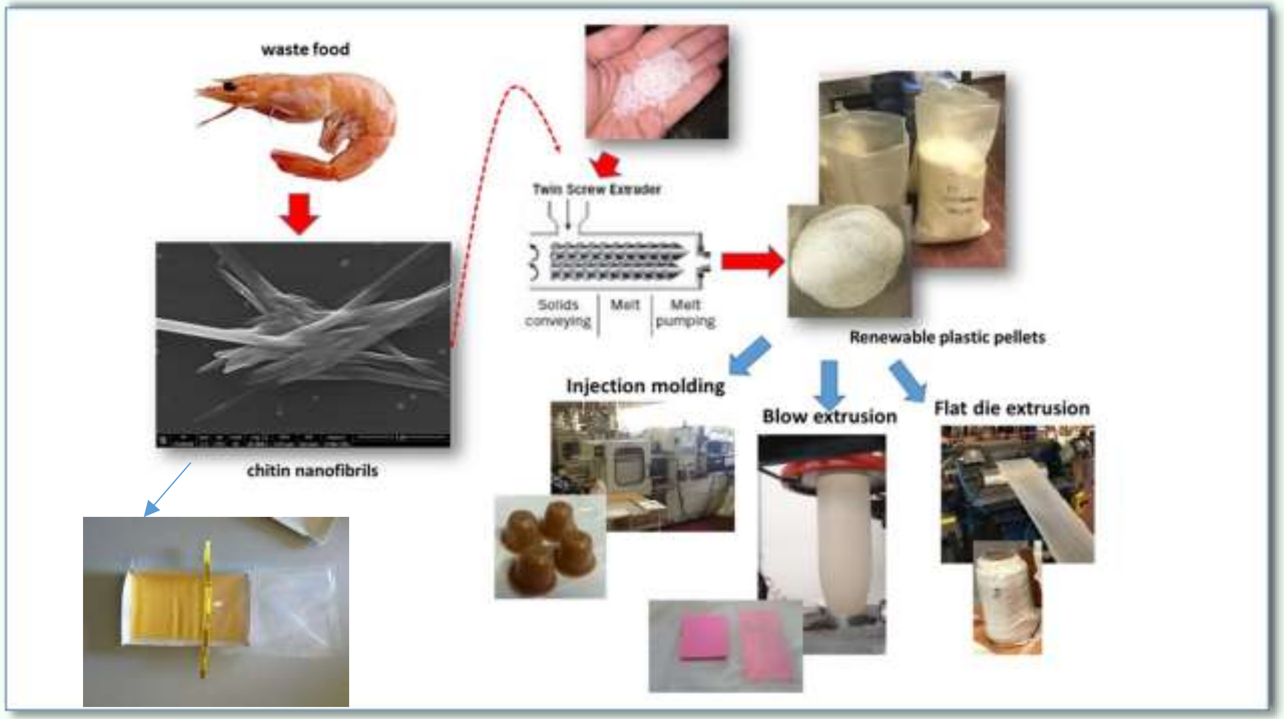
LUCCA, 23 Maggio 2019

LUCENSE

IMBALLAGGI ECO-SOSTENIBILI PER IL CONTATTO ALIMENTARE

Coating e film funzionali e biobased applicati ai prodotti cellulosici: ricerche nel contesto regionale ed europeo


waste food
Twin Screw Extruder
Solids conveying
Melt
Melt pumping
Renewable plastic pellets
chitin nanofibrils
Injection molding
Blow extrusion
Flat die extrusion

Maria Beatrice Coltelli,
Dipartimento di Ingegneria Civile ed Industriale,
Università di Pisa

UNIVERSITÀ DI PISA

Biomateriali: la natura ama i compositi…

ALTA DISPONIBILITA'

BIOMATERIALI POLISSACCARIDICI PER APPLICAZIONI STRUTTURALI

legno: micro-composito di cellulosa in resina a base di lignina

Esoscheletro degli artropodi: micro-composito di fibre di chitina in una matrice proteica (containing also calcium carbonate)

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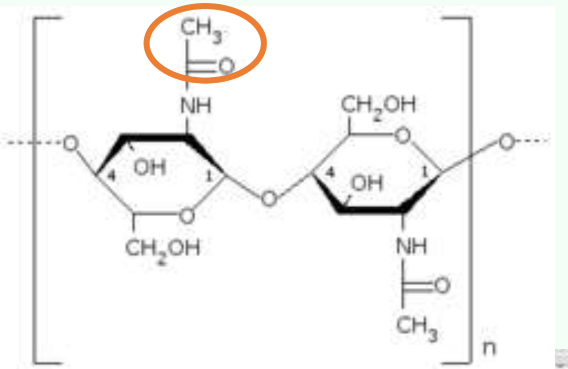
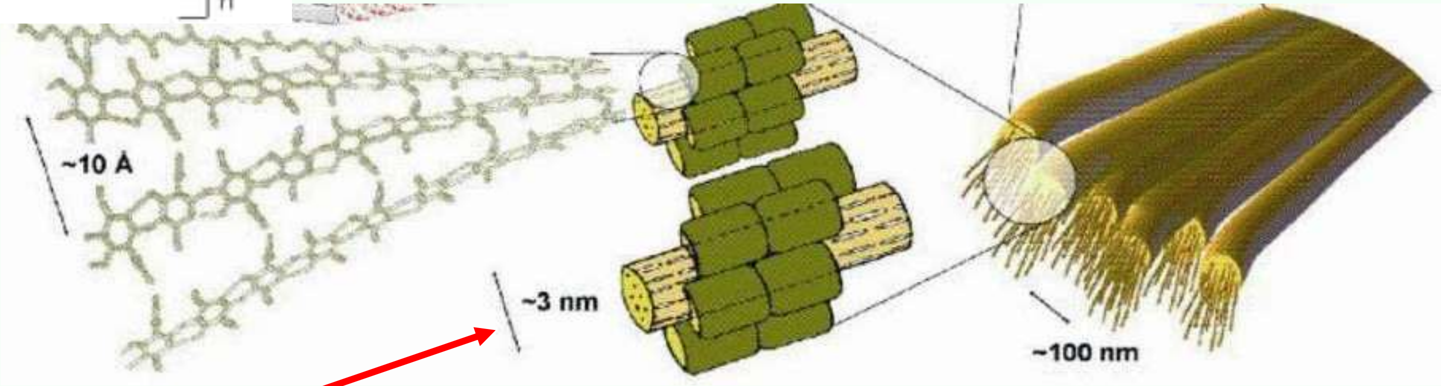
Struttura dell’esoscheletro degli artropodi

IMBALLAGGI ECO-SOSTENIBILI PER IL CONTATTO ALIMENTARE

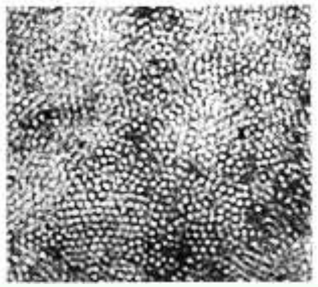
struttura


CH3
O
NH
CH2OH
O
OH
4
1
O
O
4
OH
1
CH2OH
NH
O
CH3
n

Legame ad idrogeno inter-macromolecolare

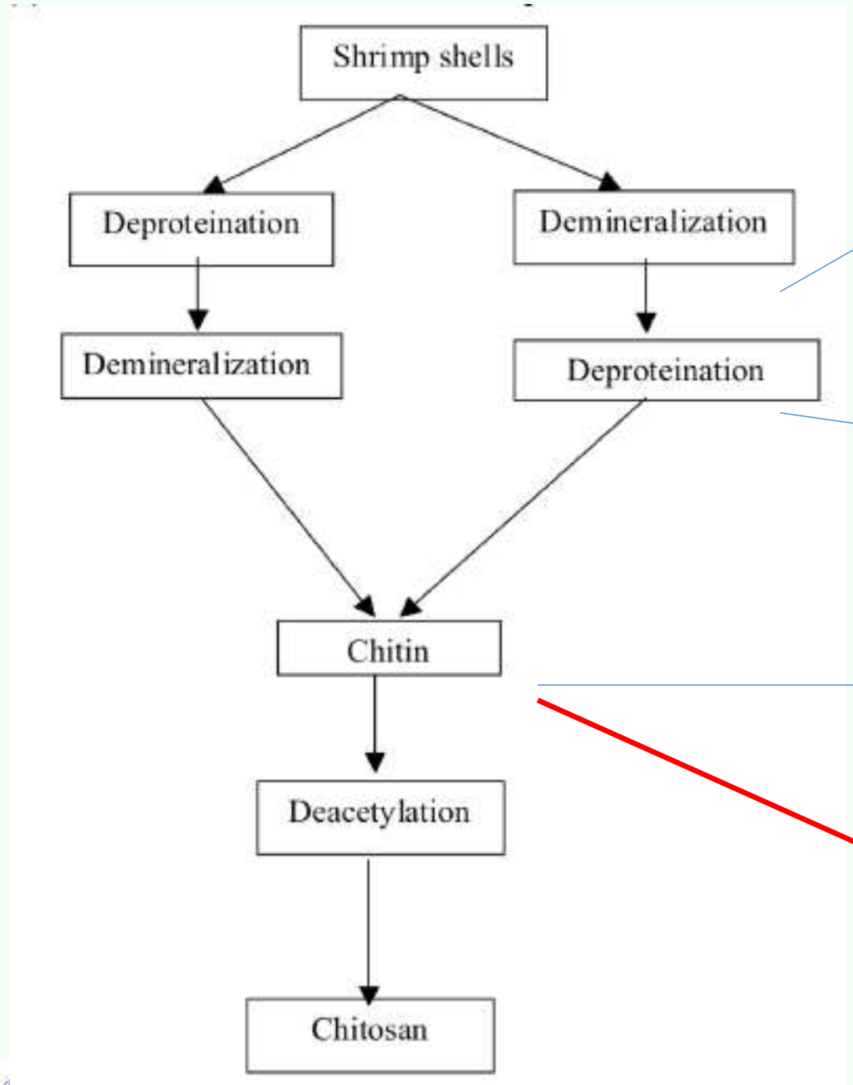

~10 Å
~3 nm
~100 nm

Ogni nano-fibrilla di chitina è composta di circa 20 catene lineari di poli(N-acetil glucosammina)



