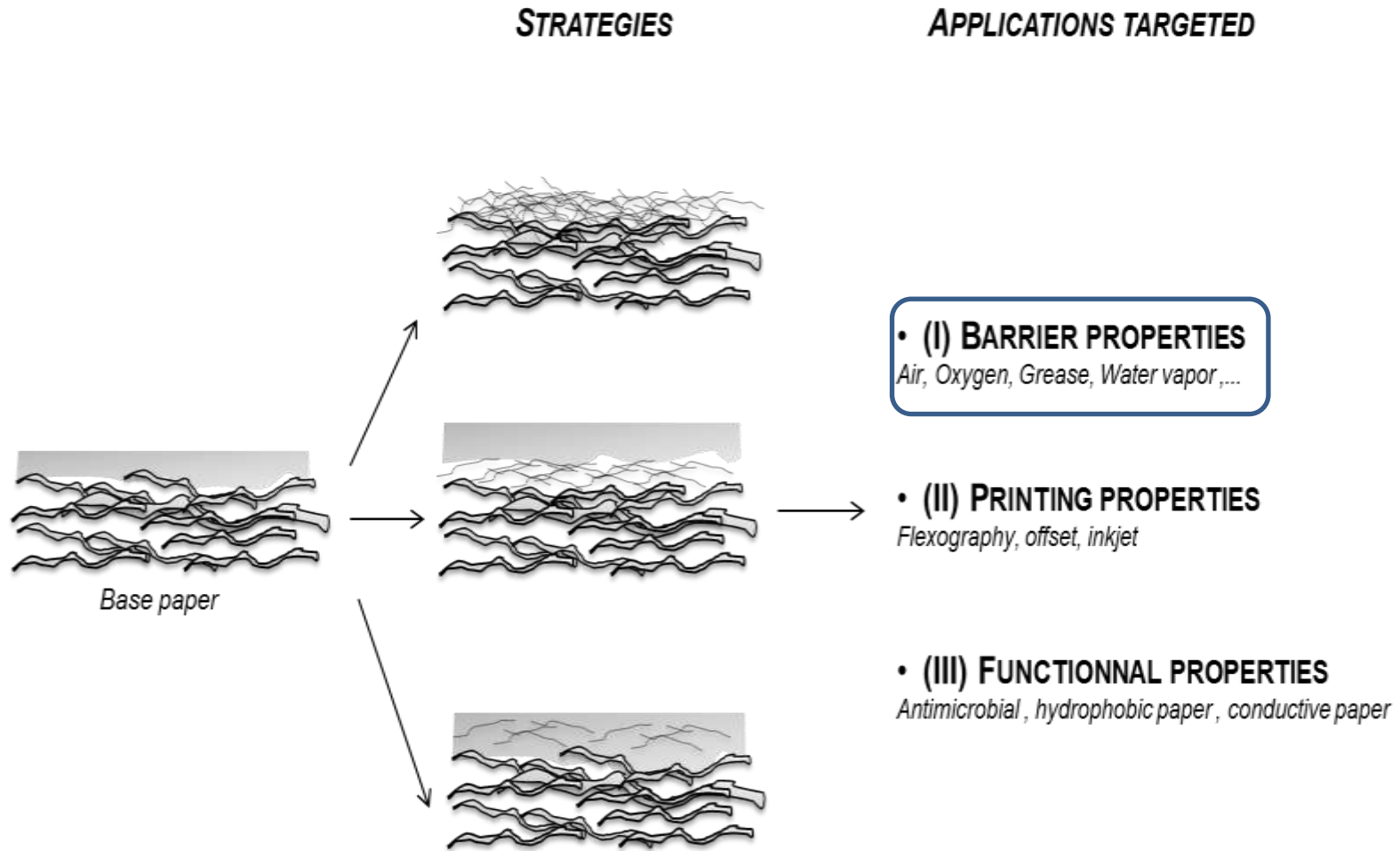


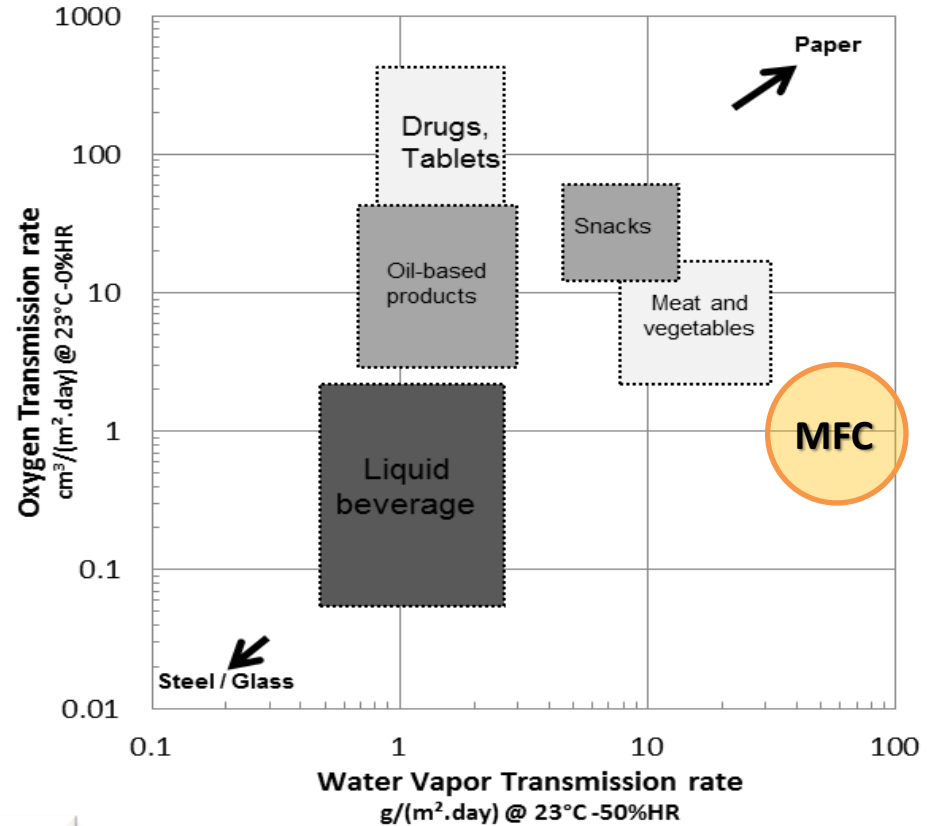
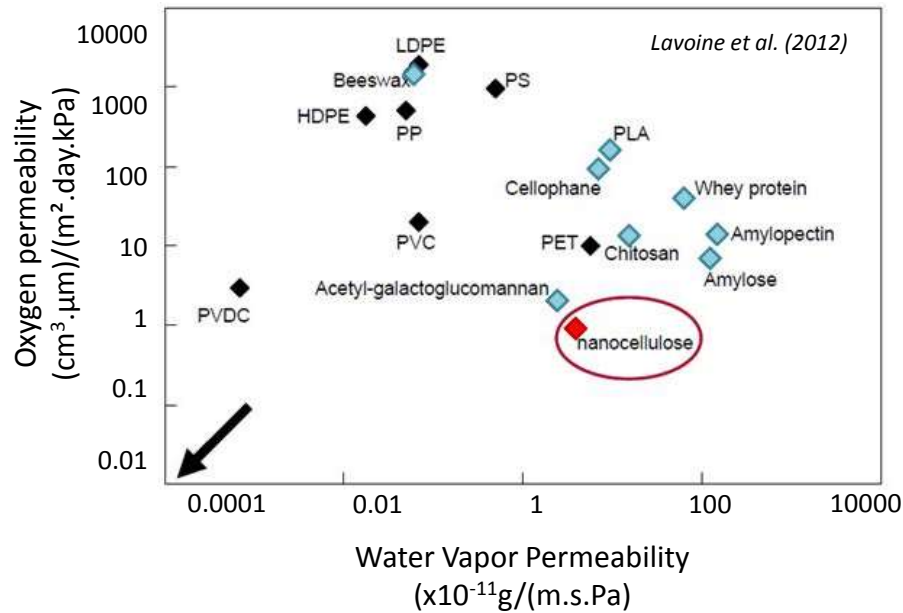
Several strategies

BUT not with NCC

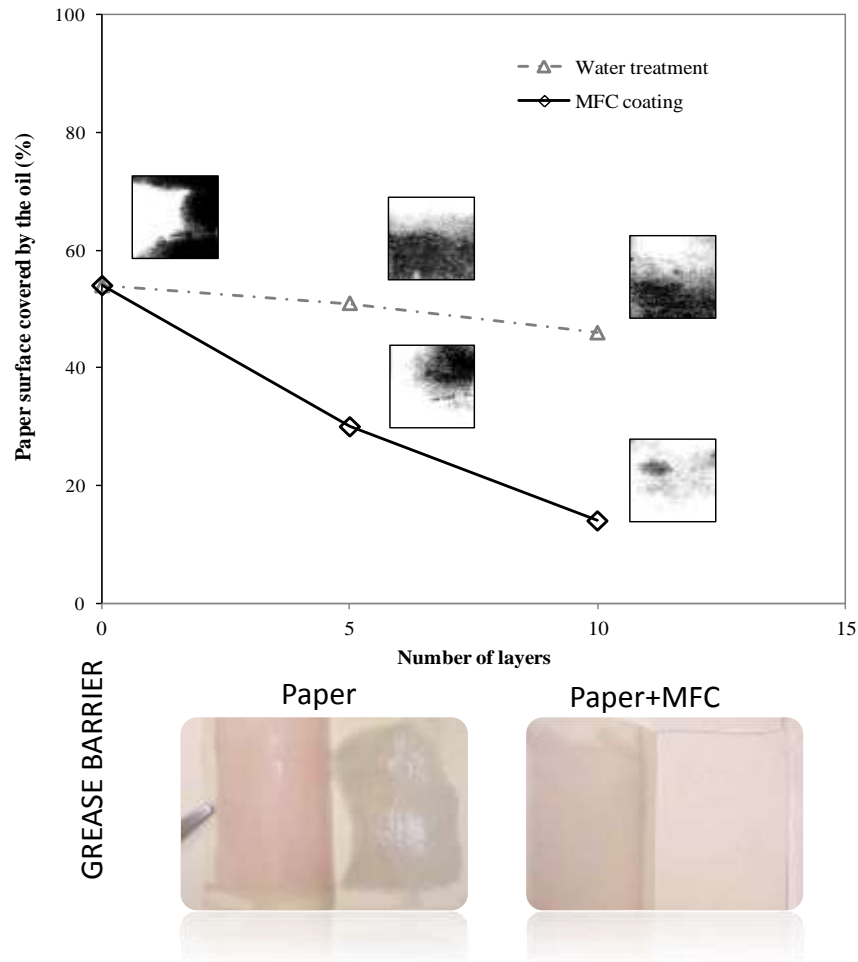


« CNF is a dry strength additive
BUT not the magic pill »
T. Lindstrom, Tokyo Paper, 2015

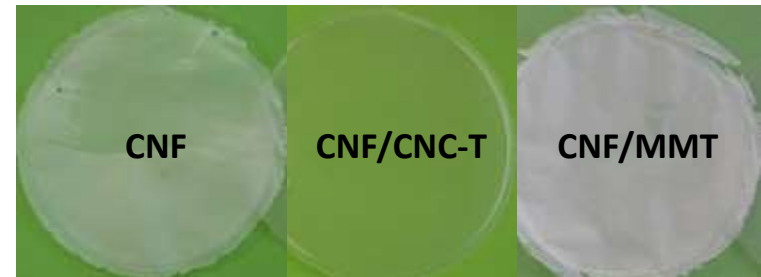
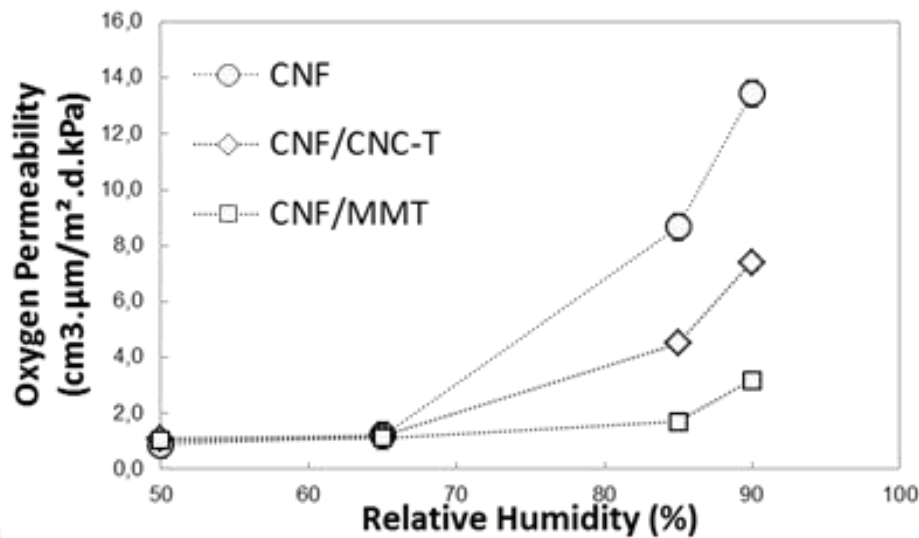
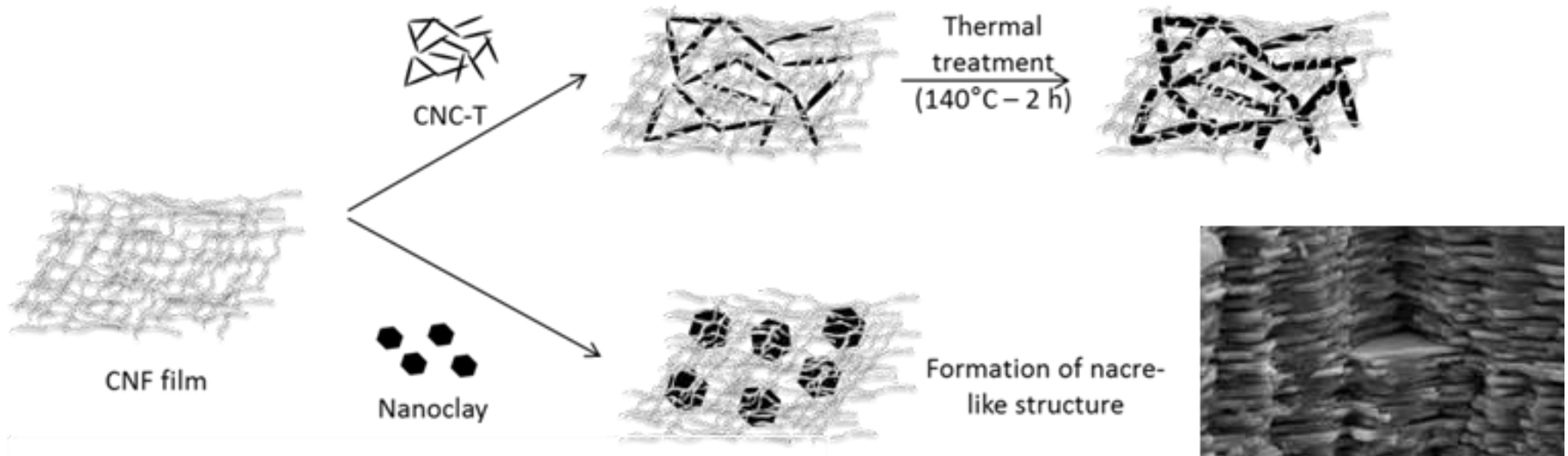


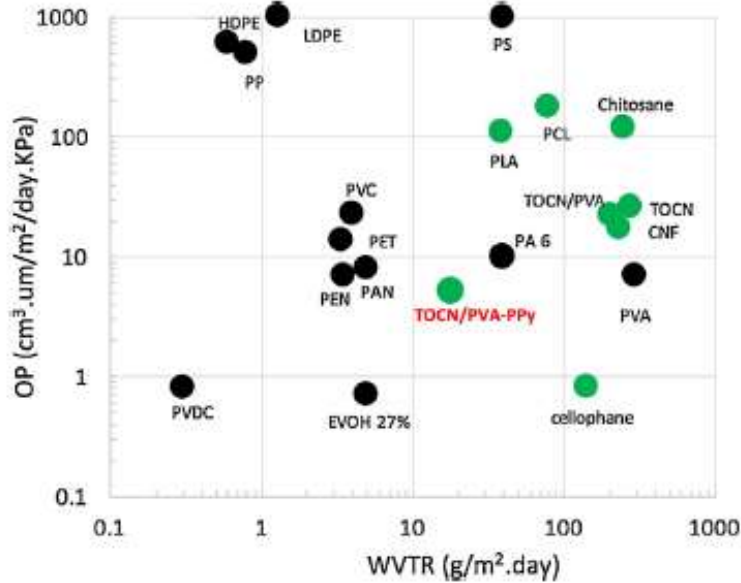


**=> CNF = The best bio-based barrier
...at low HR**



⇒ Interesting grease barrier of MFC coating





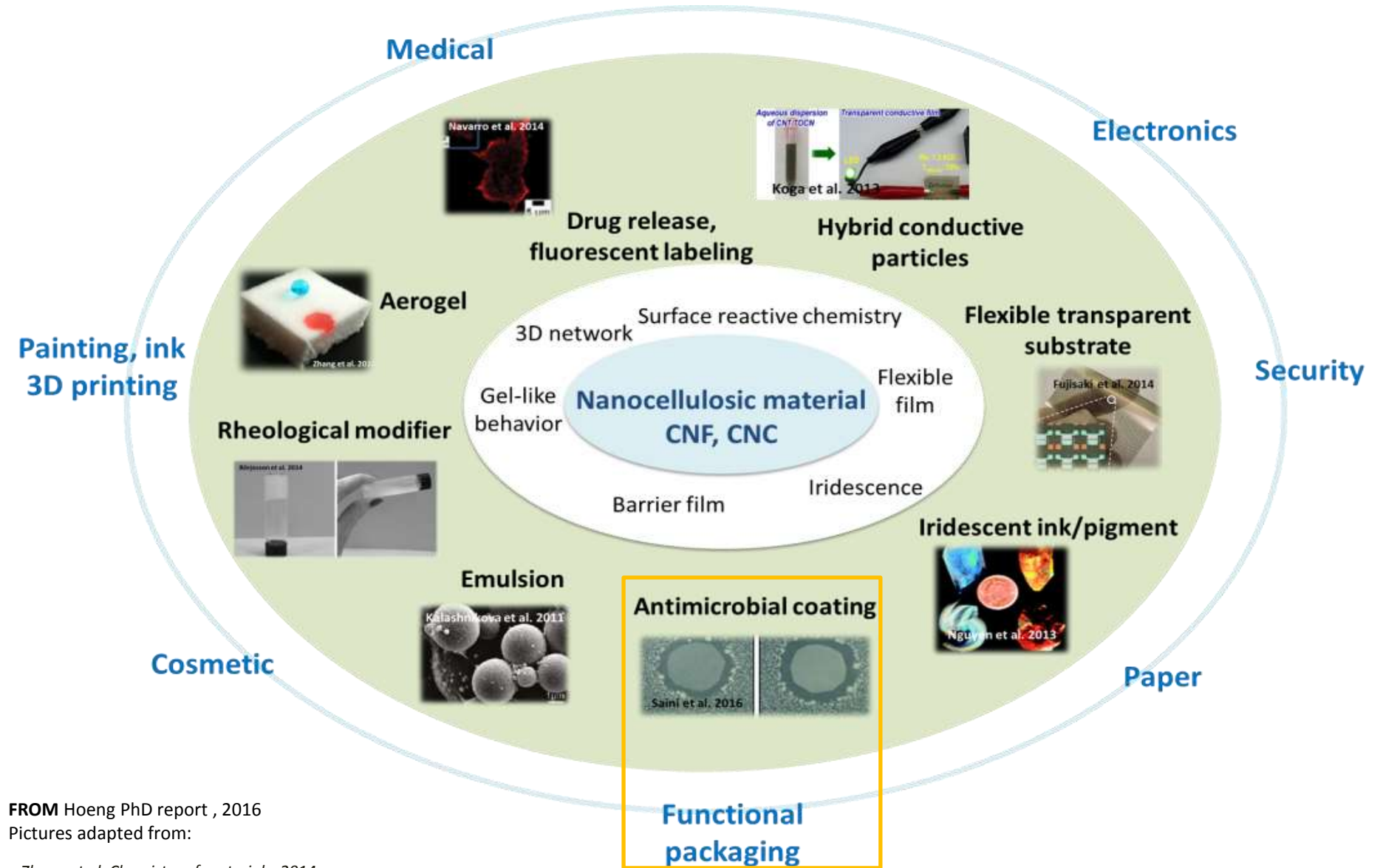
Bideau, Bras, Adoui, Loranger, Daneault ; Polypyrrole/nanocellulose composite for food preservation: Barrier and antioxidant characterization, Food Packaging and Shelf Life 12 (2017) 1–8

Fig. 4. Oxygen permeability=f(Water Vapor Transmission Rates) – Polymer films compared to TOCN, TOCN/PVA and TOCN/PVA-PPy films. Green points: Biopolymers; Dark points: petroleum polymers. Adapted from Jester (2005).

Perspectives:

Supramolecular system to scavenge molecules, radicals, oxygen



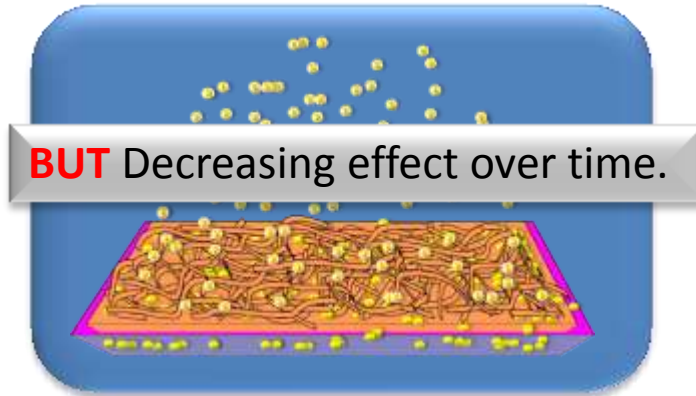


FROM Hoeng PhD report , 2016
 Pictures adapted from:

Zhang et al. *Chemistry of materials*, 2014
 Koga et al. *NPG Asia*, 2014
 Saini et al, *Applied materials & interface* 2016

Wu et al. *Langmuir*, 2008
 Moran-Mirabal, *Cellulose: fundamental aspects, Chapter 1*, 2013
 PFI, Ching-Carrasco

(i) Incorporation into CNF network

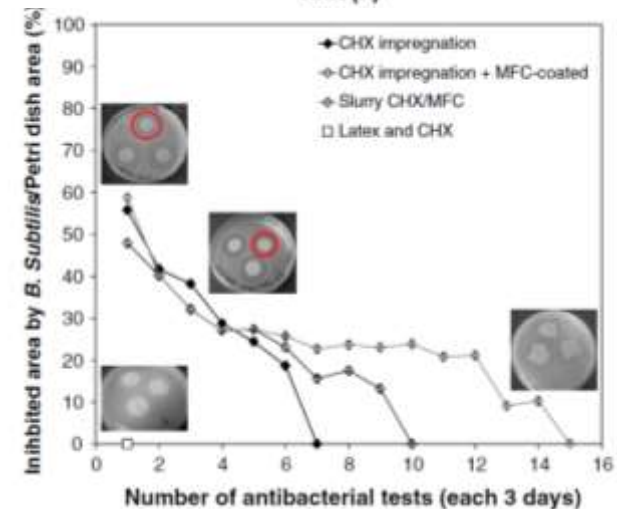
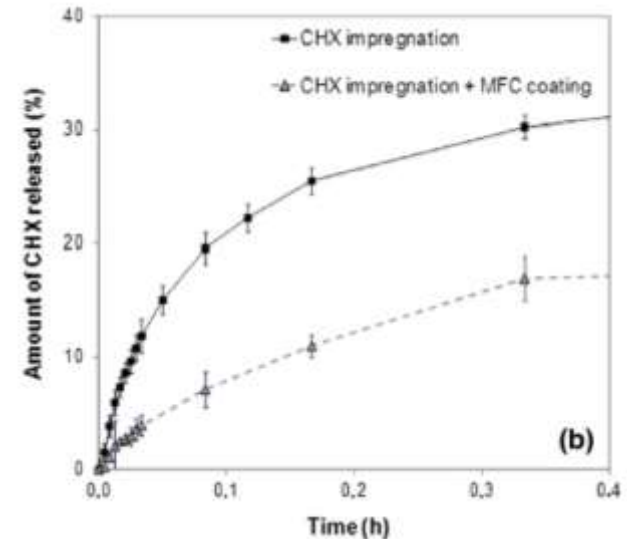


Release mechanism

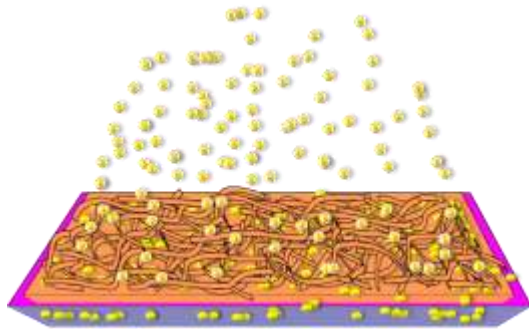
- ✓ Antimicrobial agents incorporated in the packaging.
- ✓ Migrate into food through diffusing and partitioning.
- ✓ Very positive impact of MFC for release monitoring
=> prolonged antimicrobial activity whatever molecules

Lavoine, N.; Desloges, I.; Sillard, C.; Bras, J. (2014)

Controlled release and long-term antibacterial activity of chlorhexidine digluconate through the nanoporous network of microfibrillated cellulose, *Cellulose*, 21(6), 4429-4442.



(i) Incorporation into CNF network



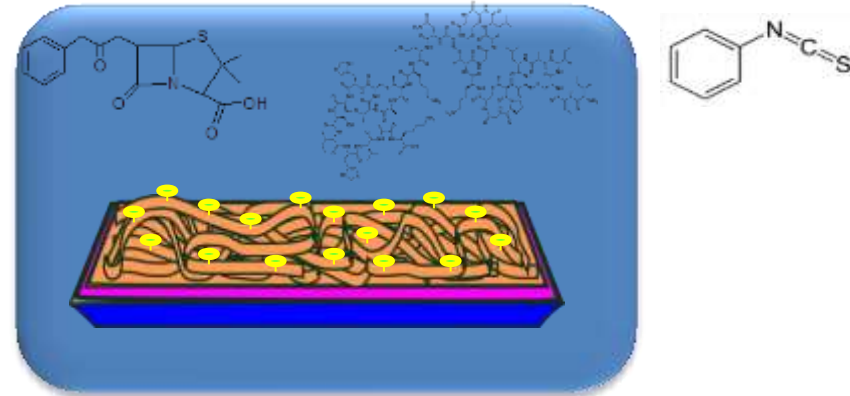
Release mechanism

- ✓ Antimicrobial agents incorporated in the packaging.
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- ✓ Decreasing effect over time.

- Saini, ; Belgacem, N; Mendes, J; Elegir, G; Bras, J
Contact Antimicrobial Surface Obtained by Chemical Grafting of Microfibrillated Cellulose in Aqueous Solution Limiting Antibiotic Release, ACS Applied Materials & Interfaces (2015), 7(32), 18076-18085

-Saini, M. N. Belgacem, K. Missoum, J. Bras,
Natural active molecule chemical grafting on the surface of microfibrillated cellulose for fabrication of contact active antimicrobial surfaces, Industrial Crops and Products (2015), Accepted-in press.

(ii) Immobilisation onto CNF

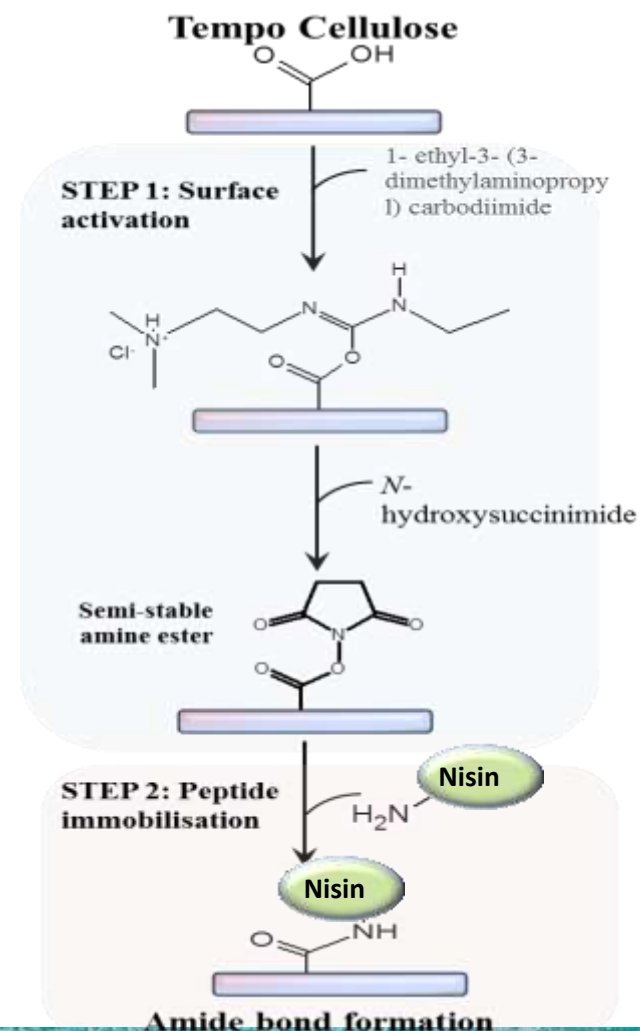
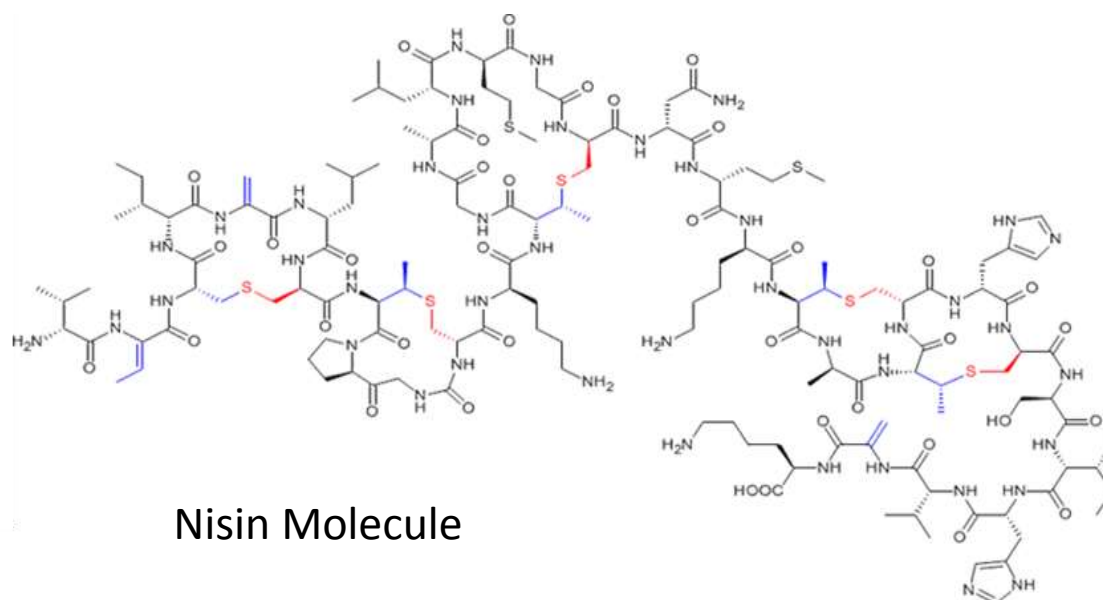


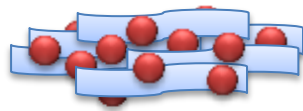
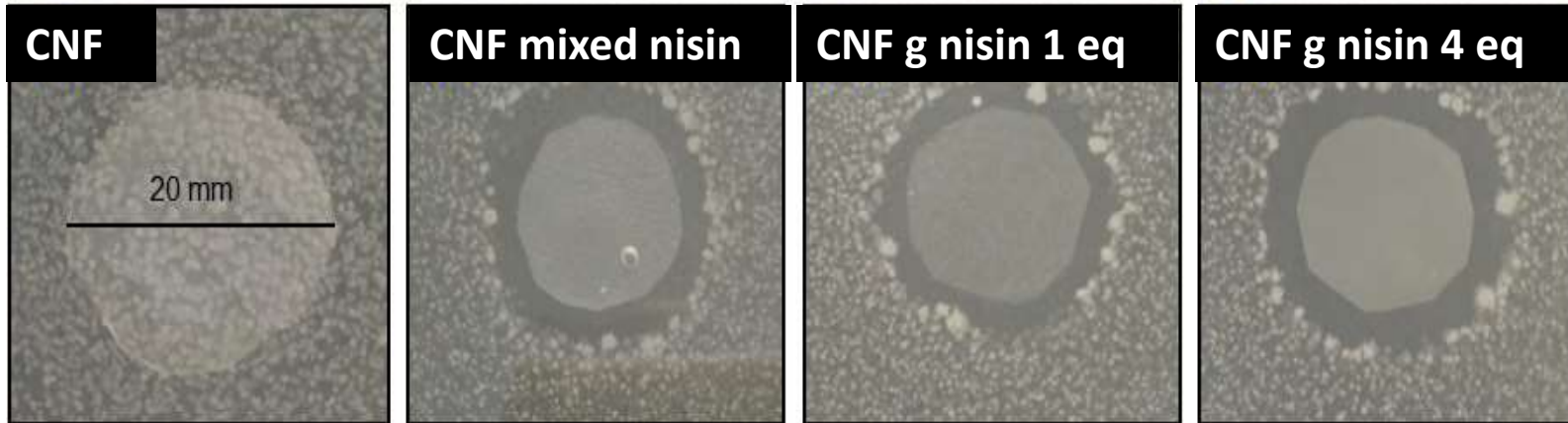
Contact mechanism

- ✓ Antimicrobial agents immobilized on the packaging.
- ✓ Microbial suppression at the contact surface without diffusion.
- ✓ Prolong effect.

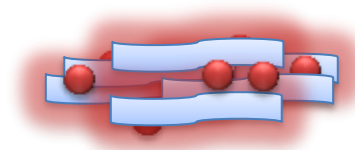
Globally, 3, 51,000 people die every year due to the food poisoning
Antimicrobials in bulk is unable to target the food surface

Nisin: approved in 40 countries over 50 years.
Allowed in canned fruits, vegetables, and cheese.