Il materiale rigido risultante a base di chitina è immerso in una matrice di proteina e carbonato di calcio

PURIFICAZIONE DELLA CHITINA


Shrimp shells
Deproteination
Demineralization
Demineralization
Deproteination
Chitin
Deacetylation
Chitosan

Trattamento acido

Trattamento basico

Chitina e
chitosano:
Disponibilità di 10
miliardi di
tonnellate l’anno

Grado di acetilazione 0,9 e peso
molecolare nell’intervallo $1\text{-}2,5*10^{6}$

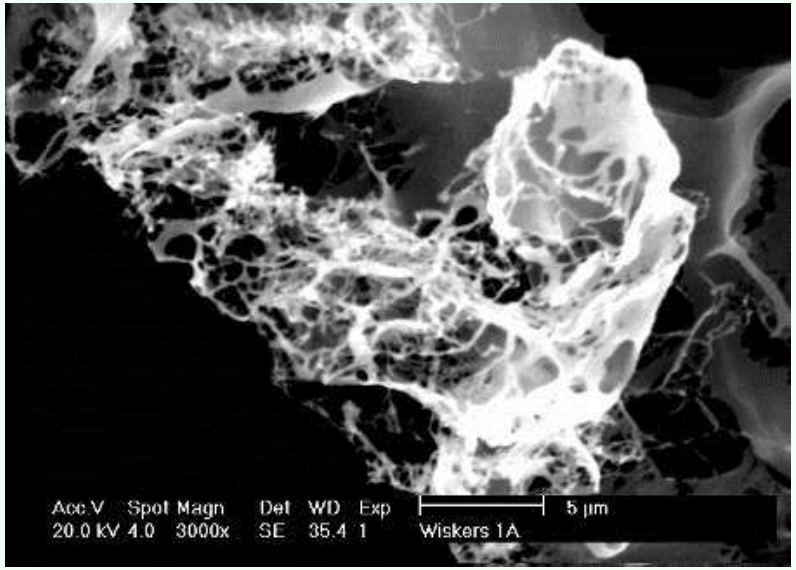
MNV Ravi Kumar, React. Funct. Polym, 46, 2000, 1-27

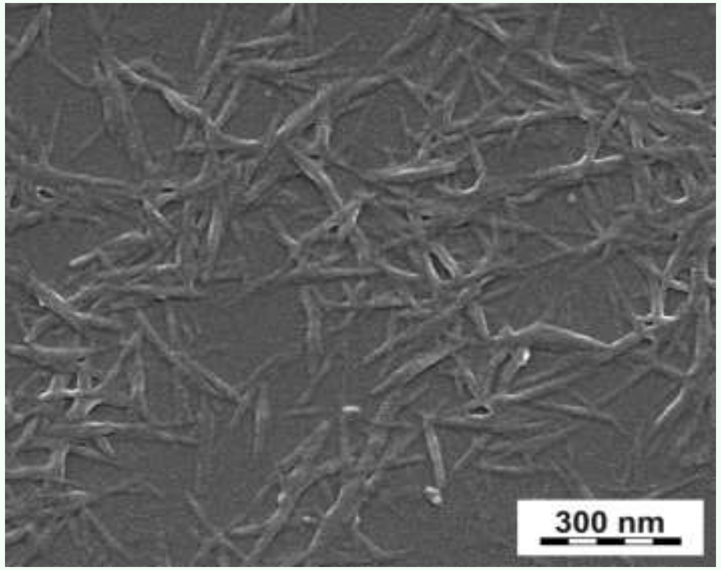
microfibre

N. Van Toan, The Open Biomaterials Journal, 1, 2009

Le microfibre contengono nano-fibre cristalline. E’ possibile produrre nano-fibrille di chitina per trattamento chimico delle microfibre

L’impianto di MAVI SUD presso Aprilia (Roma), ha brevettato questo processo e lo ha applicato principalmente a chitina proveniente da rifiuti di crostacei proveniente dai paesi baltici. Le nanofibrille di chitina sono disponibili in sospensione acquosa diluita per applicazioni cosmetiche





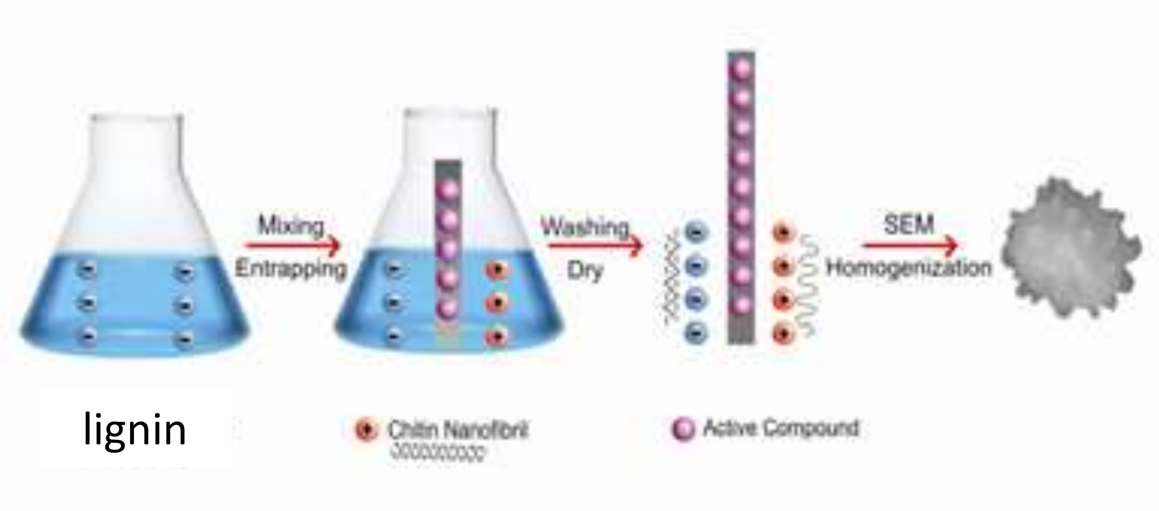
P. Morganti, in Nanofabrication using nanomaterials, Ch 1

Lunghe 300 nm e larghe 10 nm

PROPRIETÀ DELLE NANOFIBRILLE DI CHITINA (CN)

- Proprietà anti-microbiche baste sulla capacità di stimolare le difese immunitarie delle cellule
- Proprietà rigenerative per la pelle dimostrate sulle ustioni della pelle

- Possono essere usate come carrier, sfruttando la capacità di coordinarsi con nanoparticelle caricate negativamente, intrappolando così molecole funzionali


Mixing
Entrapping
Washing
Dry
SEM
Homogenization
lignin
Chitin Nanofibril
Active Compound

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Applicazione di sospensioni di CN sul cartoncino

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IMBALLAGGI ECO-SOSTENIBILI PER IL CONTATTO ALIMENTARE

INNOCARTOPACK, cassa di Risparmio di Lucca

Components

Chitin dispersion

plasticizer

Chitosan powder

Acidic solution

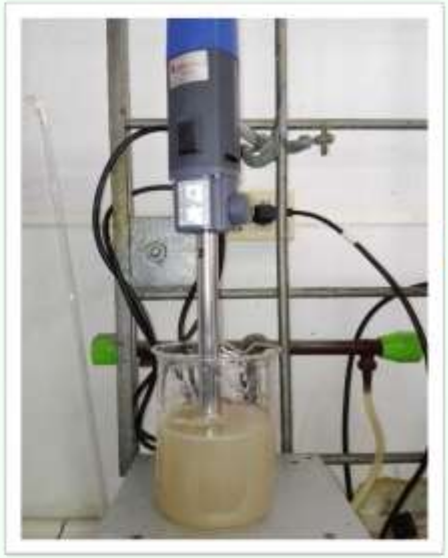


Fig. 1 Mixing process

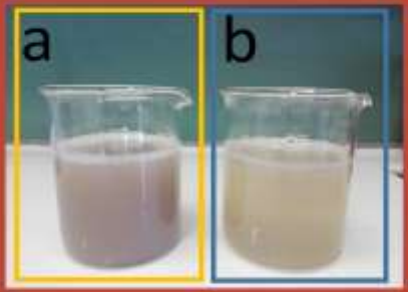
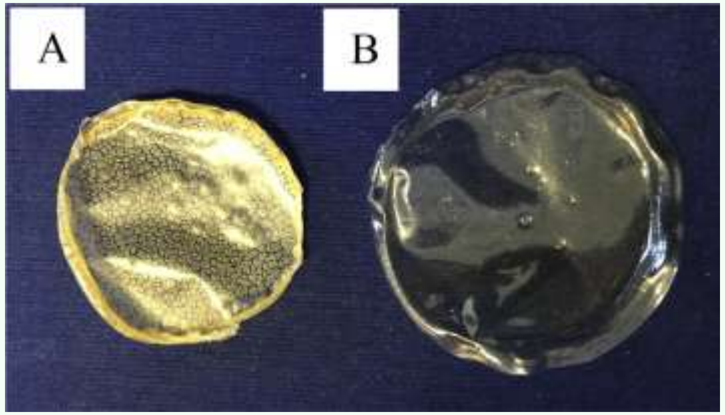