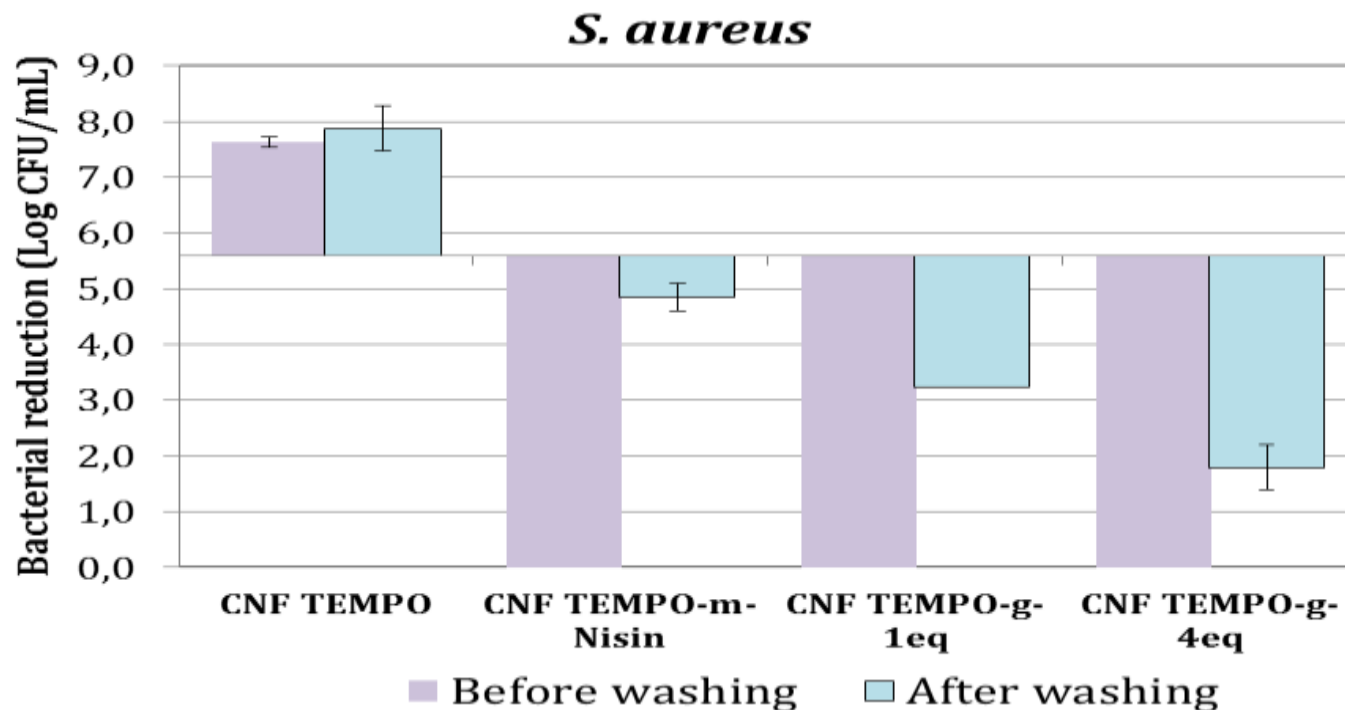


CNF film mixed
with Nisin



CNF film grafted
with Nisin

- Zone of inhibition showed presence of non-covalent bonded nisin
- uncontrolled and non-covalent immobilization of peptide forms an unstable layer



Grafting more efficient for stronger bacteria



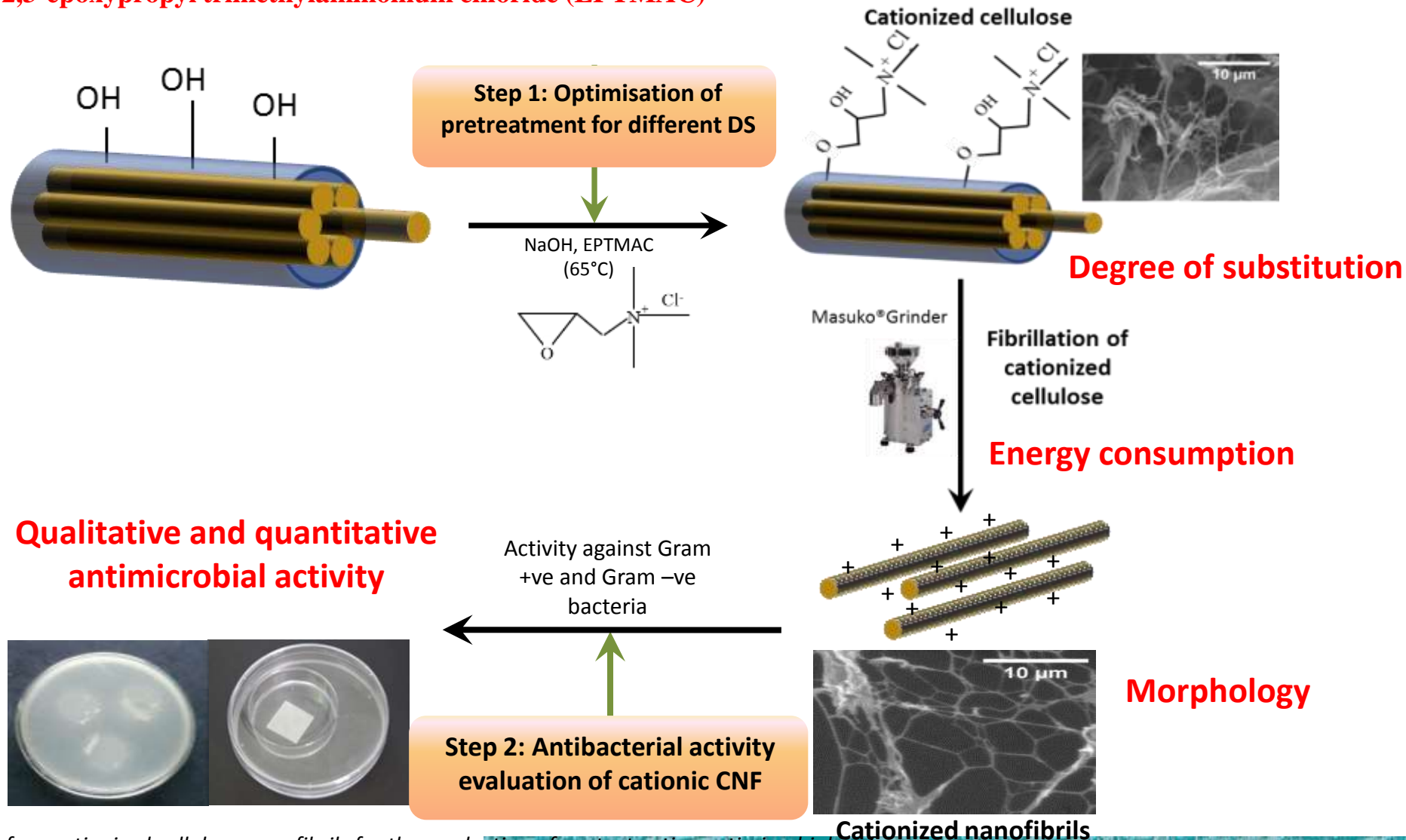
CNF film mixed with Nisin

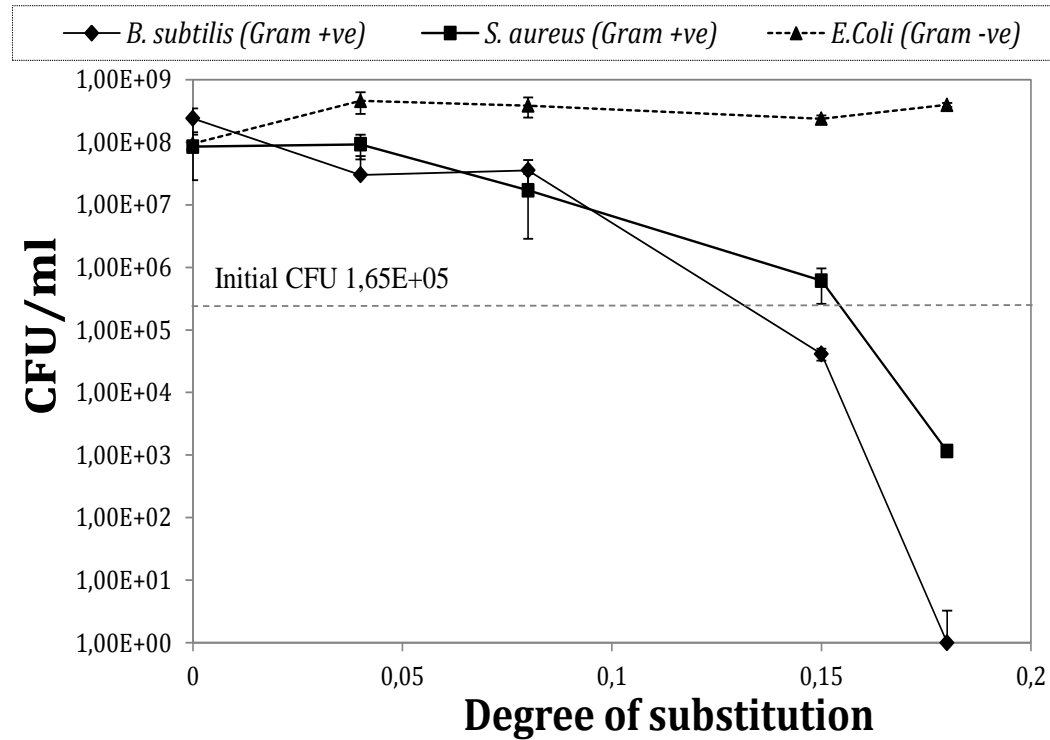


CNF film grafted with Nisin

After washing for 48 hours, high bacterial killing (60% CNF Tempo-g-1-eq and 78% CNF Tempo-g-4eq) in comparison with 40% killing effect by CNF Tempo-m-Nisin.

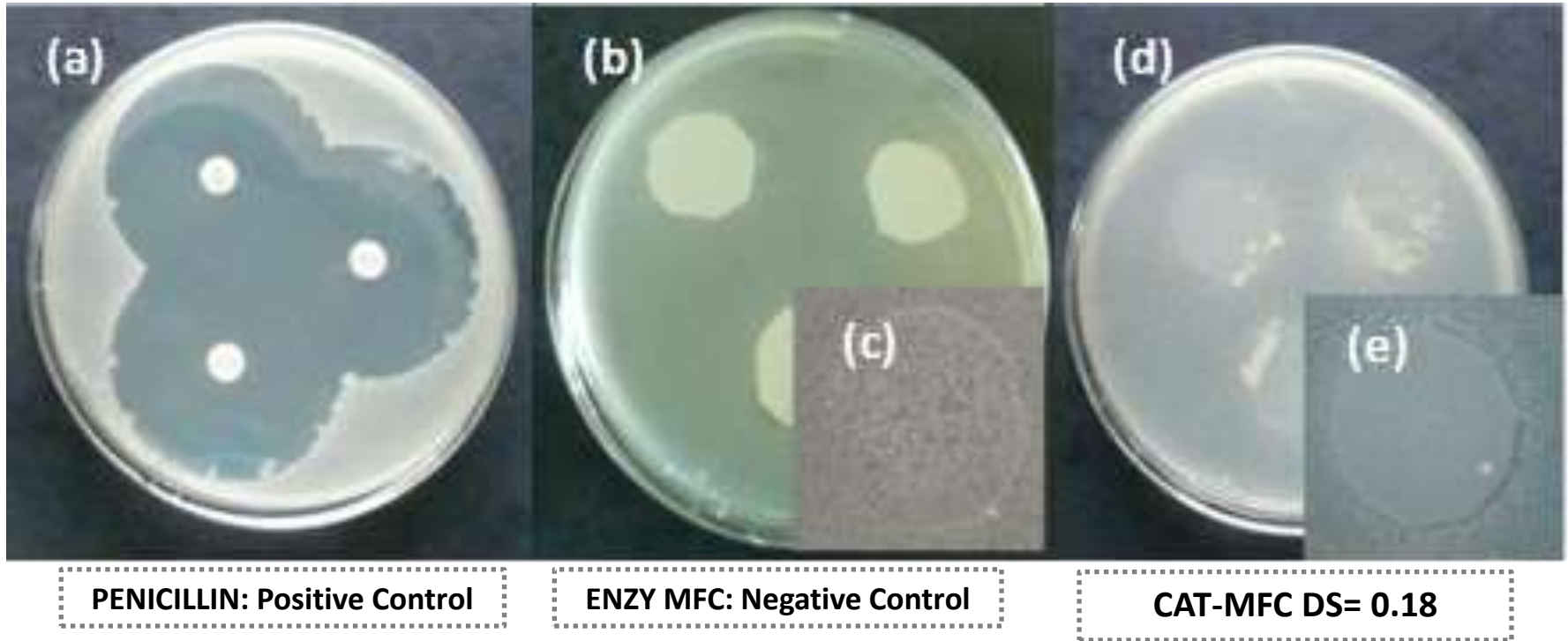
2,3-epoxypropyl trimethylammonium chloride (EPTMAC)





- ✓ CATMFC DS=0,04: Antimicrobial agents lower than Minimum inhibitory concentration.
- ✓ CATMFC DS=0,18: 3 log reduction with high SD => **samples are heterogenous.**
- ✓ E.coli – need to increase degree of substitution.

Bacteria: *Bacillus subtilis* (Level 1) Gram +ve



- No Zone of inhibition: No free EPTMAC leaching
- Antimicrobial by contact

Industrial Pilot trial at Multipackaging Solutions

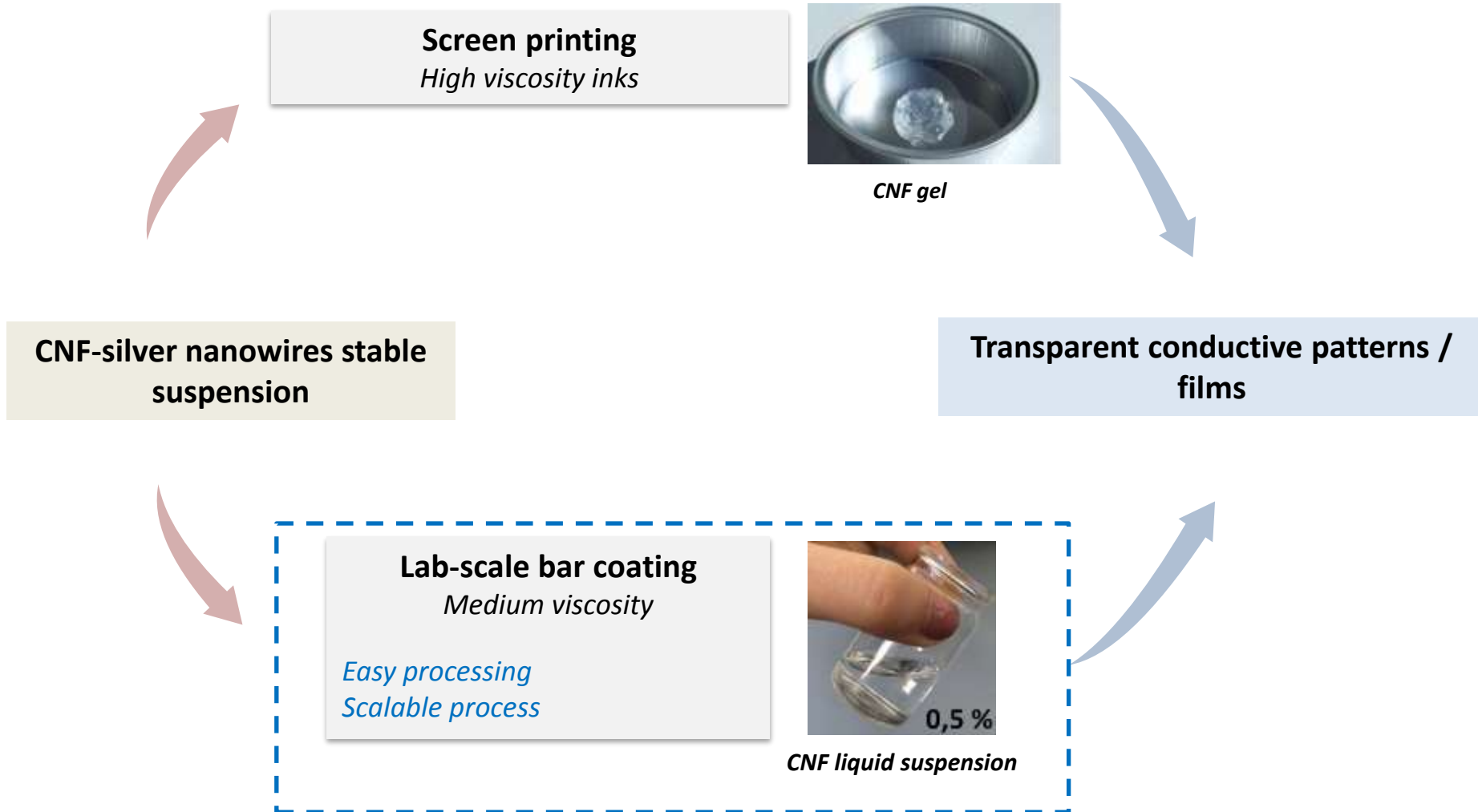


Towards demonstrator



FP1405 COST action

Transparent conductive layer using nanocellulose-silver nanowires suspension





Poly-Ink
Inkjet the future...

www.poly-ink.fr

Poly-Ink develops and markets functional ink implemented by **low viscosities printing techniques**



3 products range

Transparent and conductive inks

Poly-Ink HC

Carbon nanotubes (CNT) based inks – ITO replacement

PolyBioWire

Silver nanowires based inks – ITO replacement

Conductive ink

PolySilver

Silver nanoparticles based inks

Multi-substrate printing

PolyPrimer

Tuned surface properties for various substrates to printed electronics requirements

CNF-silver nanowires coating

R_{sh} (Ω/\square)	13 ± 3
$T_{550\text{ nm}}$ (%)	90.1 ± 0.5
ΔL^*	3.1 ± 1.8



Up-scaled trials and electro-luminescent application

Collaboration University of Pardubice- Czech Republic (ActInPak COST action) + Industrial partner



**CNF- silver nanowires
screen printing ink**



STEP 1

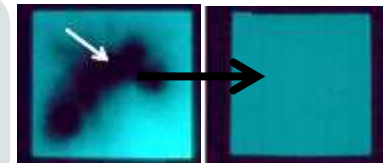
**Large scale ink
preparation**

- ✓ Easy preparation (mixing + mechanical agitation)
- ✓ Adaptable to large scale preparation

STEP 2

**Opto-electronic
devices design**

- ✓ Working opto-electrical devices
 - Issues of chemical stability



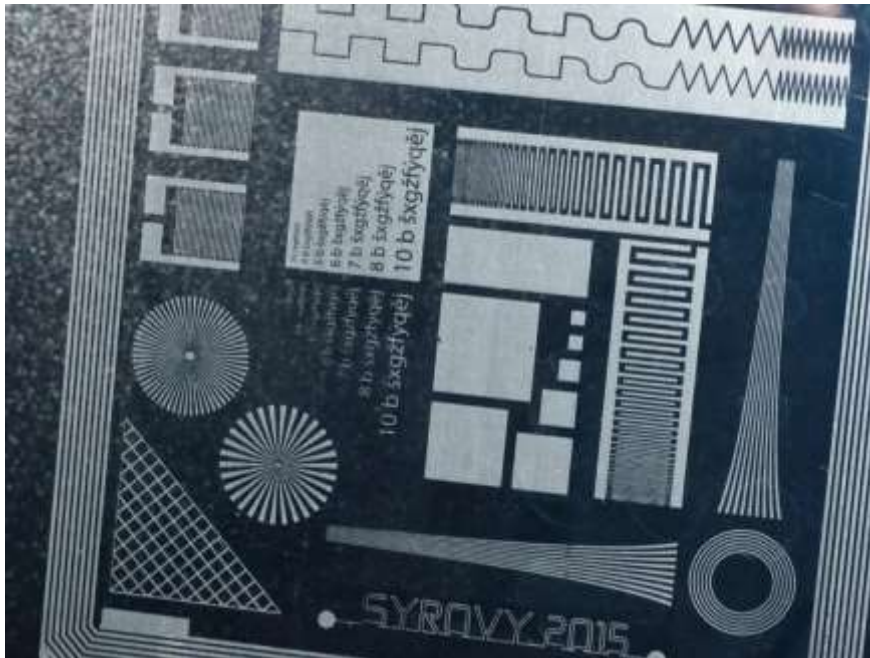
STEP 3

**Industrial roll to roll
printing**

- ✓ Effective roll to roll printing
 - Rheology is not optimal → viscous
 - Clogging of the mesh → drying rate



Printing of PolyBioWire – Screen printing grade



- Low resistance
- High transmittance
- Good patterning

PolyBioWire SP[®] film

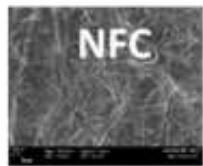
Substrate	PET
R_{sh} (Ω/\square)*	25 ± 3
T% (%)*	$83 \pm 0,5$
ΔL^*	$3,2 \pm 0,7$
Δa^*	$1,4 \pm 0,3$
Δb^*	$6,2 \pm 0,4$

**Best compromise in opto-electrical properties, transparency without substrate*

MATERIALS



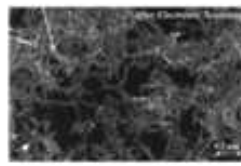
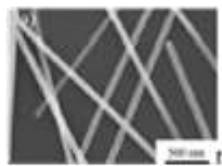
Nanocelluloses
(Stabilizing, Film forming, Entangled network, Functionalization, etc.)



Functional materials

Silver nanowires

Carbon nanotubes

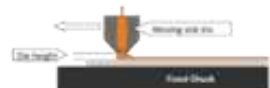


Surface treatment

Coating processes



Bar coating



Spray



Heliogravure

Sensors
(microbial breakdown, gaz, T°, temper detection)

+

PE / PLA / Paper
Antimicrobial active layer
Contact killing action

Localized treatment

Printing processes



Inkjet



Screen-printing



Flexography

Applications
SAFE & SMART packaging

Medical packaging



Food packaging



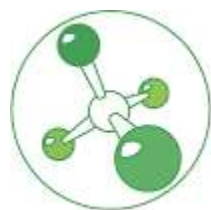
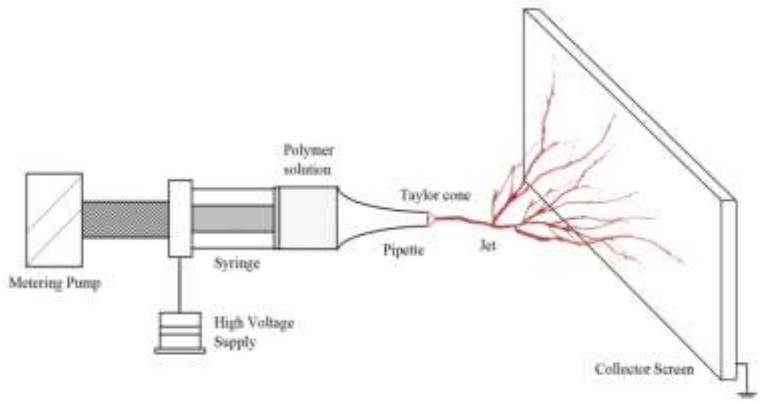
Smart detection label



- ❑ **NEW SOLUTIONS IN MATERIALS**
 - ❑ Antimicrobial Peptides for Bioactive Packaging
 - ❑ Cellulose nanofibers (CNF) as a new opportunity

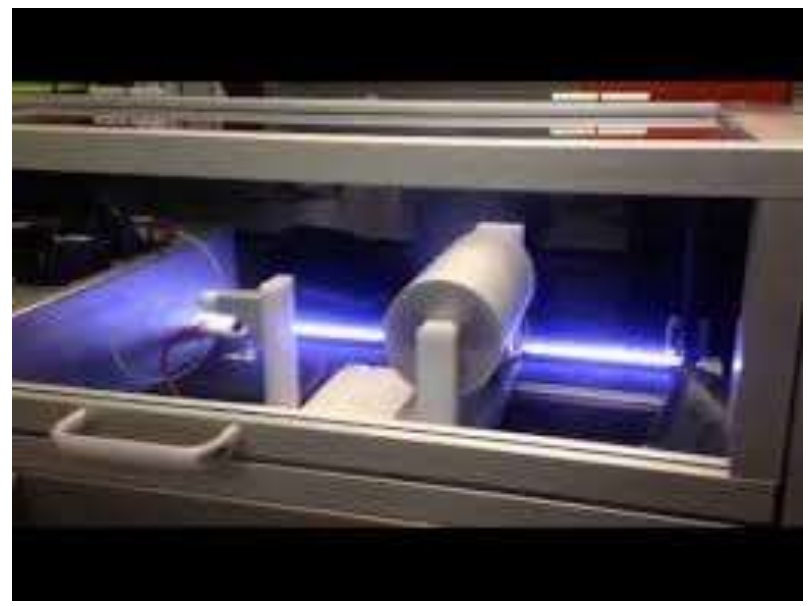
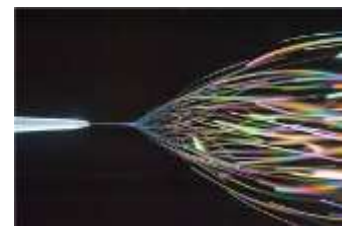
- ❑ **NEW SOLUTIONS IN PROCESSES**
 - ❑ Encapsulation systems in the packaging material
 - ❑ Electrospinning for active materials

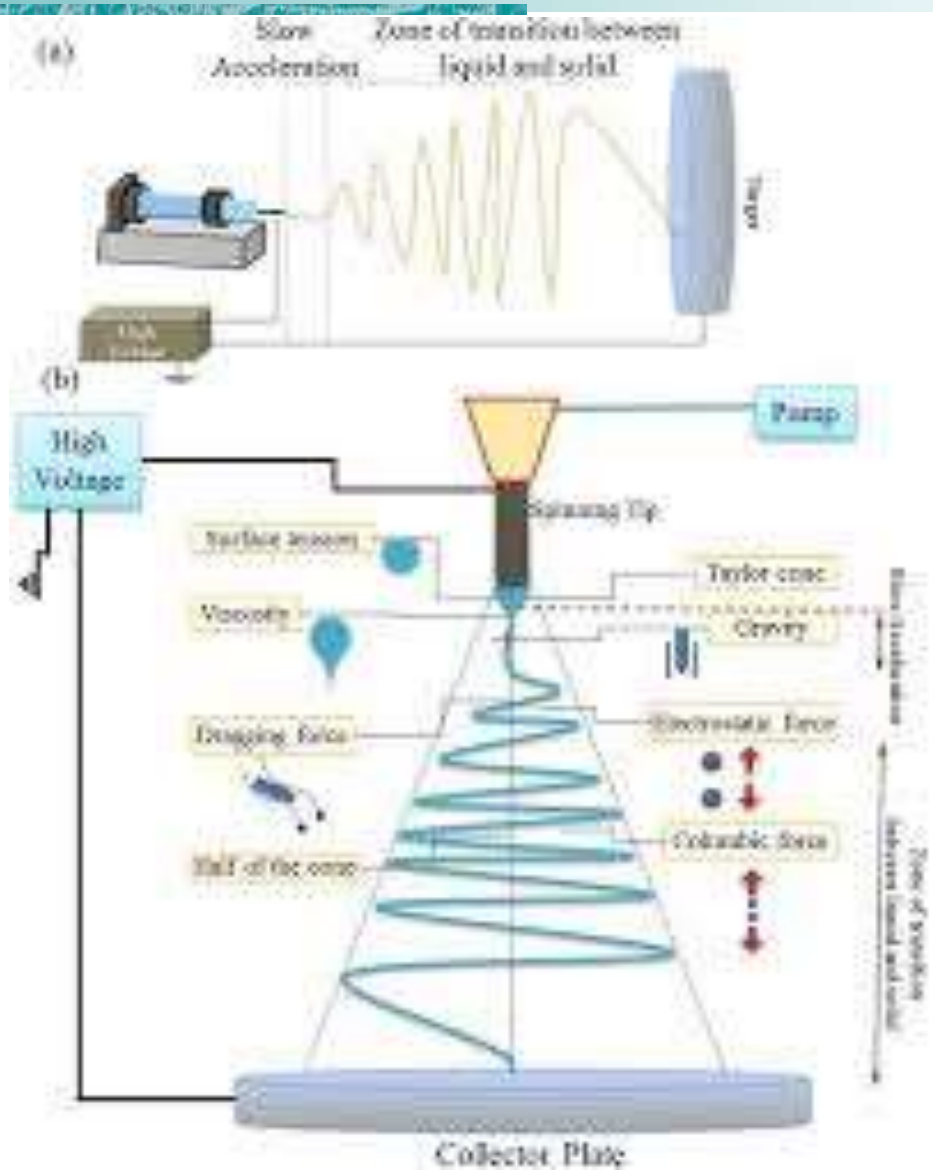
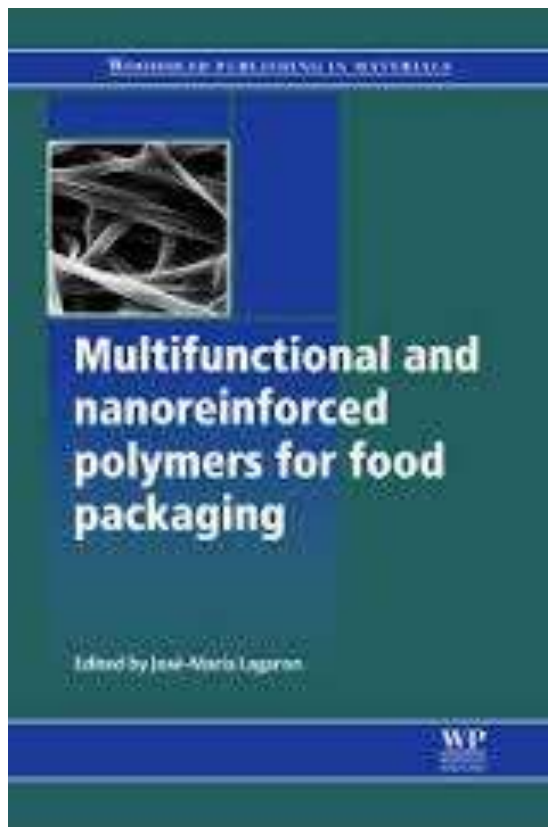
- ❑ **NEW CONCEPTS IN ACTIVE PACKAGING**
 - ❑ Bionanocomposites for active packaging