Fig. 2 Chitosan-chitin mixtures. a) mainly chitin b) mainly chitosan



Preparazione di soluzioni lievemente acide contenenti CN e chitosano per trattare cartoncini vergini o riciclati

a → principalmente CN
b → principalmente chitosano

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Mechanical tests

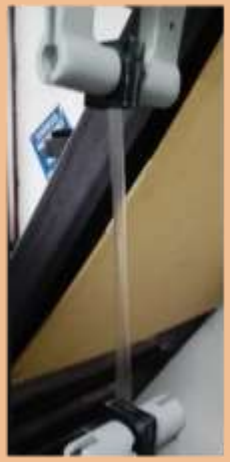
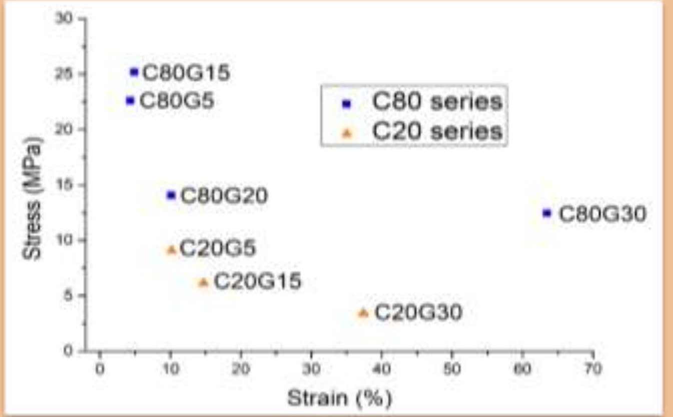


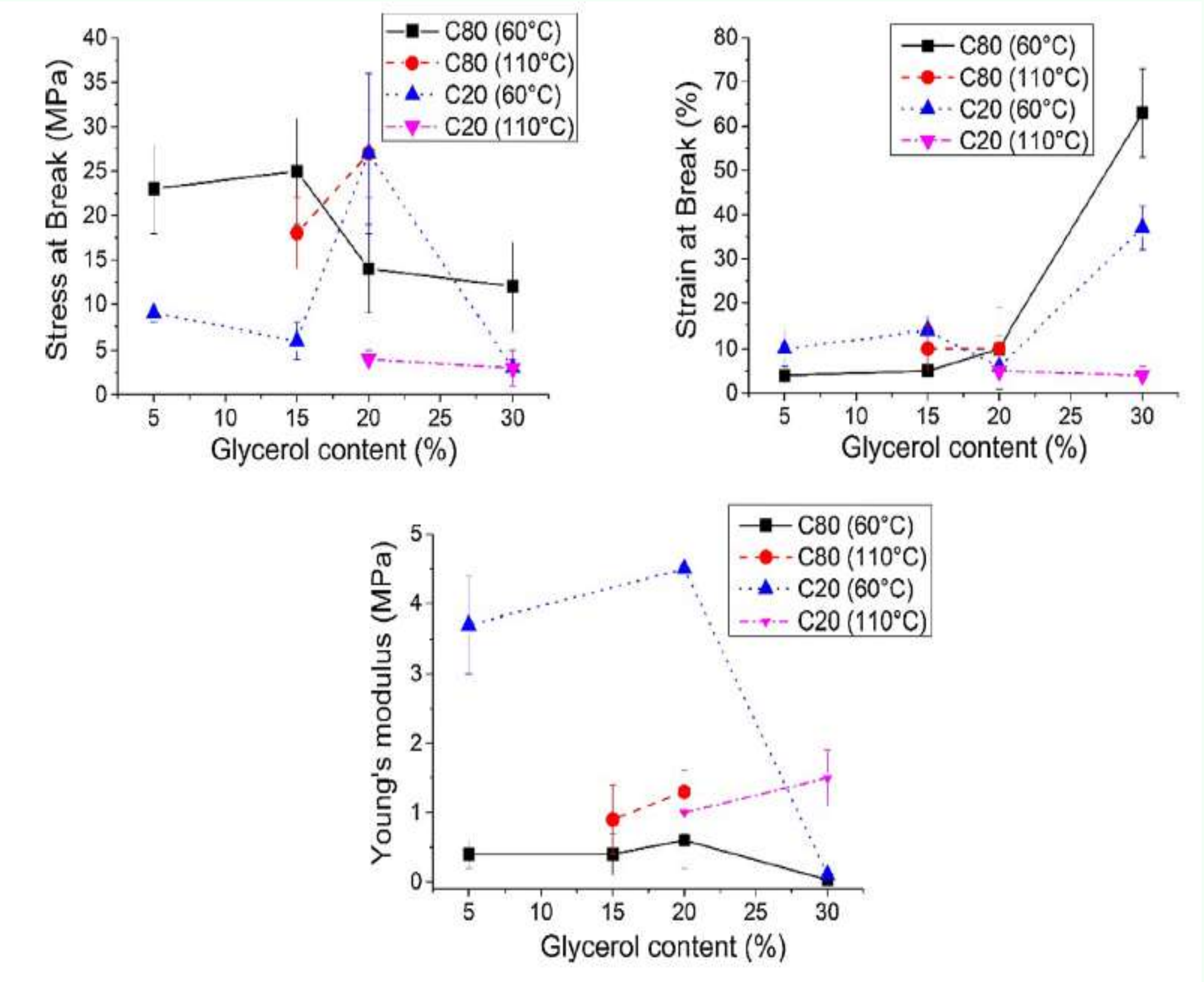
Fig.4 Tensile test of C80G30 film

Table 2 Tensile test results. Stress at break (σ_b) and percentage strain at break (ε_b) are reported

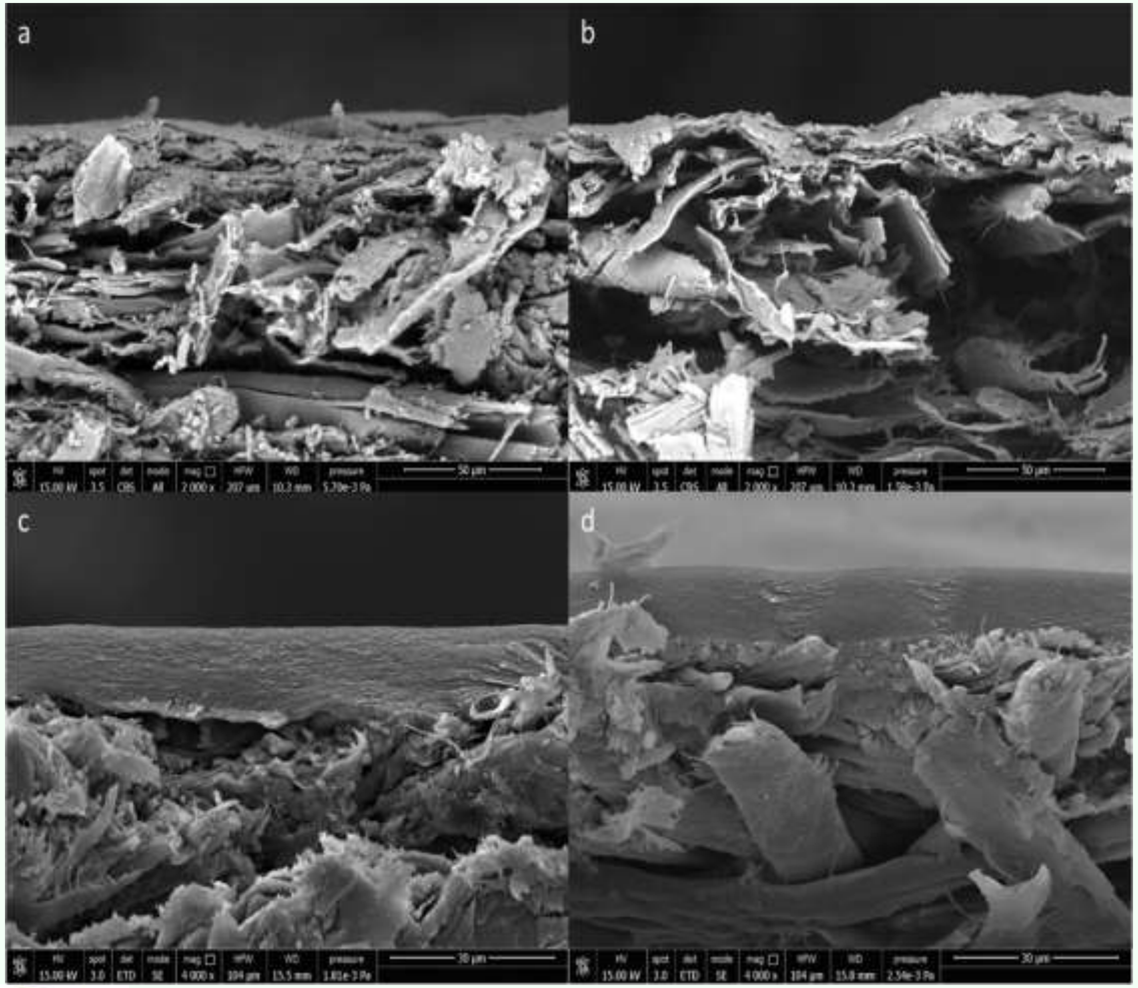
	Film sample	σ_b (MPa)	ε_b (%)
	C80G5	22,62±5,22	4,35±2,03
	C80G15	25,18±5,83	4,87±1,30
B	C80G20	14,06±4,75	10,13±9,36
	C80G30	12,45±4,80	63,45±10,37
	C20G5	9,09±1,08	10,20±3.83
A	C20G15	6,16±2,41	14,70±3,45
	C20G30	3,41±0,34	37,4±5,43

Fig.5 Films were prepared from the mixtures by solution casting method.





riciclato **vergine**



	Bending M (mN)	Bending C (mN)
virgin	370	160
Based on chitin (brush)	400	220
Based on chitosan (bar)	380	210
recycled	265	125
Based on chitosan (bar) 60°	400	200

$$E_{treatment} = \frac{M\cdot\rho - E_{board}\cdot I_{board}}{I_{treatment}}$$

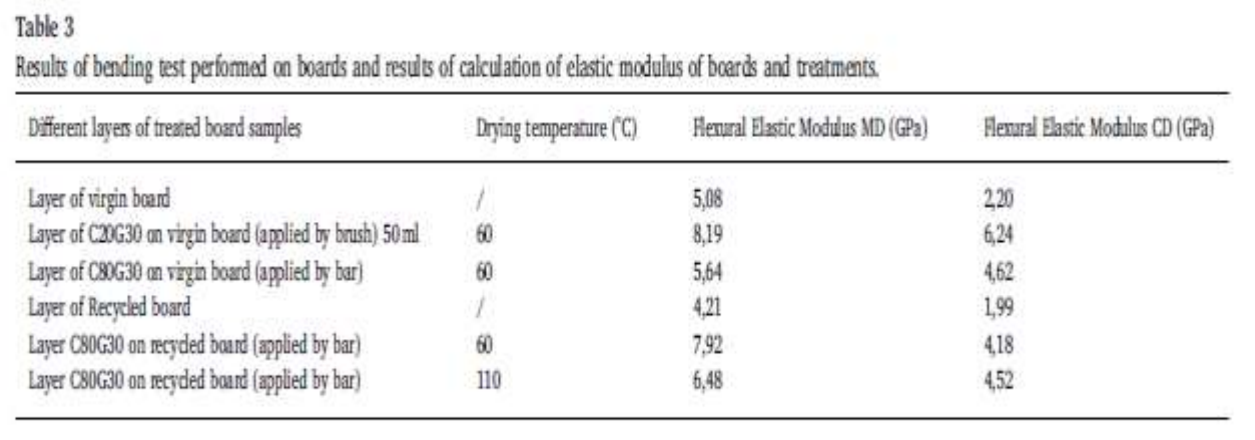


Table 3

Results of bending test performed on boards and results of calculation of elastic modulus of boards and treatments.

Different layers of treated board samples	Drying temperature (°C)	Flexural Elastic Modulus MD (GPa)	Flexural Elastic Modulus CD (GPa)
Layer of virgin board	/	5,08	2,20
Layer of C20G30 on virgin board (applied by brush) 50 ml	60	8,19	6,24
Layer of C80G30 on virgin board (applied by bar)	60	5,64	4,62
Layer of Recycled board	/	4,21	1,99
Layer C80G30 on recycled board (applied by bar)	60	7,92	4,18
Layer C80G30 on recycled board (applied by bar)	110	6,48	4,52

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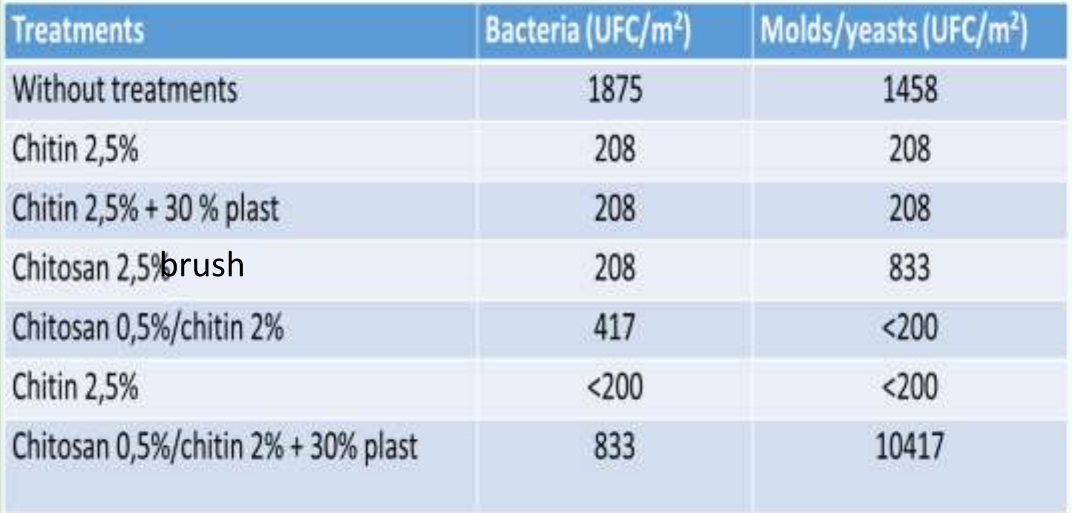
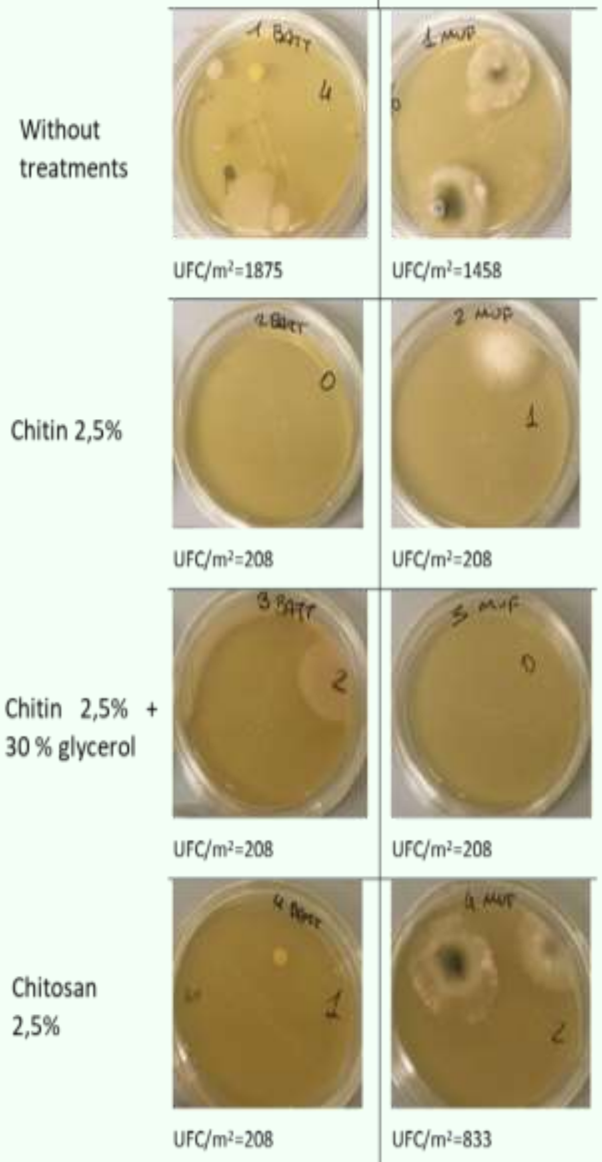


Treatments	Bacteria (UFC/m^2)	Molds/yeasts (UFC/m^2)
Without treatments	1875	1458
Chitin 2,5%	208	208
Chitin 2,5% + 30 % plast	208	208
Chitosan 2,5% brush	208	833
Chitosan 0,5%/chitin 2%	417	<200
Chitin 2,5%	<200	<200
Chitosan 0,5%/chitin 2% + 30% plast	833	10417

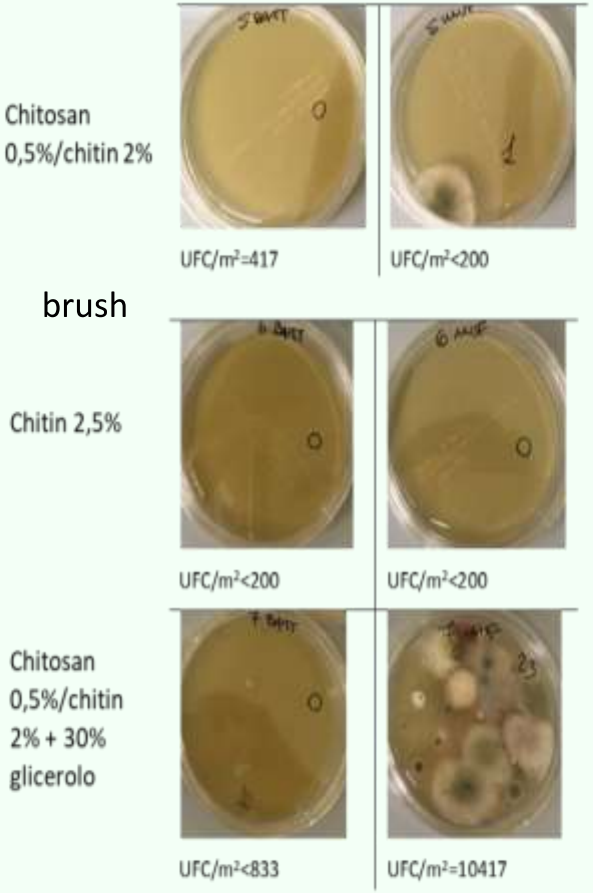
Questo studio conferma l'effetto antimicrobico di chitina e chitosano per l'applicazione su imballaggi in carta. In particolare, la chitina è più efficace come antimicrobico rispetto al chitosano, specialmente contro muffe e lieviti. Inoltre, l'aggiunta di plastificante in alcuni casi aumenta la proliferazione di microrganismi.