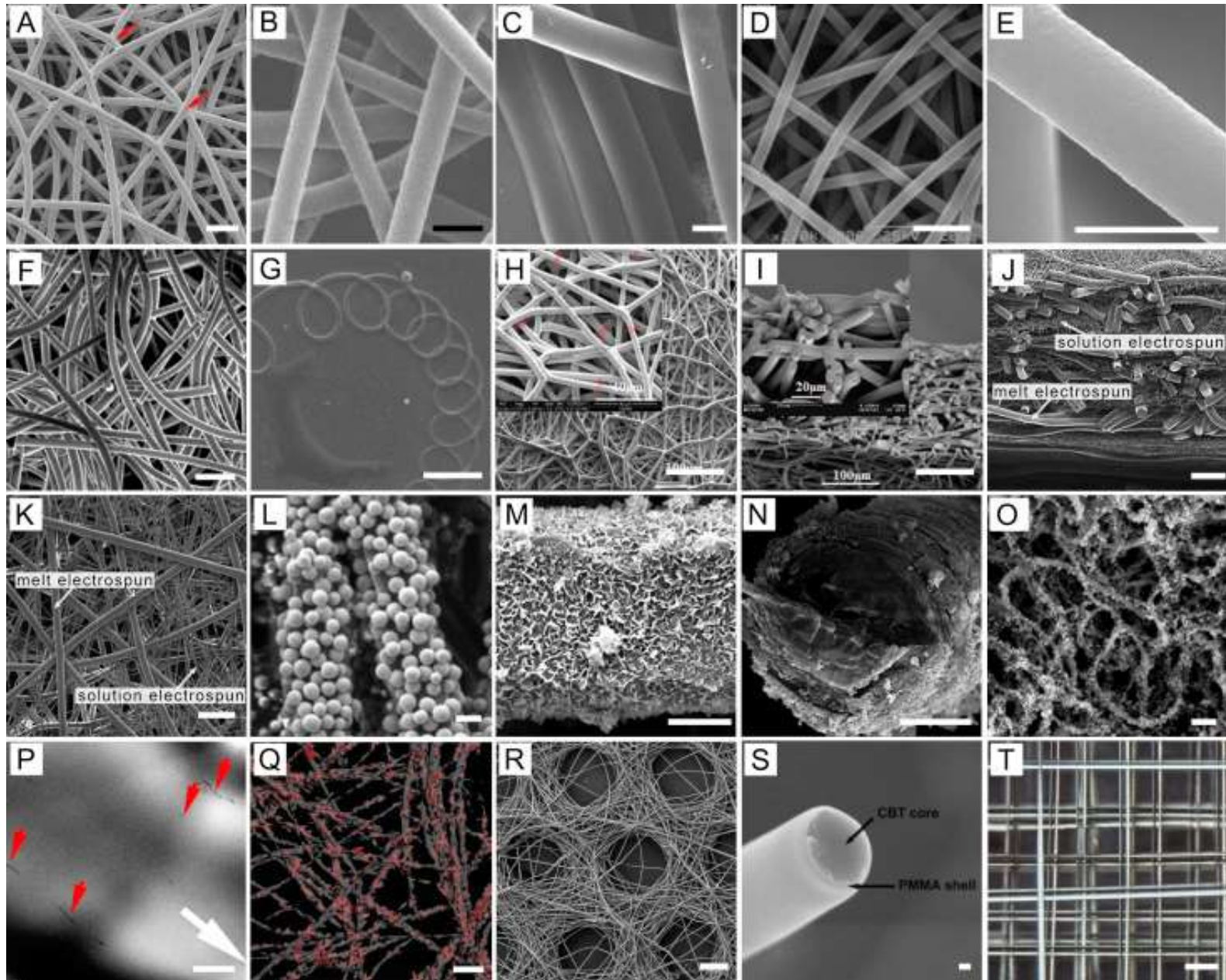


Biolnicia

2010's: From Labscale to Industry

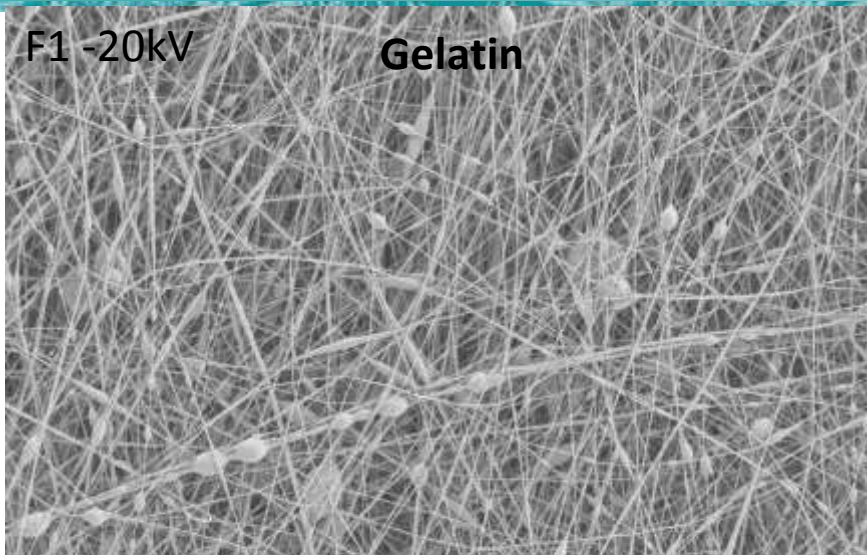






F1 -20kV

Gelatin

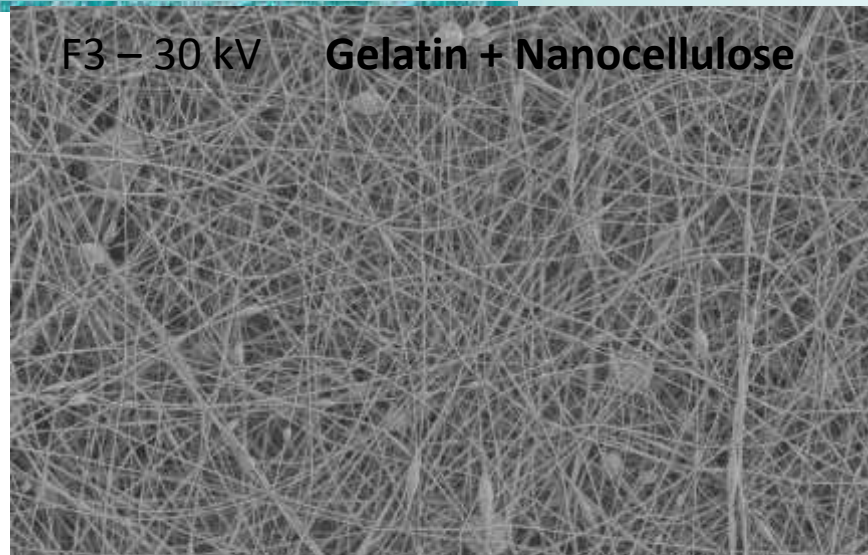


2.0kV 19.0mm x3.00k SE(M) 8/17/10 09:19

10.0um

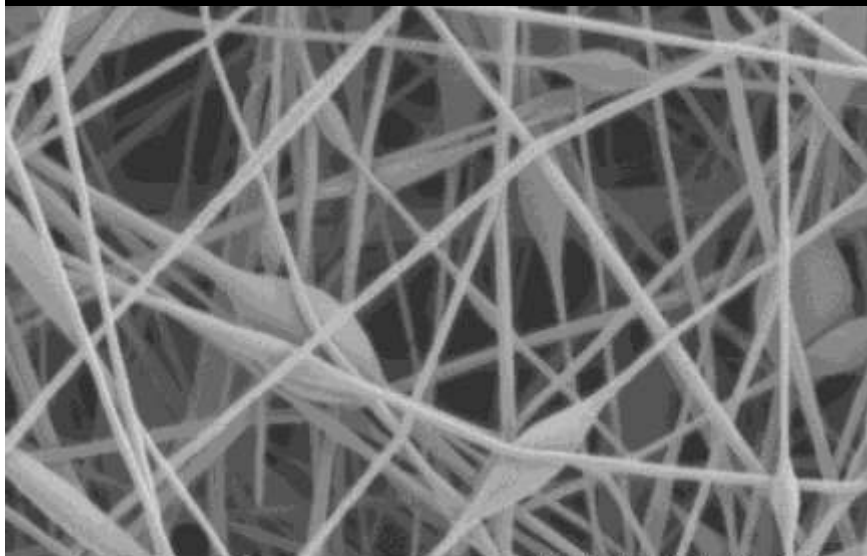
F3 - 30 kV

Gelatin + Nanocellulose



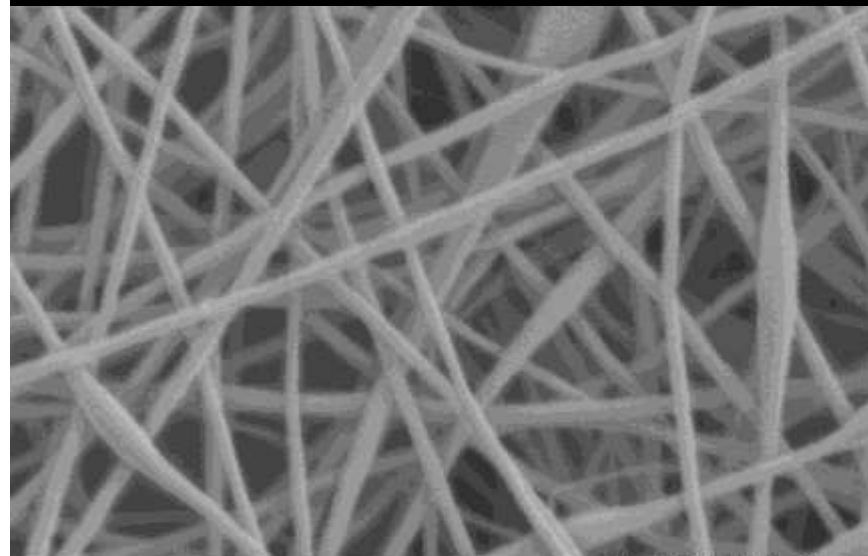
2.0kV 19.3mm x3.00k SE(M) 8/17/10 11:20

10.0um



2.0kV 19.0mm x20.0k SE(M) 8/17/10 09:26

2.00um



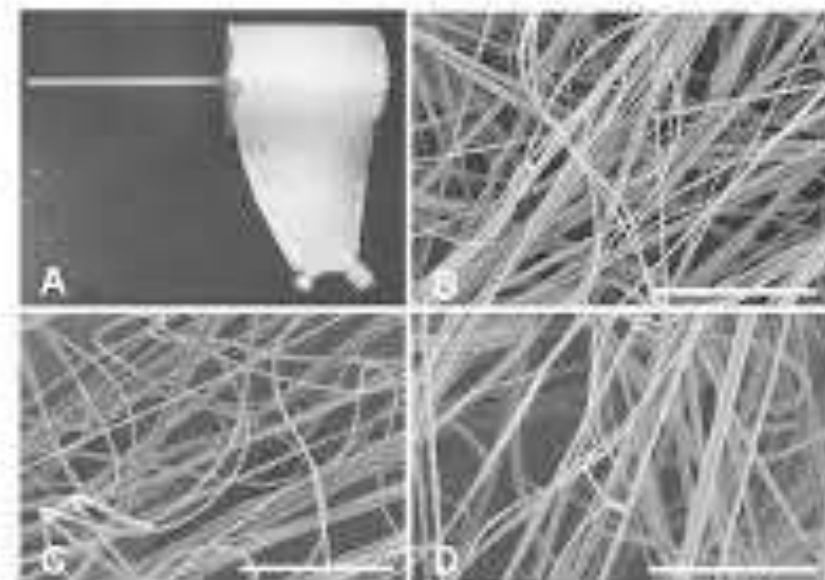
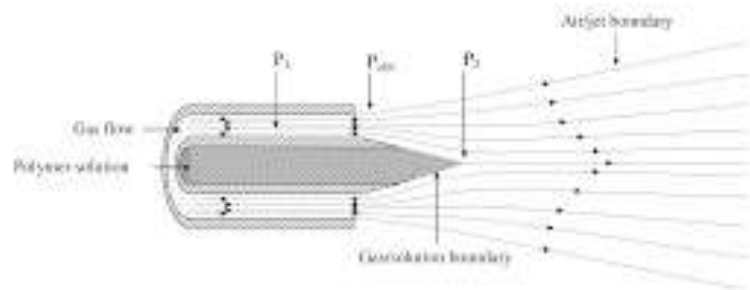
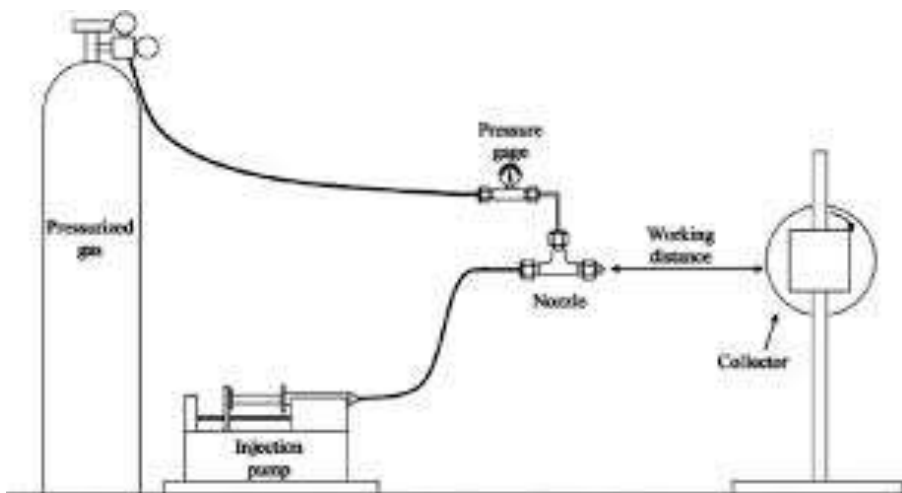
2.0kV 19.3mm x30.0k SE(M) 8/17/10 11:27

1.00um

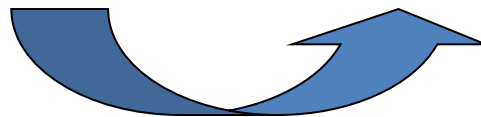
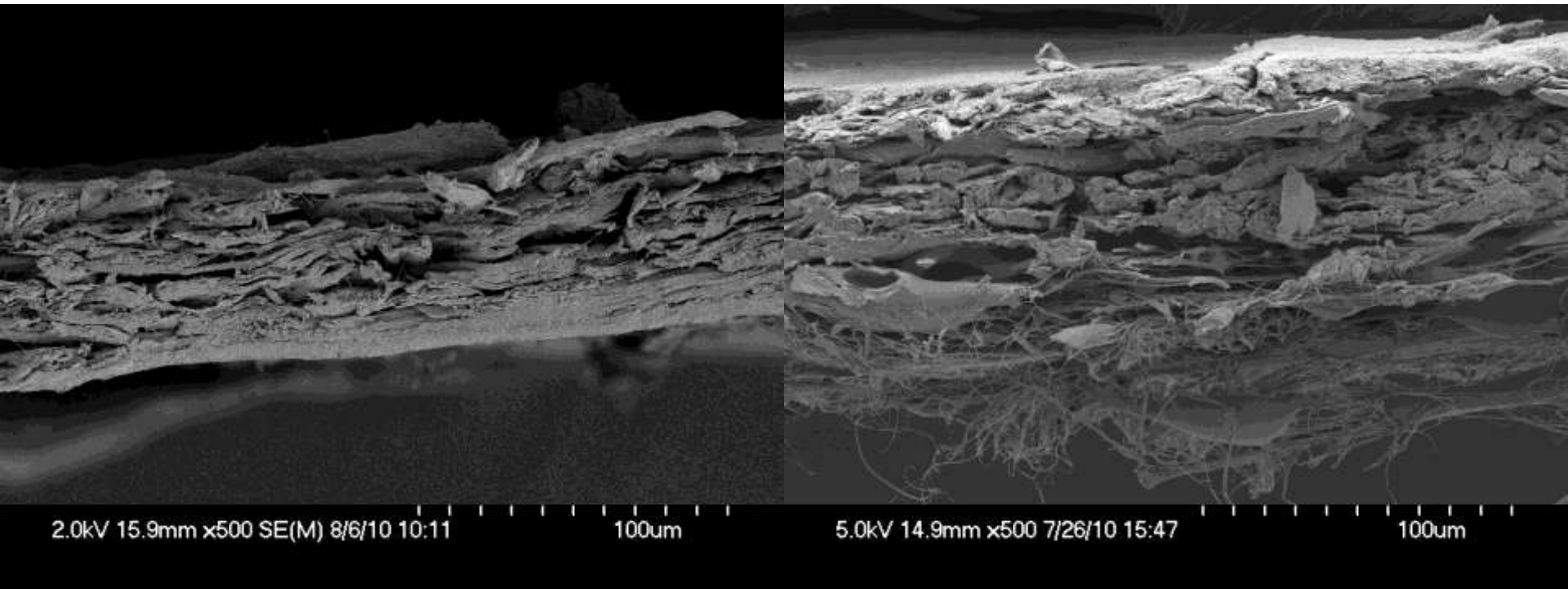
Electrospinning vs Solution Blow spinning