batteri muffe/lieviti


Without treatments
UFC/m^2=1875
UFC/m^2=1458
Chitin 2,5%
UFC/m^2=208
UFC/m^2=208
Chitin 2,5% + 30 % glycerol
UFC/m^2=208
UFC/m^2=208
Chitosan 2,5%
UFC/m^2=208
UFC/m^2=833

batteri muffe/lieviti

brush


Chitosan 0,5%/chitin 2%
UFC/m^2=417
UFC/m^2<200
Chitin 2,5%
UFC/m^2<200
UFC/m^2<200
Chitosan 0,5%/chitin 2% + 30% glicerolo
UFC/m^2<833
UFC/m^2=10417

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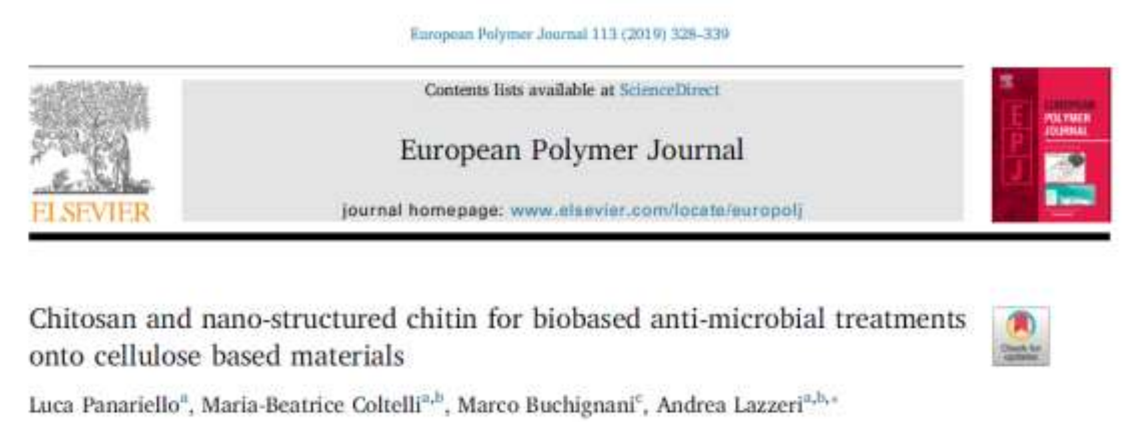

European Polymer Journal 113 (2019) 328–339

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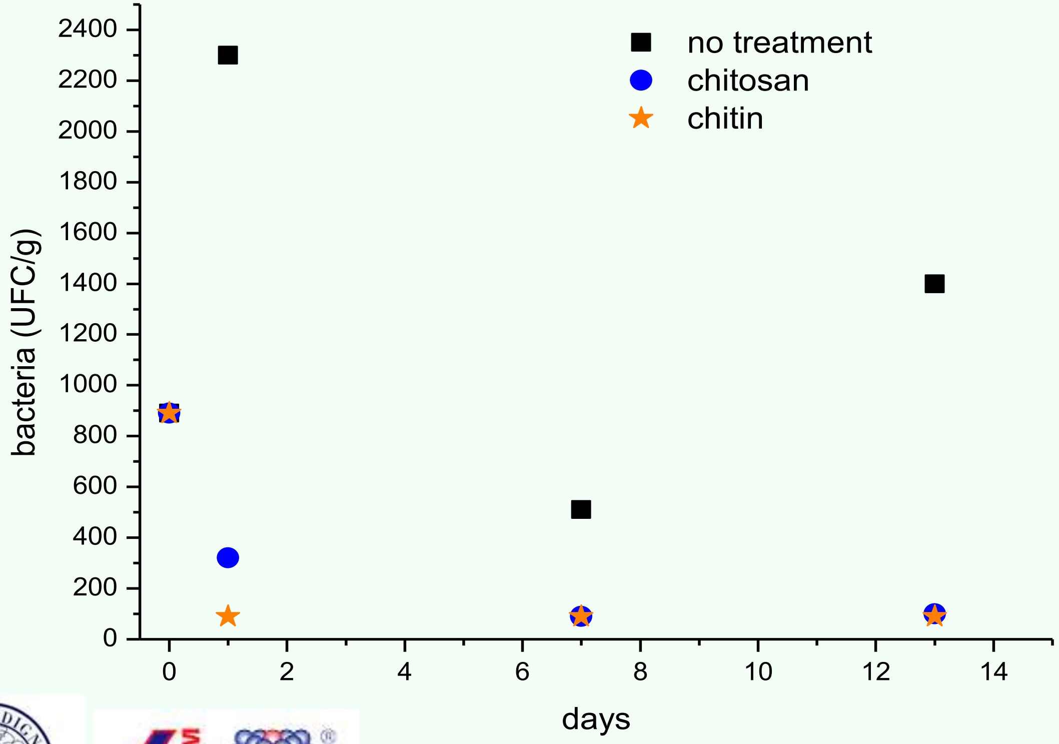
European Polymer Journal

journal homepage: www.elsevier.com/locate/europolj

Chitosan and nano-structured chitin for biobased anti-microbial treatments onto cellulose based materials

Luca Panariello[a], Maria-Beatrice Coltelli[a,b], Marco Buchignani[c], Andrea Lazzeri[a,b,*]



Recycled paperboard

TEST
MICROBIOLOGICI
SUL CIBO

> Shelf life !!!!

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IMBALLAGGI ECO-SOSTENIBILI PER IL CONTATTO ALIMENTARE

n-chitopack

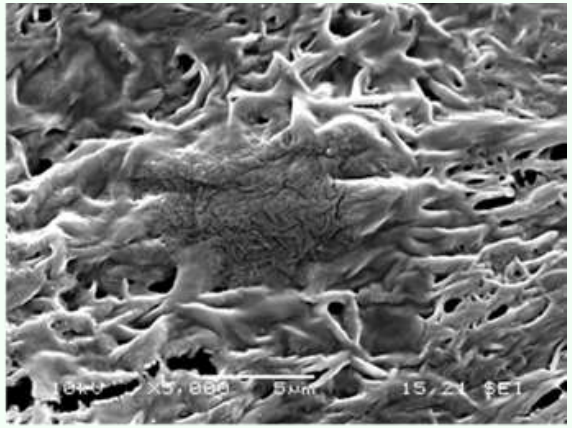
perché non applicare su substrati cellulosici dei film a base bioplastica contenenti CN ?

Per ottenerli alcuni studi sono stati effettuati nell'ambito del progetto europeo NANO-CHITOPACK

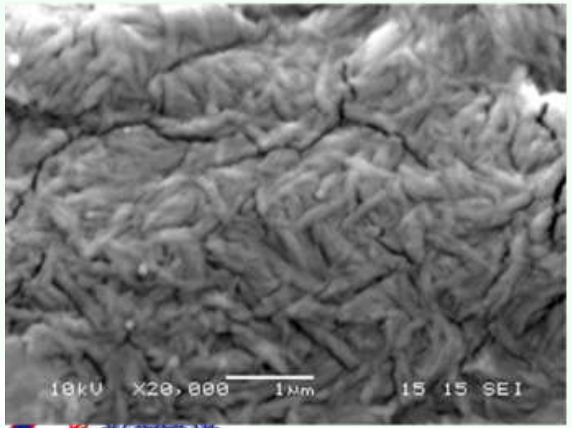
***N-CHITOPACK** - Sustainable technologies for the production of biodegradable materials based on natural chitin-nanofibrils derived by waste of fish industry, to produce food grade packaging,* **Research for the benefit of SME**, 01.11.2012-31.10.2014, European FP7 project

Dispersione delle CN nel biopoliestere allo stato fuso

Pre-composito preparato con PEG e CN


10kV X5,000 5µm 15 21 SEI

(a)

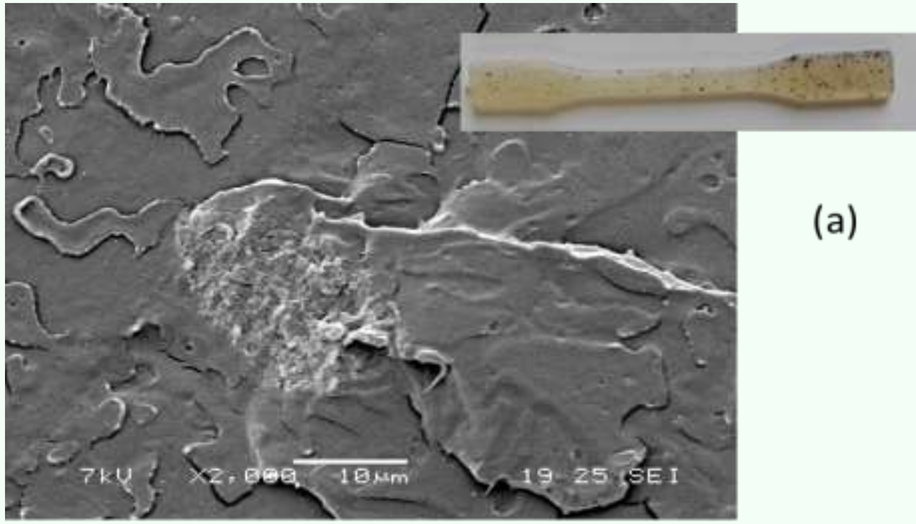

10kV X20,000 1µm 15 15 SEI

(b)

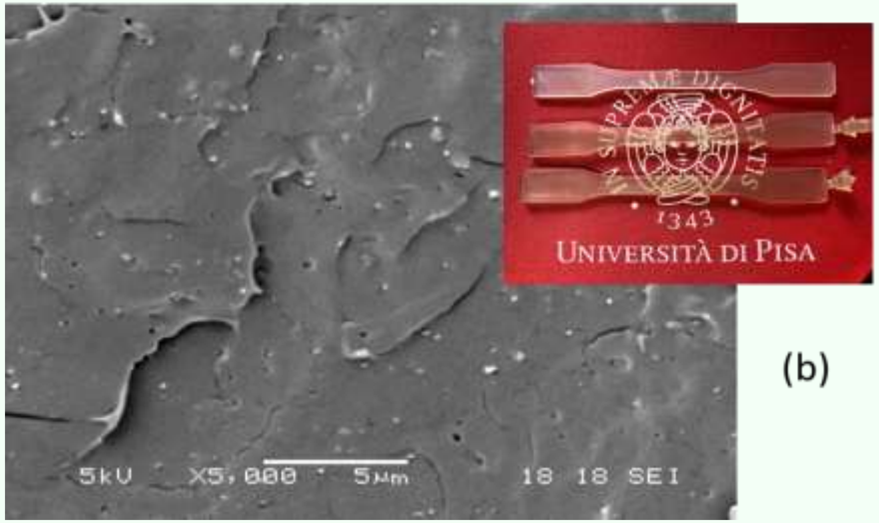
miniestrusore

Mini injection molding press

POLI(ACIDO LATTICO) (PLA)


7kV X2,000 10µm 19 25 SEI

(a)


5kV X5,000 5µm 18 18 SEI

(b)

TENSILE TESTS

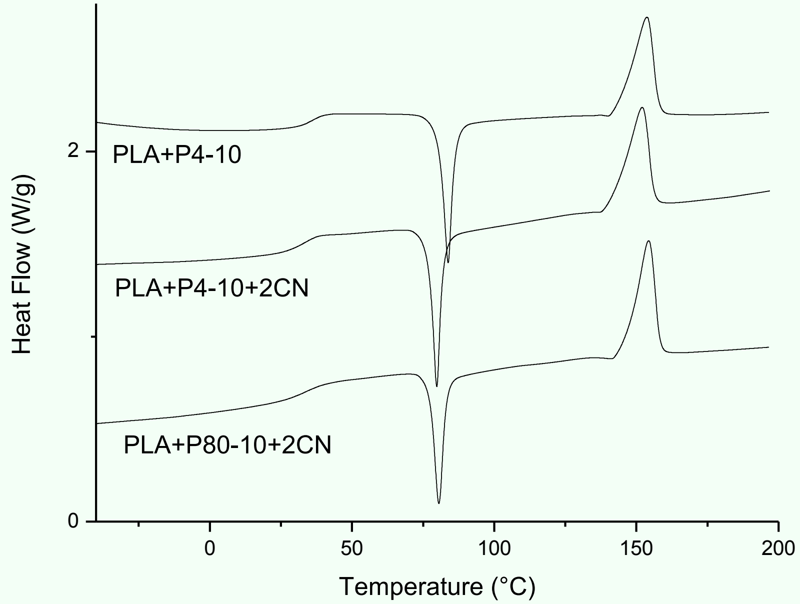
	E (GPa)	s_b (MPa)	e_b (%)
PLA	3.5 ± 0.1	60.4 ± 0.3	4.1 ± 0.5
PLA+2CN*	2.9 ± 0.1	58.0 ± 2.0	2.3 ± 0.4
PLA +P4-10	2.3 ± 0.3	33.0 ± 2.0	180.0 ± 10.0
PLA+P4-10+2CN	1.8 ± 0.3	32.0 ± 2.0	160.0 ± 10.0
PLA+P80-10+2CN	2.5 ± 0.1	34.0 ± 2.0	160.0 ± 10.0

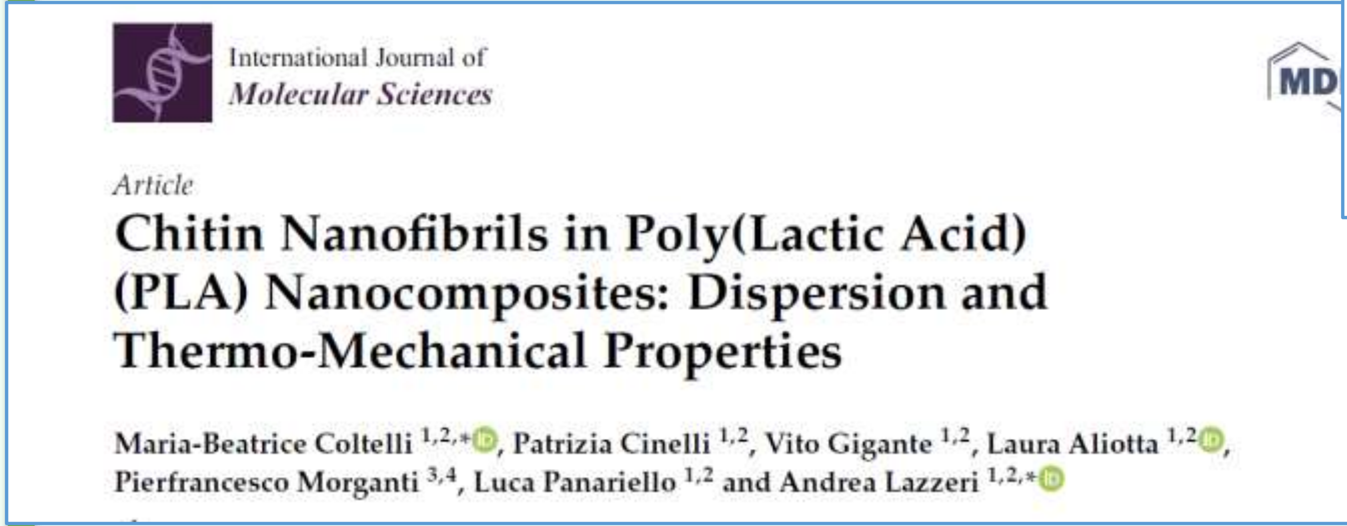
Thermal properties : DSC

	T_g (°C)	T_{cc} (°C)	DH_{cc} (J/g)	T_m (°C)	DH_m (J/g)
PLA	59.1	109.4	23.8	148.3/157,4	2.6
PLA+2CN*	55.2	108.8	23.3	147.9/155,8	1.2
PLA + P4-10	36.1	83.8	21.3	153.7	11.3
PLA+P4-10+2CN	32.7	79.8	17.1	152.1	12.1
PLA+P80-10+2CN	31.4	80.5	17.5	154.2	11.8

Sebbene i CN non abbiano migliorato il modulo elastico e lo stress a rottura, il mantenimento di un elevato allungamento a rottura indica che è stata ottenuta una dispersione sulla nanoscala, in accordo con le valutazioni morfologiche e ottiche.

Le proprietà termiche non vengono influenzate significativamente dalla presenza delle CN



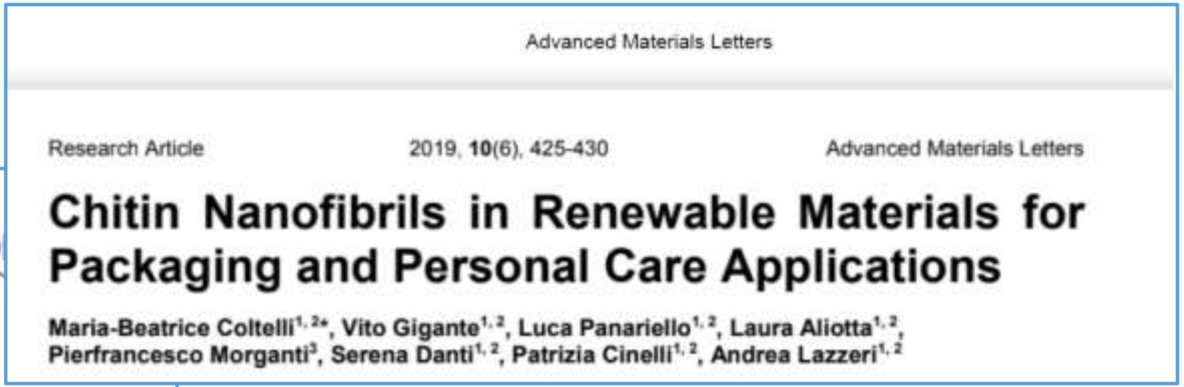


International Journal of *Molecular Sciences*

Article

Chitin Nanofibrils in Poly(Lactic Acid) (PLA) Nanocomposites: Dispersion and Thermo-Mechanical Properties

Maria-Beatrice Coltelli[1,2,*], Patrizia Cinelli[1,2], Vito Gigante[1,2], Laura Aliotta[1,2], Pierfrancesco Morganti[3,4], Luca Panariello[1,2] and Andrea Lazzeri[1,2,*]