Solution Blow Spinning =

Electro spinning without electricity

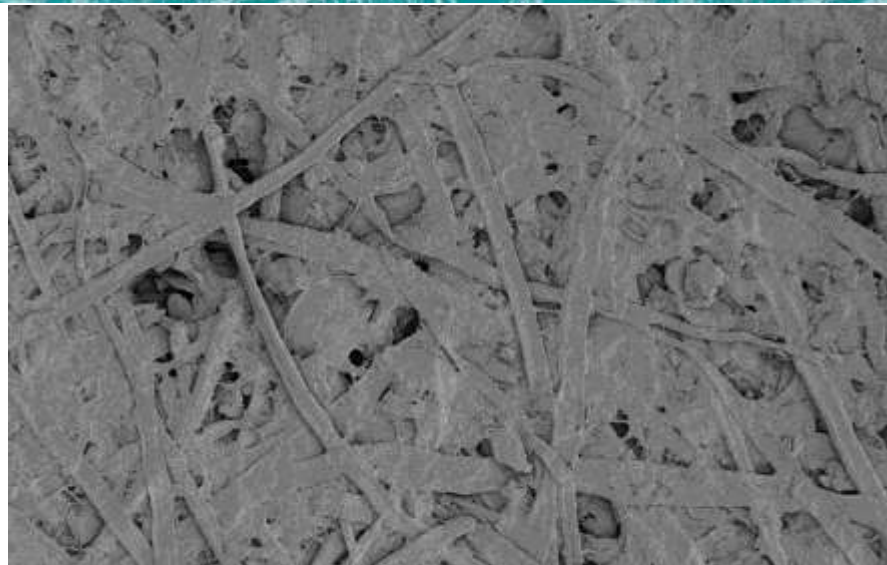


. Pre-trials on paper: sticking possibility

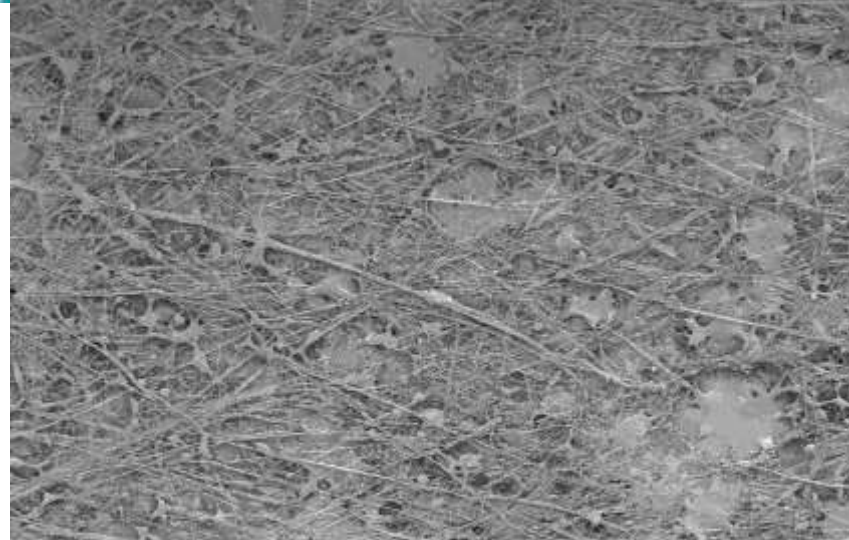


SBS of PLA

Solution Blow spinning

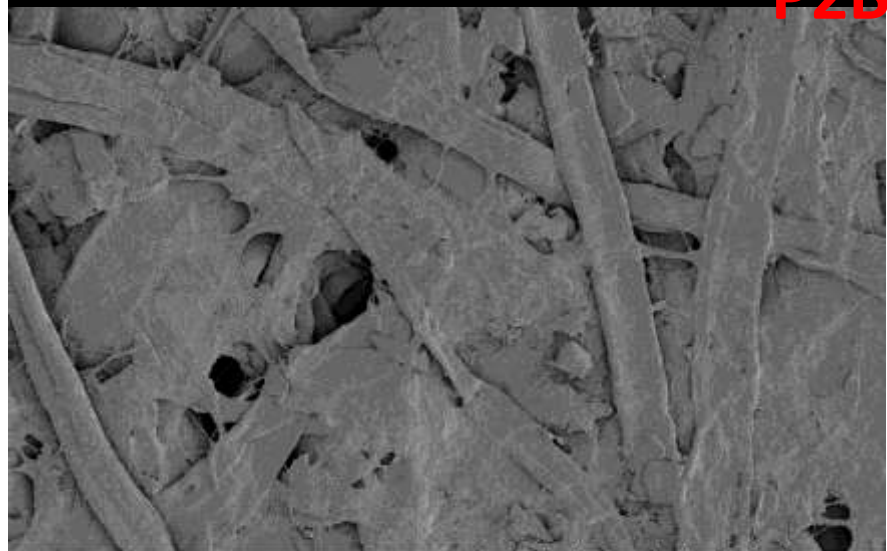


2.0kV 19.5mm x200 SE(M) 8/6/10 10:15 200um

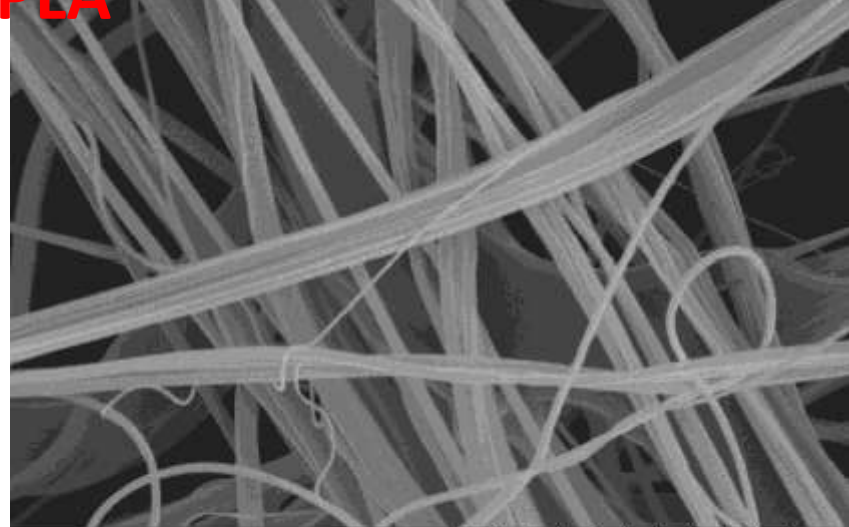


5.0kV 19.3mm x35 7/26/10 16:43 1.00mm

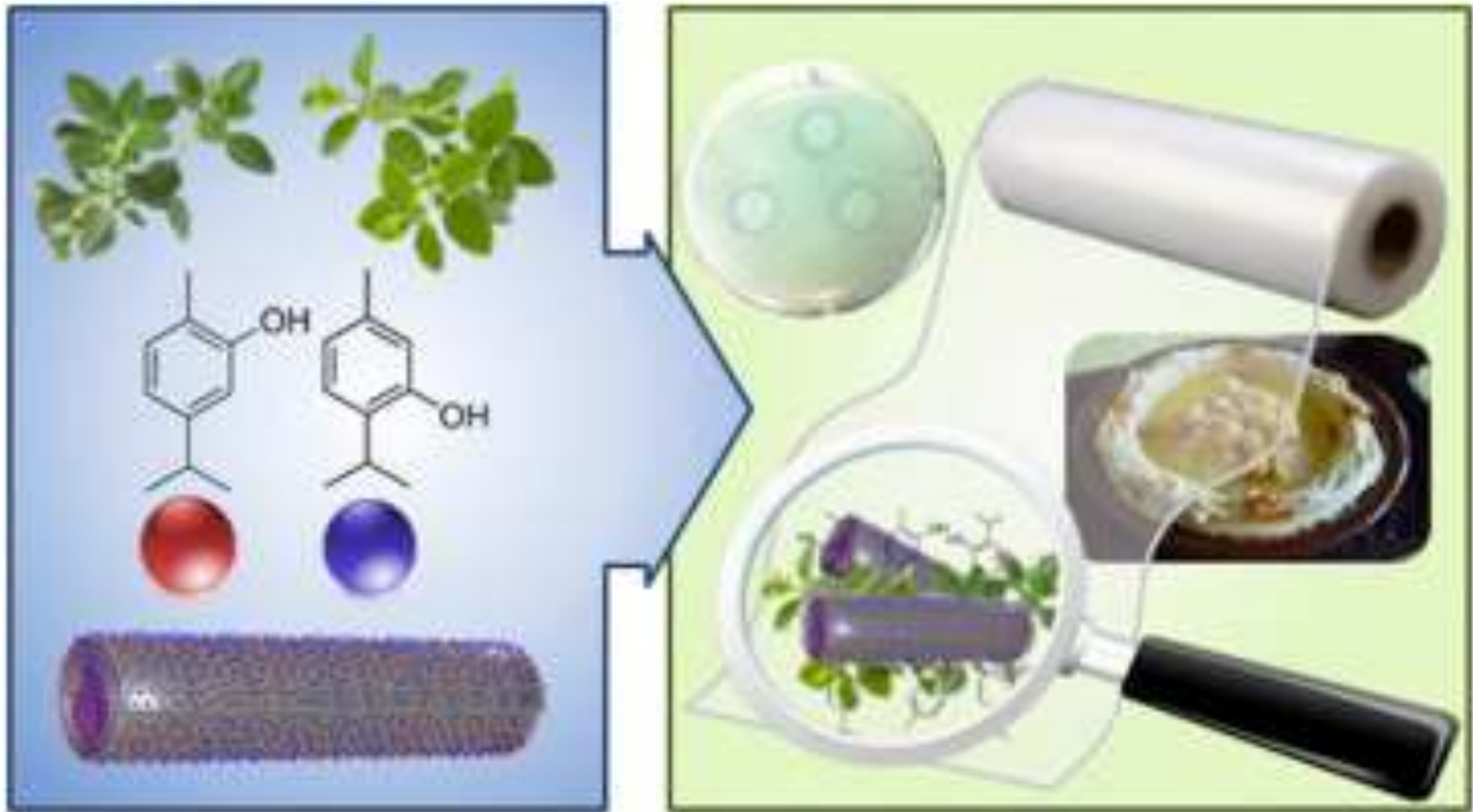
P2B/SBS/PLA



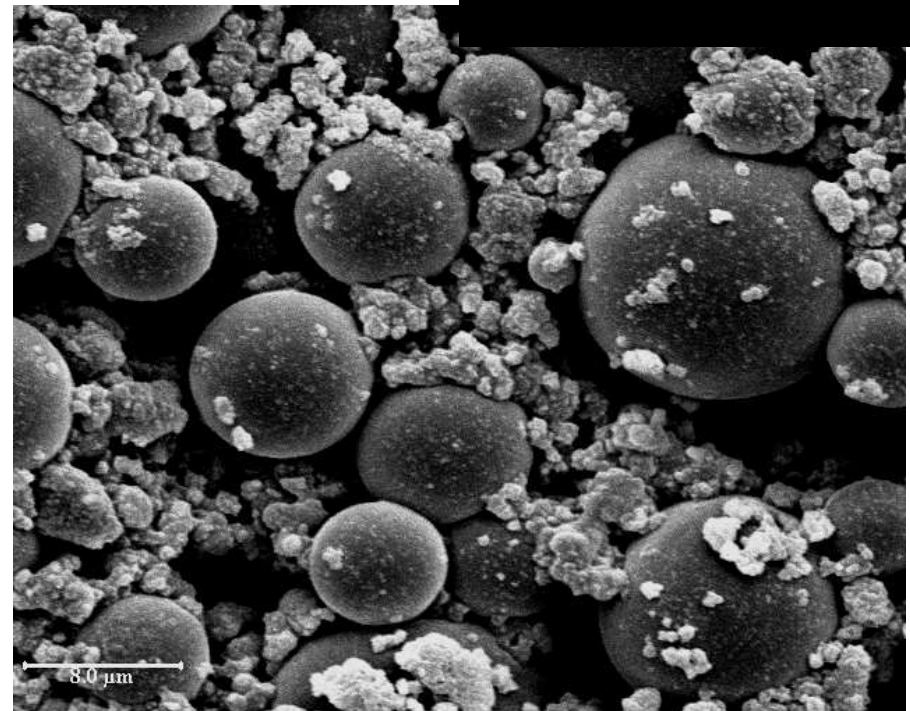
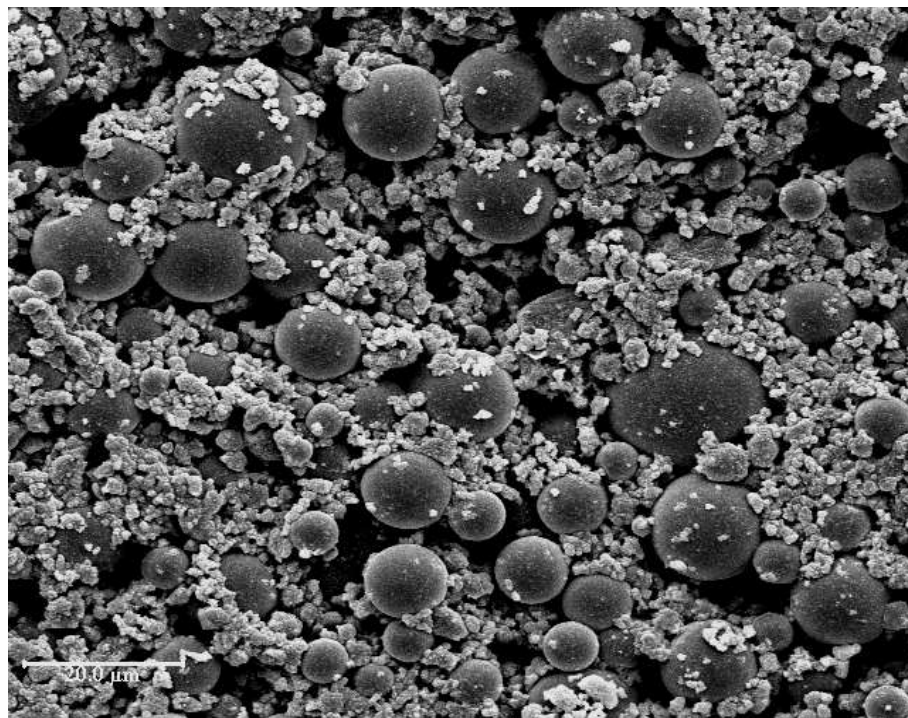
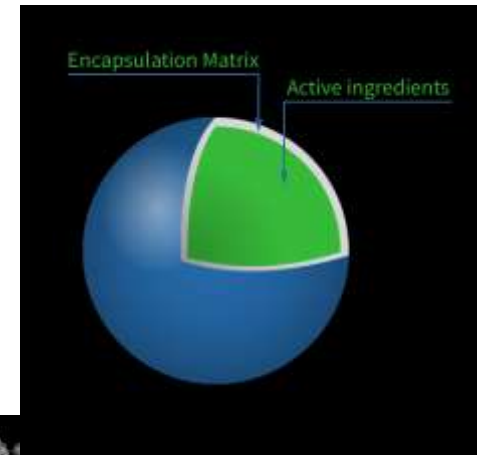
2.0kV 19.4mm x500 SE(M) 8/6/10 10:17 100um



5.0kV 19.3mm x10.0k 7/26/10 16:23 5.00um



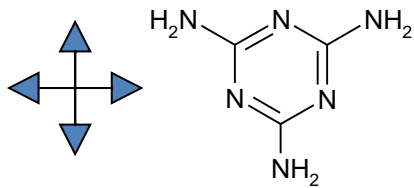
1. Aminoplaste Encapsulation



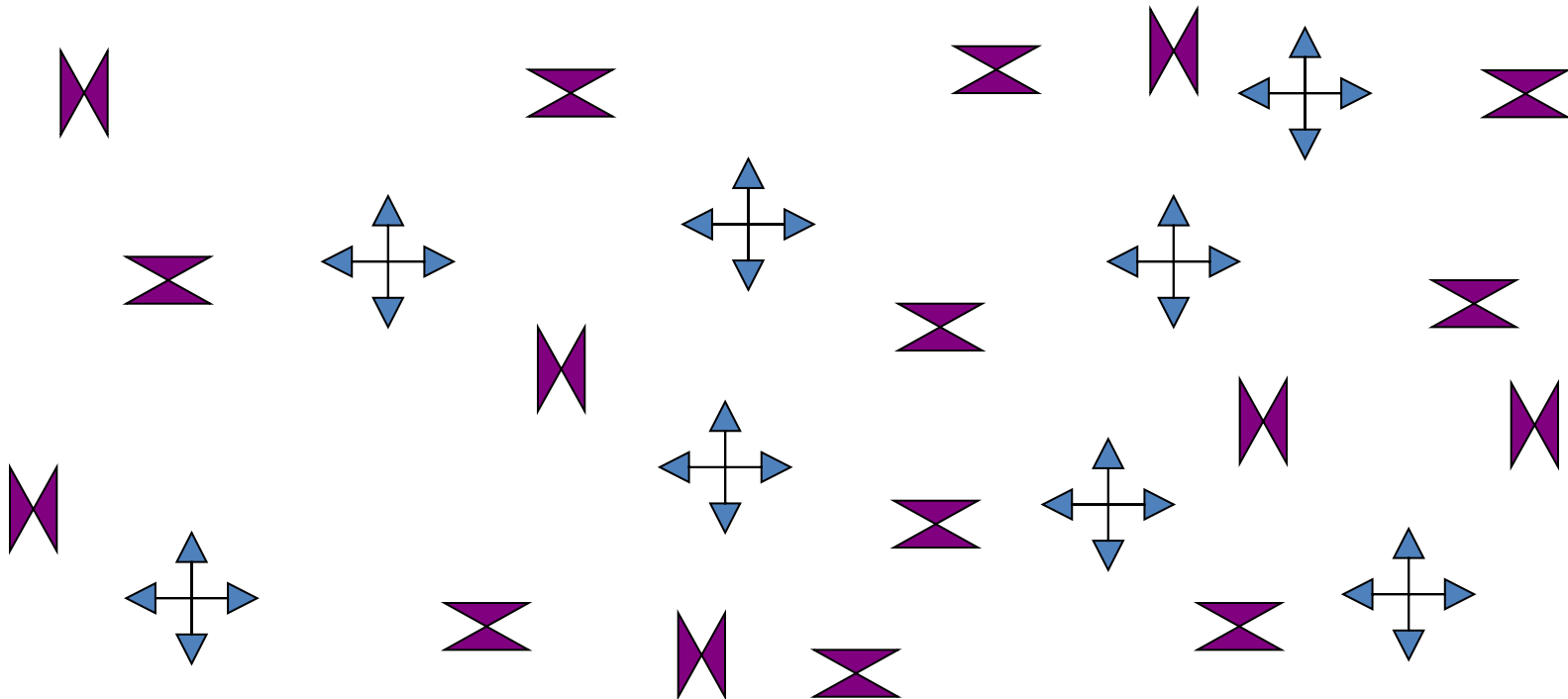
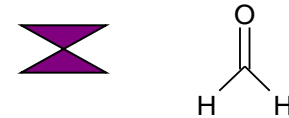
1. Aminoplaste Encapsulation

- Polycondensation non linéaire

Melamine

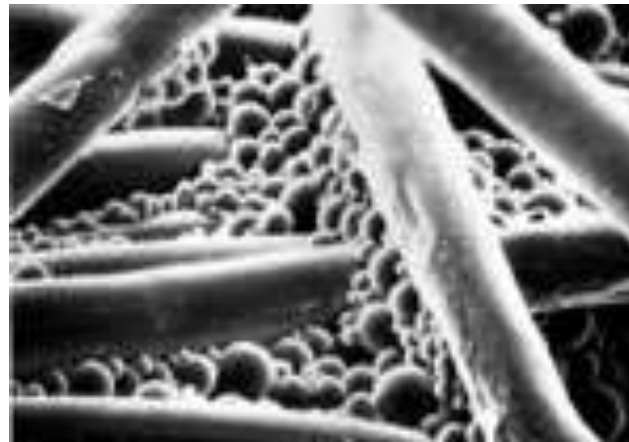
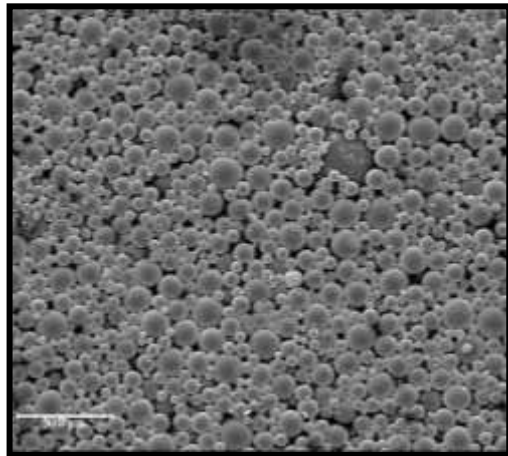
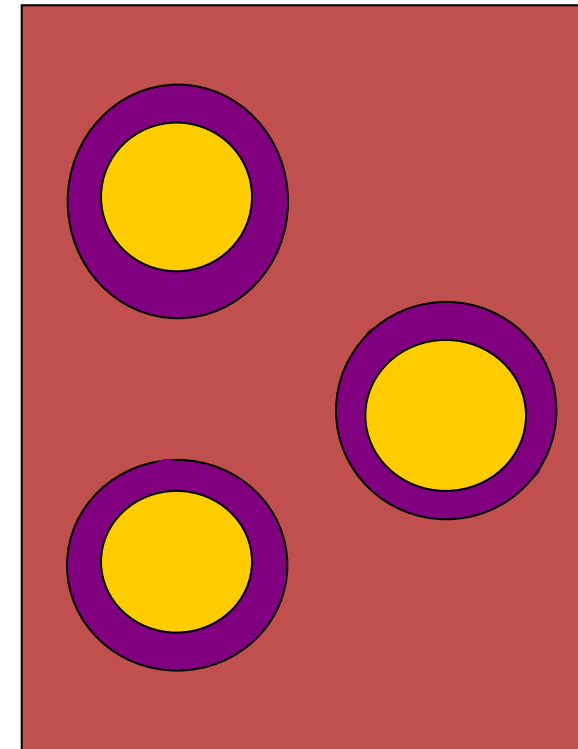
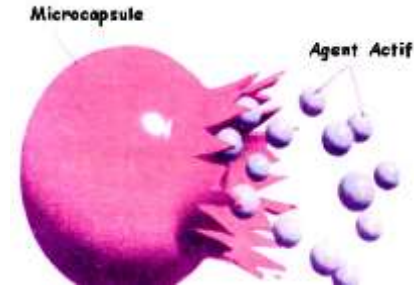
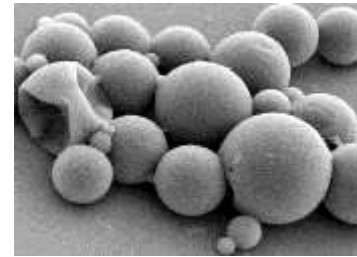


Formaldehyde

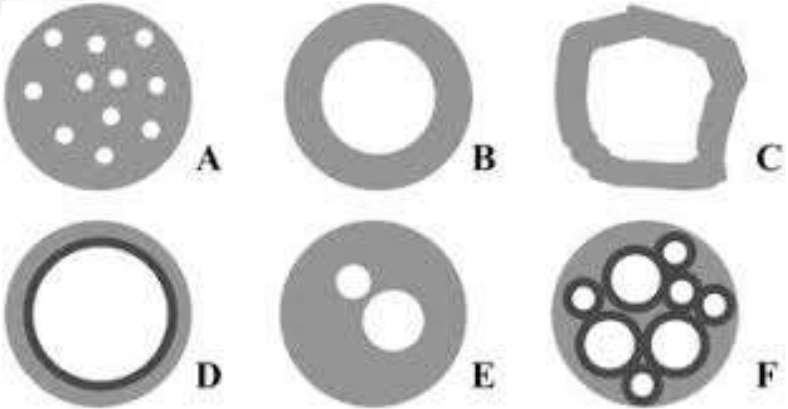
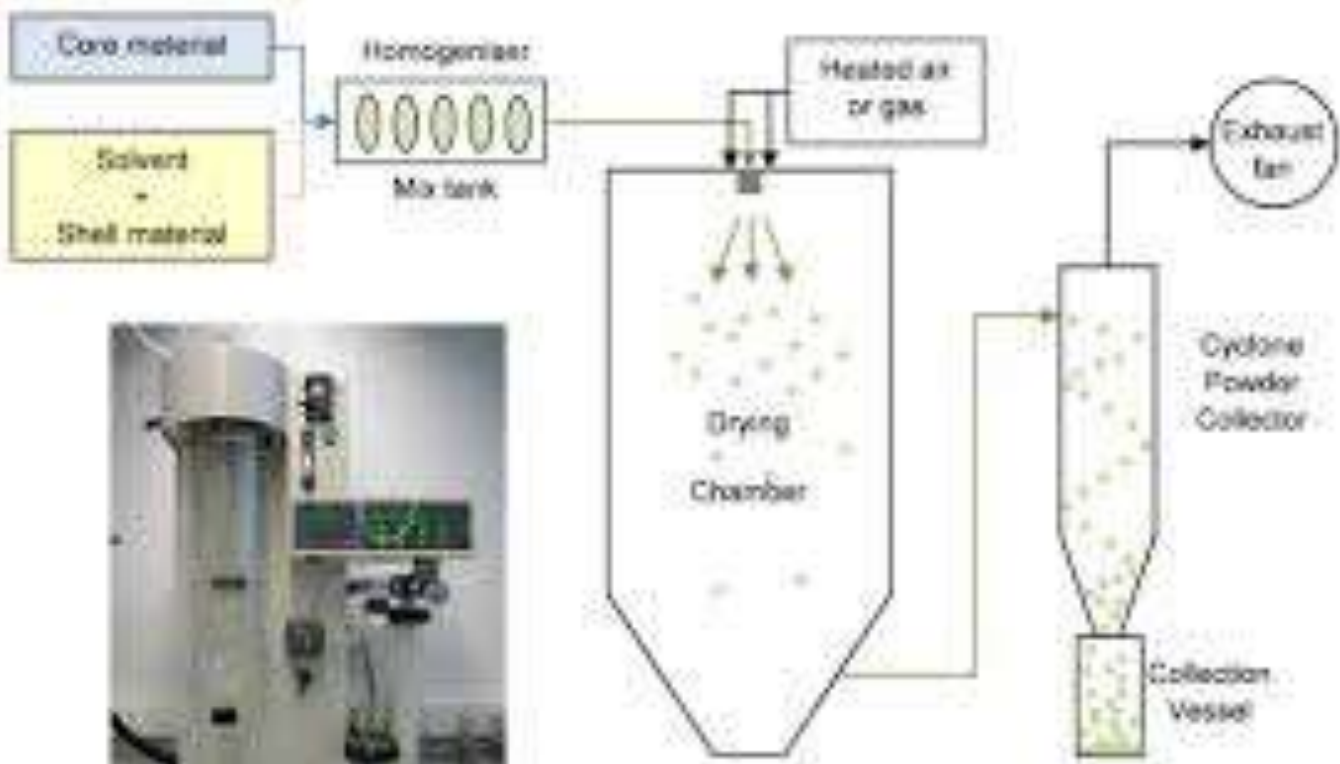


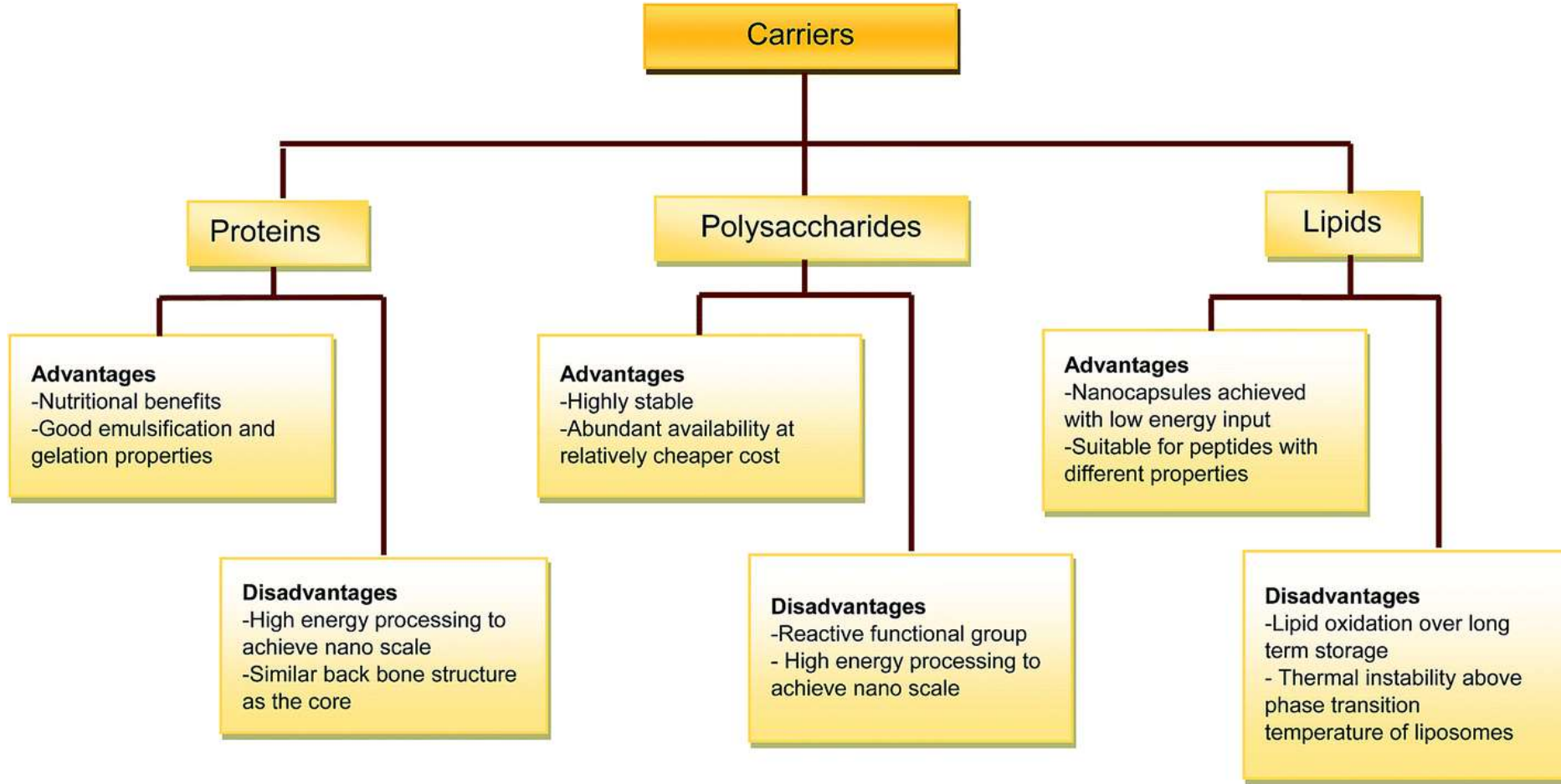
2. Coacervation

- Principe
 - Active agent in polymer emulsion
 - Physico-chemical parameter change
 - Precipitation & coacervation on micels
 - Solidification of shell



Microencapsulation





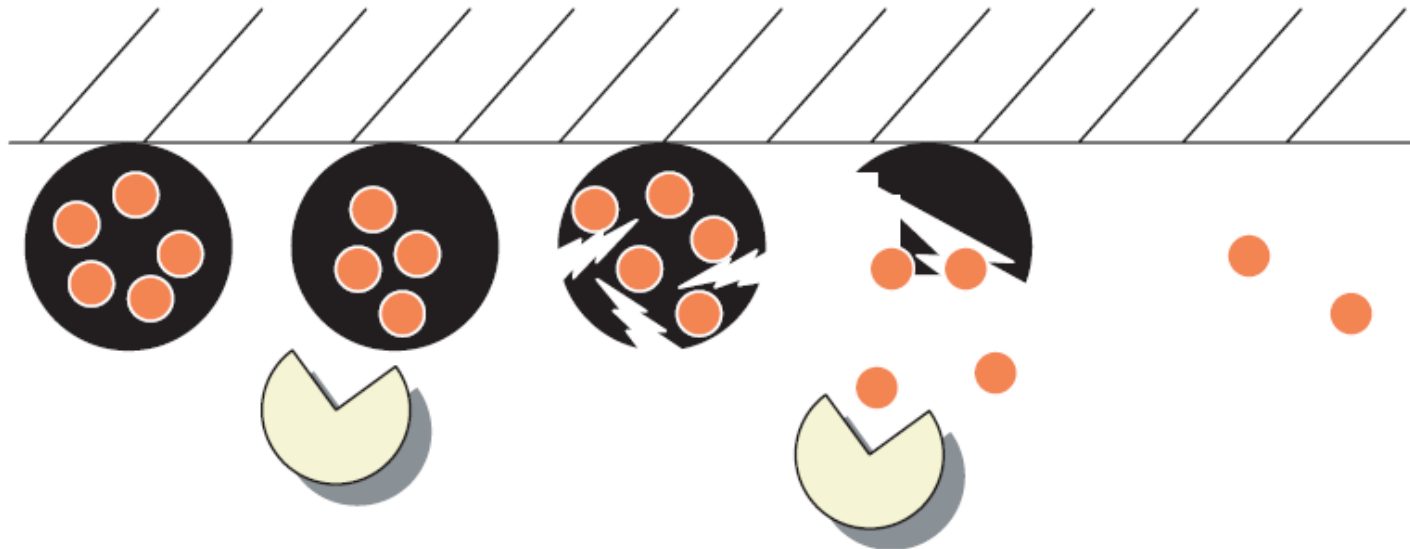


Figure 2. Schematic representation of anti-microbial active-packaging. Micro-organisms hydrolyze starch-based particles, causing release of the antimicrobial lysozyme, resulting in inhibition of microbial growth.

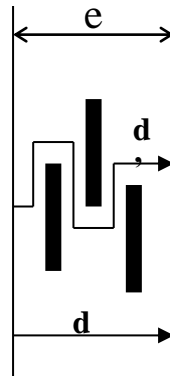
- ❑ **NEW SOLUTIONS IN MATERIALS**
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↪ decrease permeability to gases:

1. ... nanoloads with high ratio Length / thickness
⇒ increase of the tortuosity



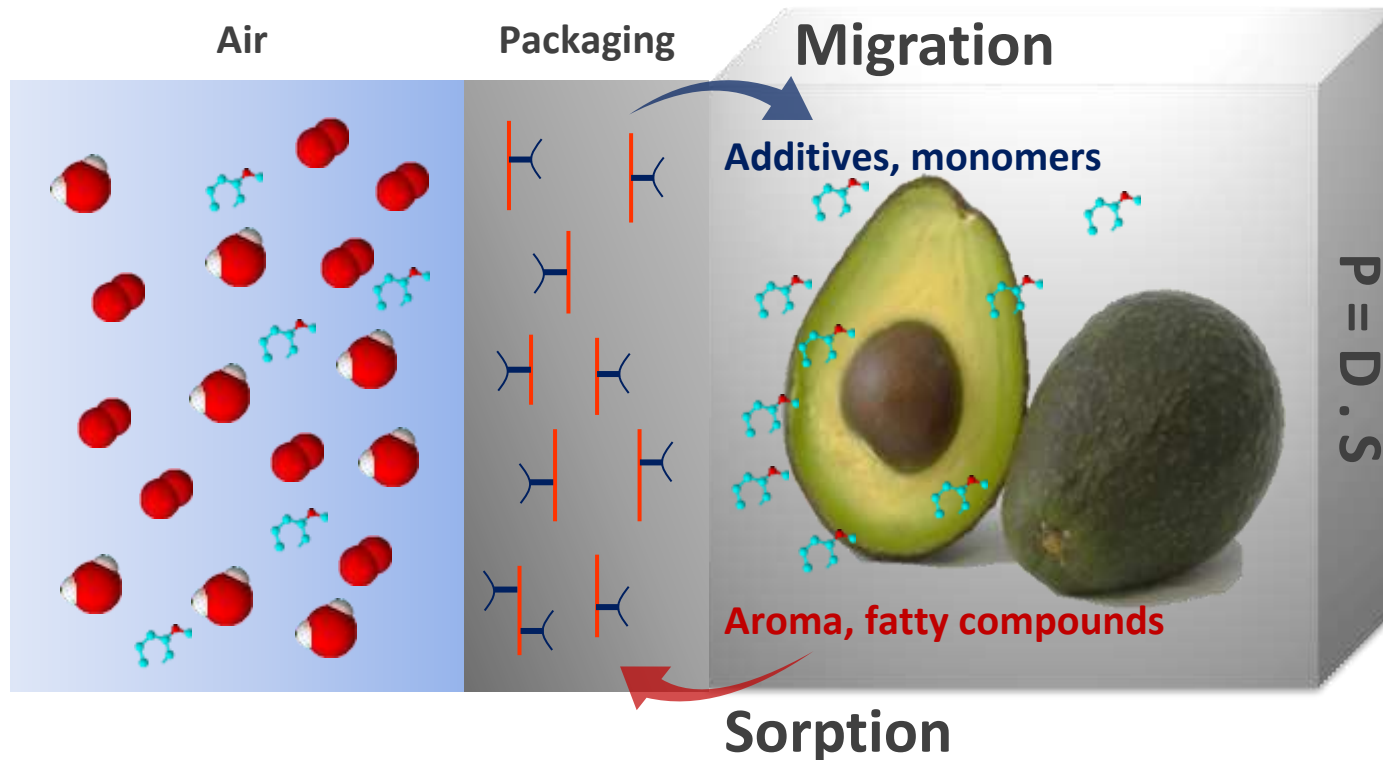
2. ... a perfect dispersion of the nanoloads allowing their orientation (case of exfoliated nanocomposite)

Example: O₂ and CO₂ transmission rates halved compared with standard films

PA 6 or PET + nanoclay :

↪ O₂ barrier : + 50 to 80%

↪ CO₂ barrier : + 60 to 80%



Develop materials with **functional barriers** by the inclusion of **biobased nanoadsorbents (CNC)** in a polymer matrix

Engineering training

Eloi Mortain



Challenges!