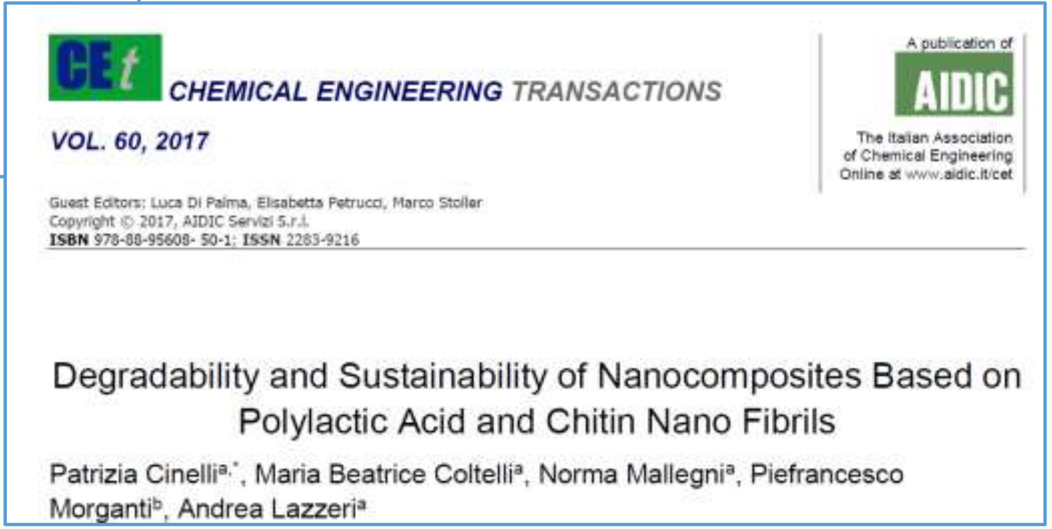


Advanced Materials Letters

Research Article 2019, **10**(6), 425-430 Advanced Materials Letters

Chitin Nanofibrils in Renewable Materials for Packaging and Personal Care Applications

Maria-Beatrice Coltelli[1, 2*], Vito Gigante[1, 2], Luca Panariello[1, 2], Laura Aliotta[1, 2], Pierfrancesco Morganti[3], Serena Danti[1, 2], Patrizia Cinelli[1, 2], Andrea Lazzeri[1, 2]



CEt *CHEMICAL ENGINEERING TRANSACTIONS*

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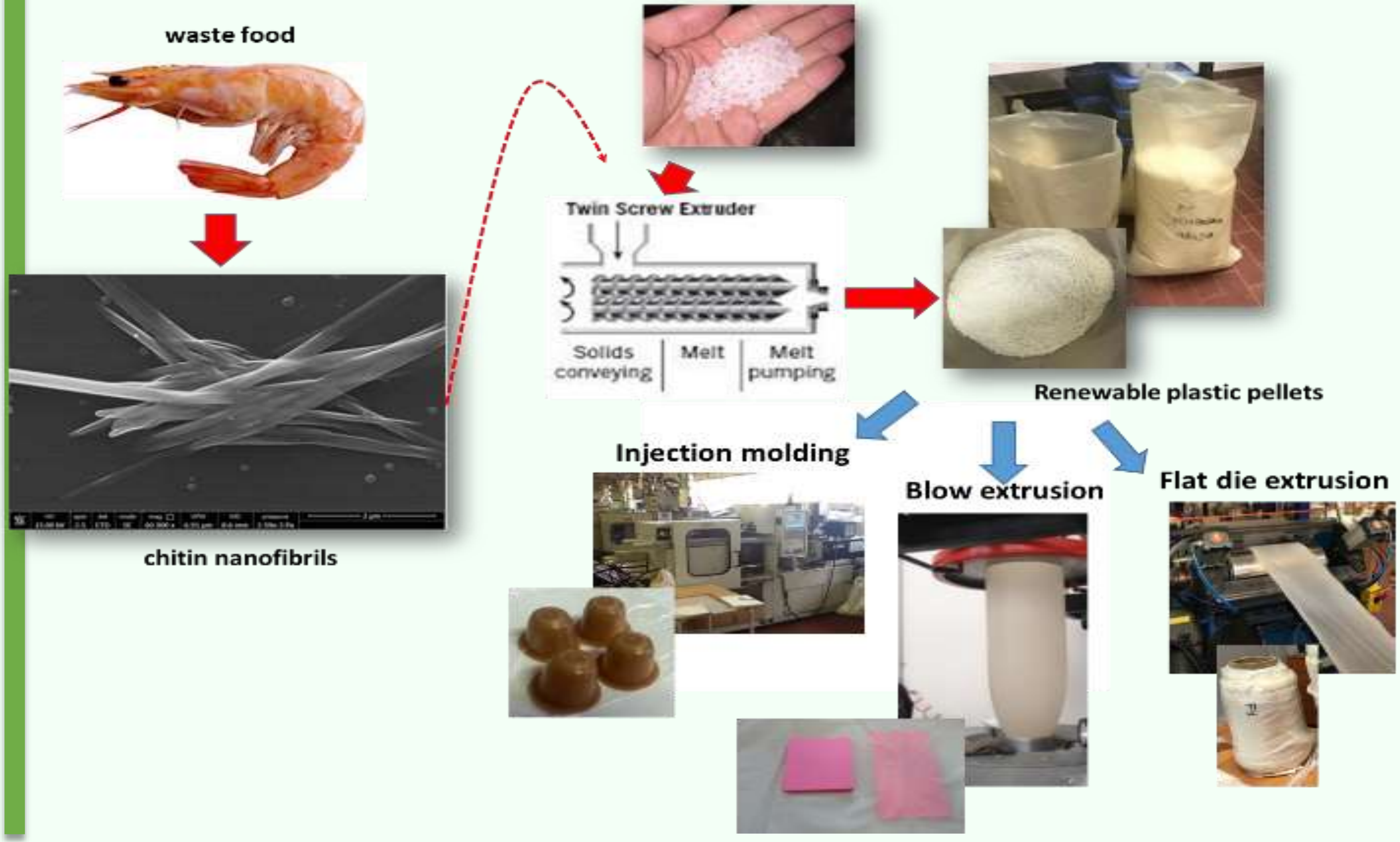
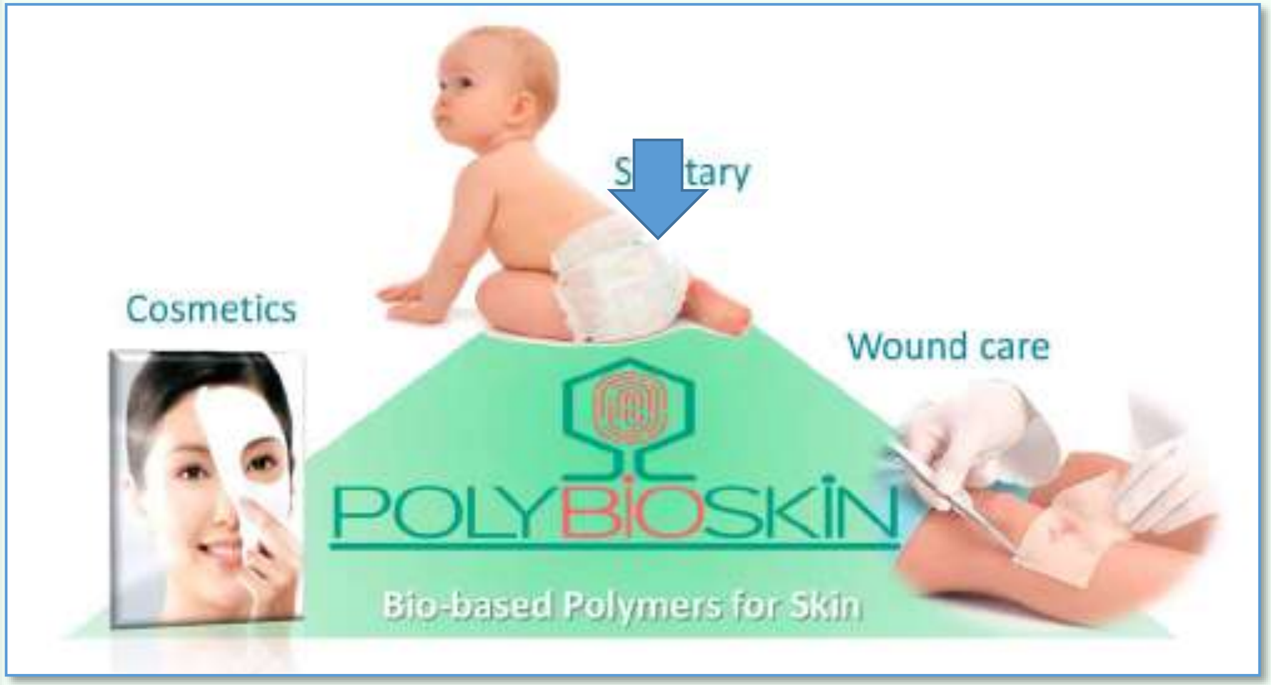
Degradability and Sustainability of Nanocomposites Based on Polylactic Acid and Chitin Nano Fibrils

Patrizia Cinelli[a,*], Maria Beatrice Coltelli[a], Norma Mallegni[a], Piefrancesco Morganti[b], Andrea Lazzeri[a]


waste food
chitin nanofibrils
Twin Screw Extruder
Solids conveying
Melt
Melt pumping
Renewable plastic pellets
Injection molding
Blow extrusion
Flat die extrusion

Questi risultati possono aprire la strada allo sviluppo di nanocompositi funzionali contenenti queste nanofibrille antimicrobiche e rigenerative, adatte per essere utilizzate in una vasta gamma di applicazioni (imballaggio, cura della persona e biomedicina).