- Find a CNC feeding method for ensuring good dispersion in PLA
- Optimize the extruder profile to avoid CNC degradation
- Develop one-step CNC functionalization by *SolReact* & incorporation in PLA

Lab process



Up-scale

LULEÅ
UNIVERSITY
OF TECHNOLOGY
Doctoral summer training

Etzael Espino



GASP (ANR)

AGENCE NATIONALE DE LA RECHERCHE
ANR

High Barrier biobased polymer for packaging

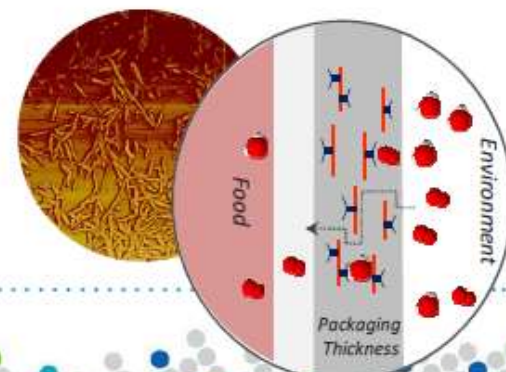
Optmise nanofiller-Bioplastic interface and interphase to develop 100% biobased high barrier to gaz packaging

PI: **GENIAL (INRA) (91)**

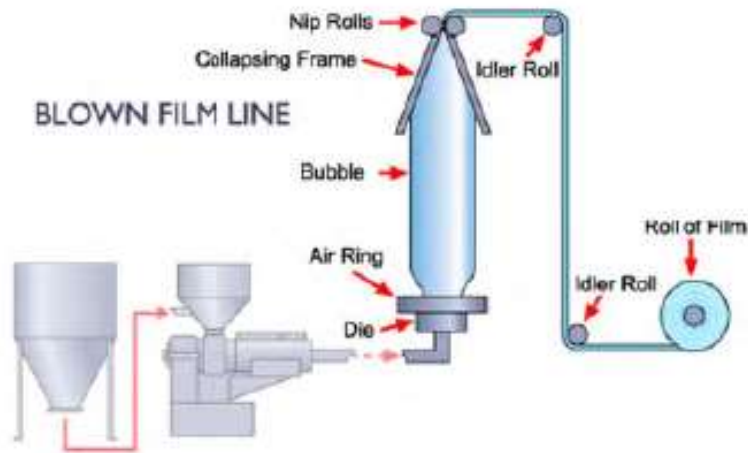
Partners:

CGL Pack (74)	PBS - Université de Rouen (76)
ICMMO – Université Paris Sud (91)	PIMM – CNAM (75)
IMP – Université Lyon 1 (69)	Wipak (59)
LGP2 - INP Grenoble (38)	

Budget: 2 635 082 €
Time: (2016-2020)



Blown-Film Extrusion of PLA and PLA/CNC Composites



www.eastman.com



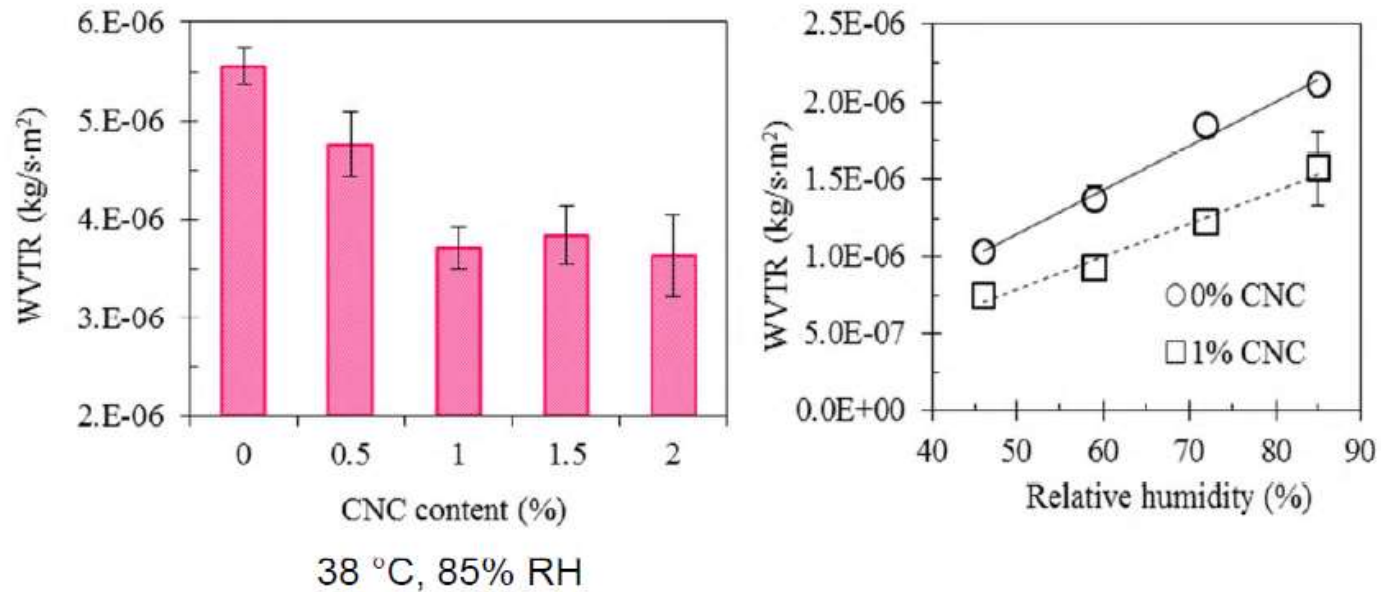
PLA, BUR = 5



2% CNC/PLA, BUR = 5

(Karkhanis et al., Journal of Applied Polymer Science, 2017)

Water Vapor Barrier Performance

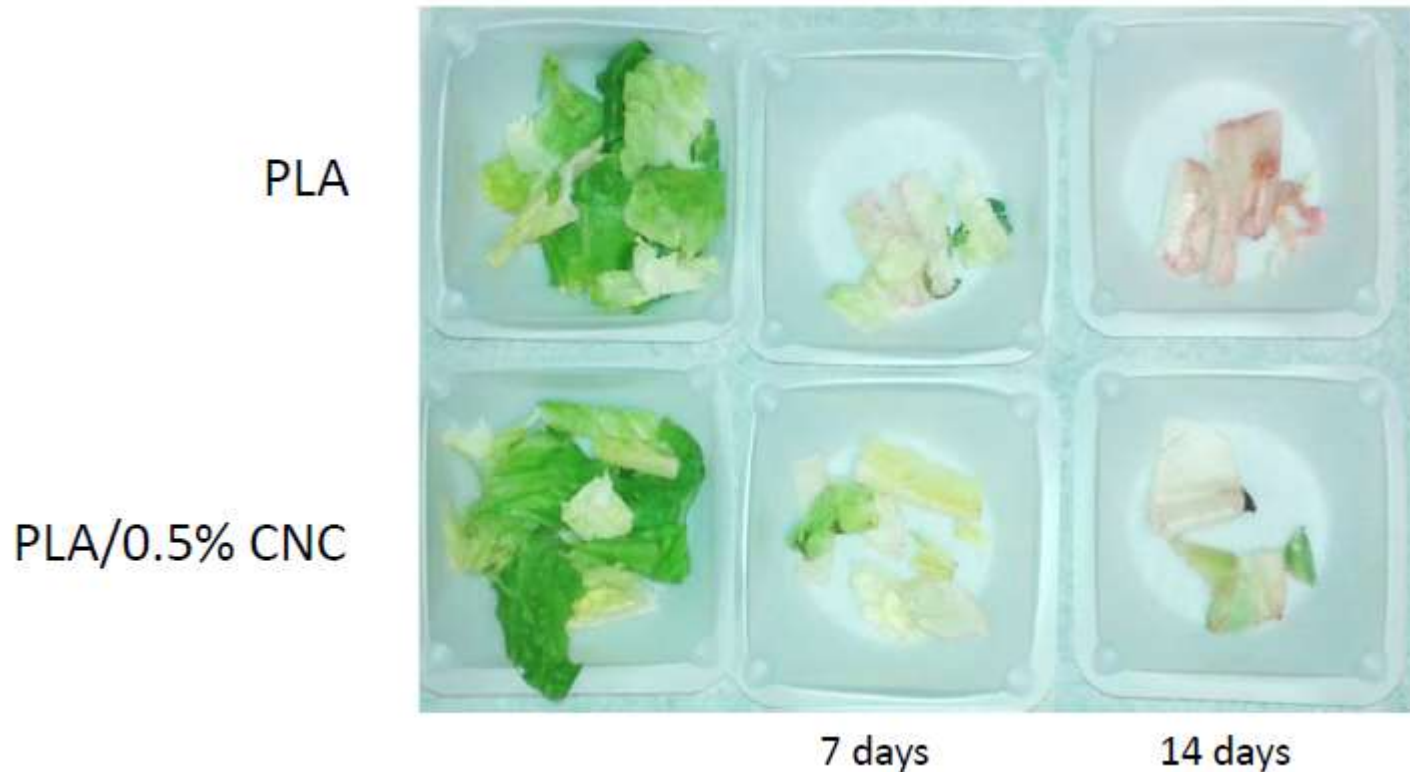


Gas Permeability

Permeability (10 ⁻¹⁸ kg·m/m ² ·s·Pa) at 23°C		
Samples	Oxygen 0% RH	Carbon dioxide at 0% RH
Control	9.69 ± 1.97	27.6 ± 0.28
1% CNC	3.71 ± 0.32	17.9 ± 0.01

N.Stark, Tappi 2017

Discoloration of Cut Lettuce



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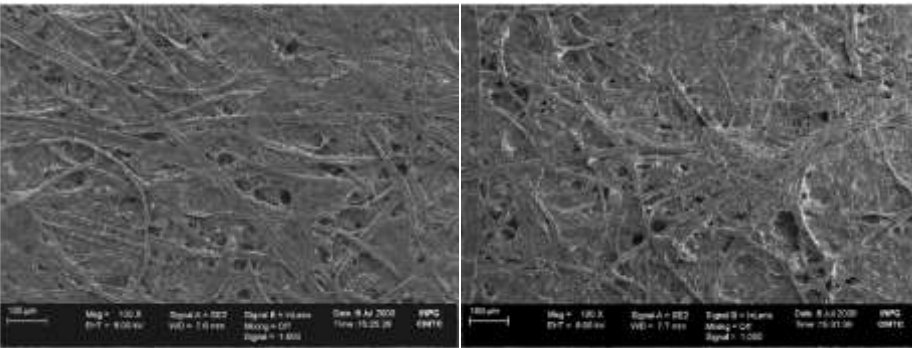
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Conclusions

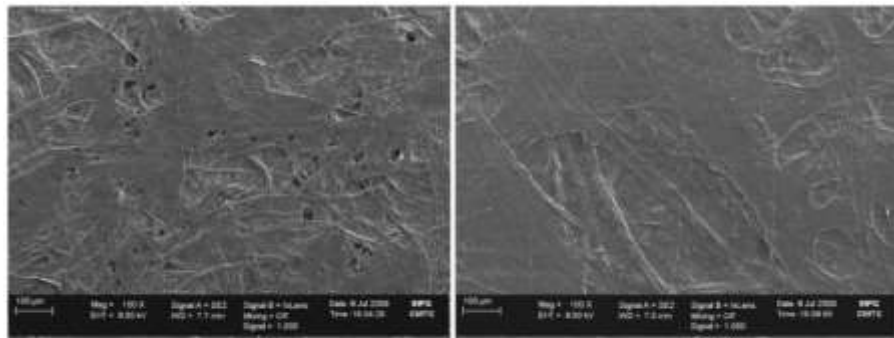
- New active packaging **but not** so many new technologies
 - High **focus on new nanomaterials** or new peptides
 - **Nanocellulose promising biomaterials**
 - What about **scavengers** ?
 - What could be next process / technology ?
-

Next: New technologies?



pulp

A4



MFC1

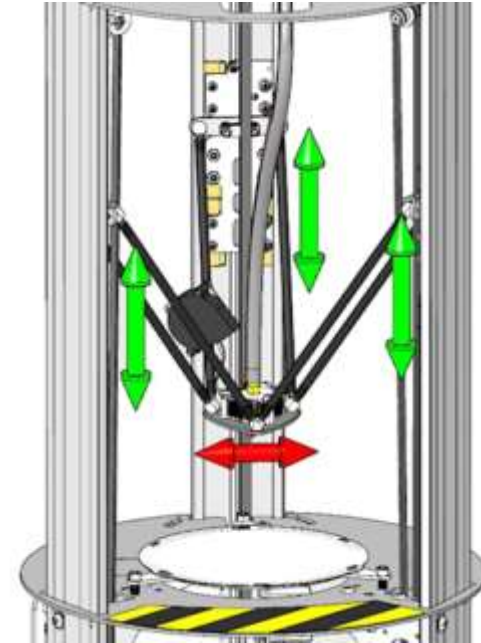
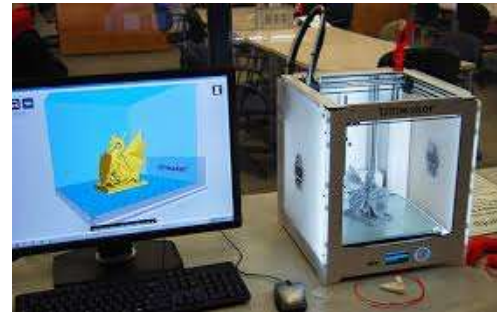
MFC2



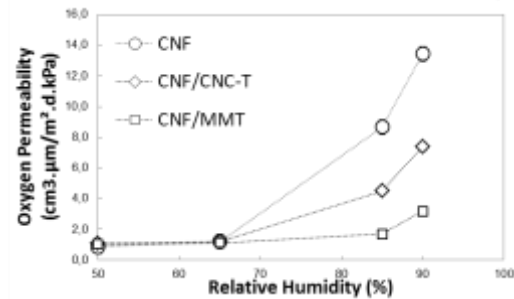
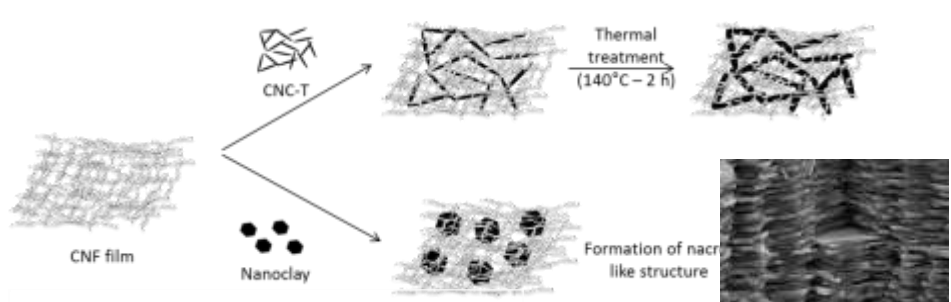
CTP's curtain coater located above Grenoble INP Pagora's paper machine

Next: Additive technologies?

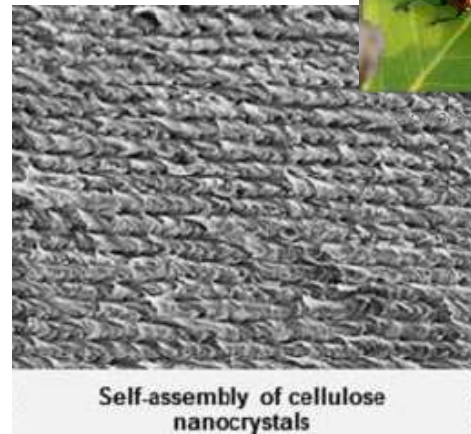
3D Printing ?



Next: Inspired from nature ?

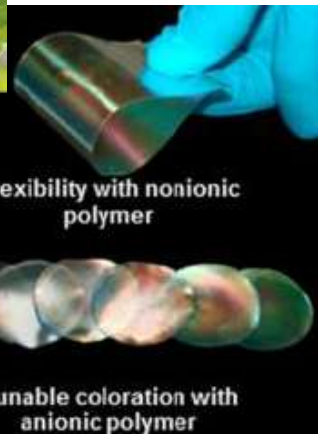
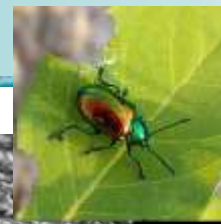


Bardet, Reverdy, Belgacem, Leirset, Syverud, Kristin, Bras, J (2015) Cellulose, 22(2), 1227-1241



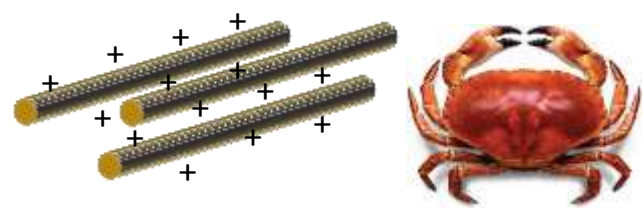
Self-assembly of cellulose nanocrystals

Bardet, R; Belgacem, N; Bras, J,, ACS Applied Materials & Interfaces (2015), 7(7), 4010-4018.



Flexibility with nonionic polymer

Tunable coloration with anionic polymer



Be inspired by Nature



S. Saini, C. Yucel-Falco, M. N. Belgacem, J. Bras Carbohydrate polymers (2015), 135, pp 239-247.

THANK YOU FOR YOUR ATTENTION

Julien.Bras@grenoble-inp.fr

Join us in the LinkedIn group « Nanocellulose Materials »