OBIETTIVI DI POLYBIOSKIN



La visione del progetto PolyBioSkin è di incrementare l'uso di biopolimeri che offrono attività antimicrobica, antiossidante, assorbenza, compatibilità cutanea in applicazioni strategiche ed esigenti per il contatto con la pelle (pannolini, maschere facciali e medicazioni):

- miglioramento della qualità della vita e del benessere;
- riduzione dell'impatto ambientale e opzioni di fine vita più sostenibili;
- maggiore competitività dei settori europeo sanitario, cosmetico e biomedico.

>90% di contenuto bio-based in tutte le applicazioni

Nell'ambito di questo progetto è stato dimostrato che le nanofibrille chitina e le miscele di biopoliestere non riducono la vitalità delle cellule della pelle

This project has received funding from the Bio Based Industries Joint Undertaking under the European Union's Horizon 2020 research and innovation programme - grant agreement N° 745839

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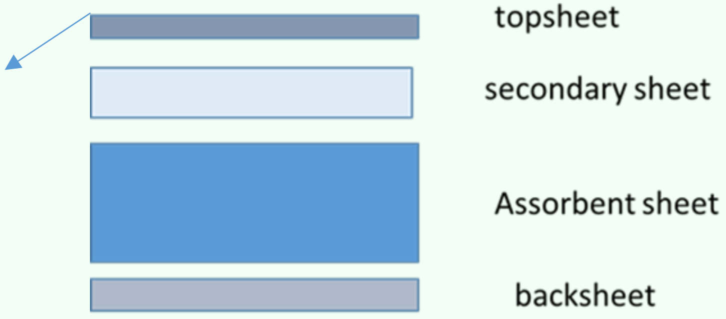
Sono stati formulati film di biopoliesteri con le proprietà necessarie per il **topsheet**.

	F1 MD	F1 CD	F2 MD	F2 CD
E (GPa)	≈ 0.140	≈0.111	≈0.080	≈0.080
σ_b (MPa)	21 ± 3	16 ± 1	20 ± 6	25.1 ± 5.5
ε_b (%)	380 ± 30	33 ± 20	410 ± 30	423.2 ± 46.1

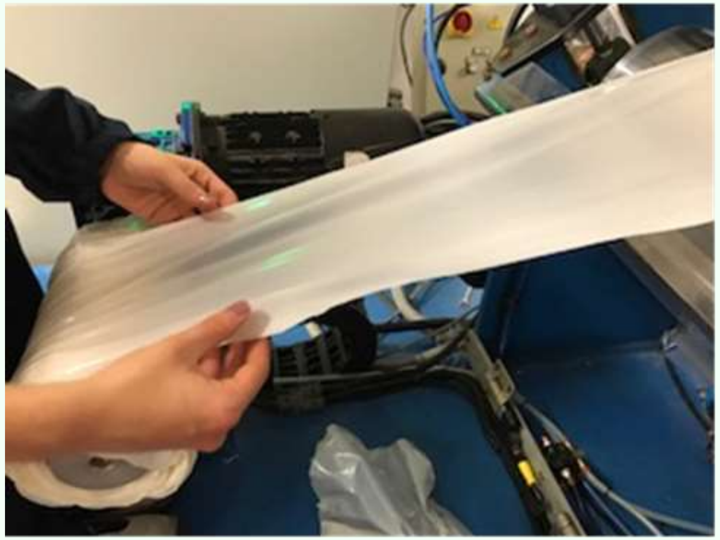


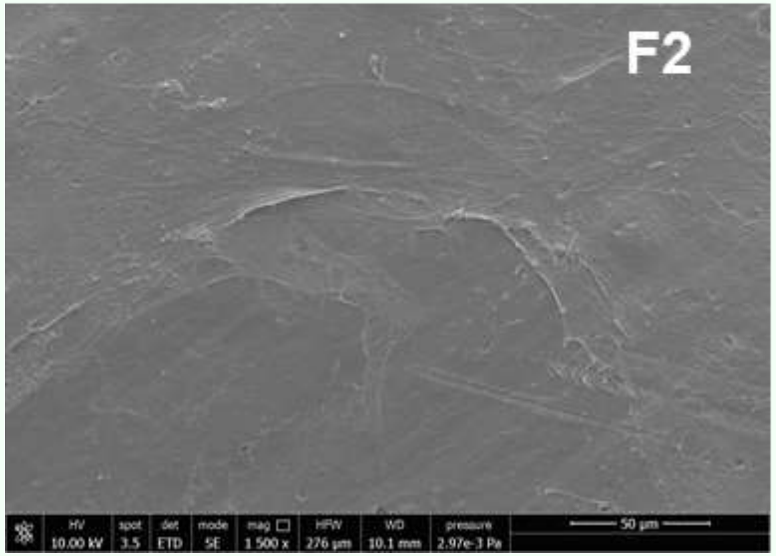
topsheet

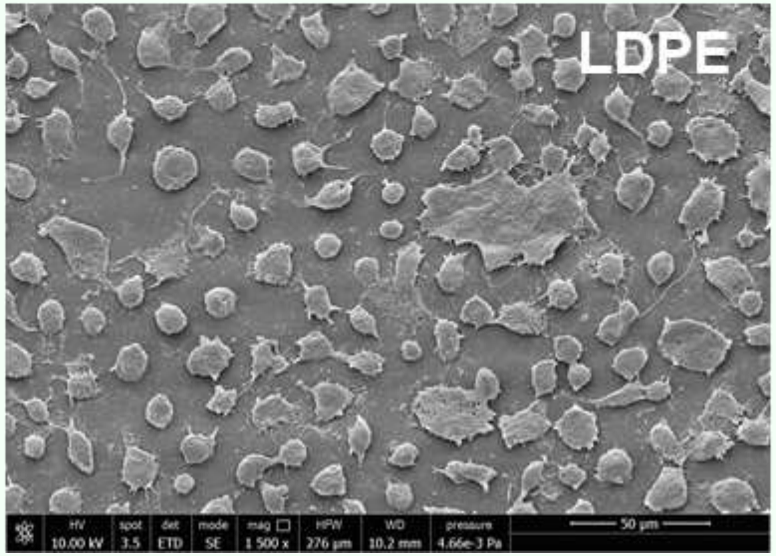
secondary sheet

Assorbent sheet

backsheet







La compatibilità dei film con cheratinociti e cellule staminali mesenchimali è stata trovata molto buona, con una leggera azione antimicrobica dovuta all'attivazione di defensine

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Attività in corso sull’uso di CN nei film

Aggiungere CN per funzionalizzare I film per topsheet

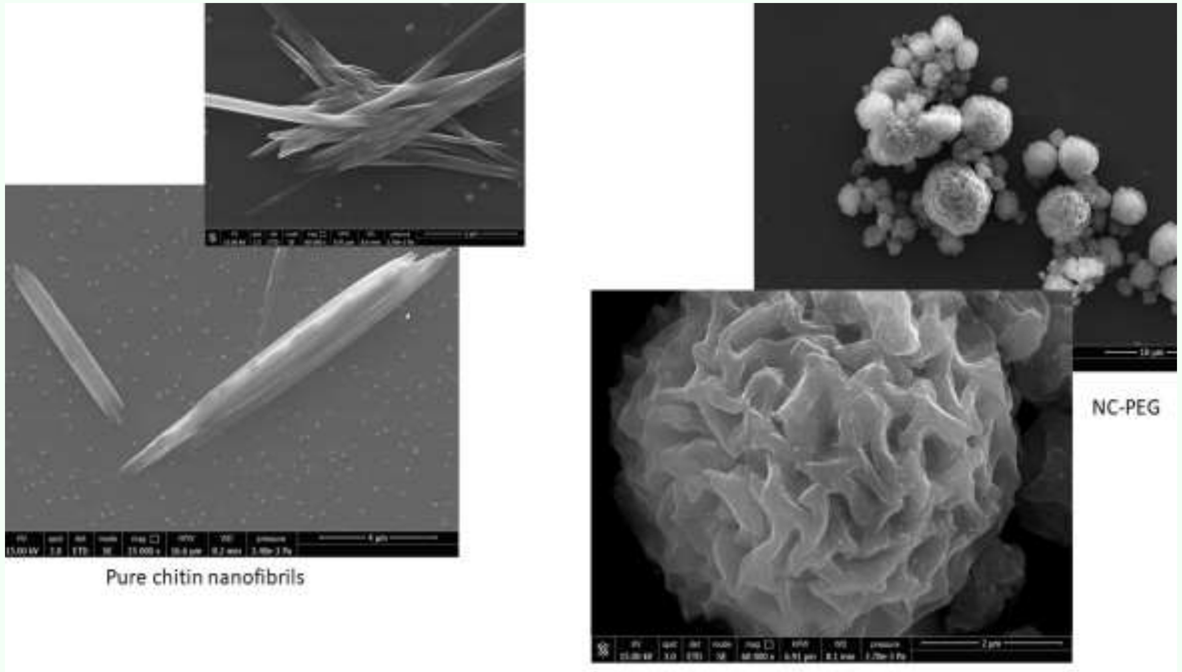
2 pre-compositi :
- Spray dried 98:2 CN/PEG powder
- Pre-composito 50:50 CN/PEG

blend	E (GPa)	σ_b (MPa)	ε_b (%)
PLA pure	3,5	61,4	2,7
PLA + 2% CN	3,5	48	**1,60**
PLA70/P30 + 20%ATBC	0,3	16,8	210
PLA70/P30 + 2% CN + 20%ATBC	0,68	13,9	264
PLA70/P30 +4%nc(50)+18%ATBC	0,13	20,1	490
PLA70/P30 +4%nc(50)+16%atbc+2%bioS	0,15	21,1	335,4
PLA70/P30 +4%nc(50)+14%atbc+4%bioS	0,10	19,1	370

Uso di un melt strngth enhancer

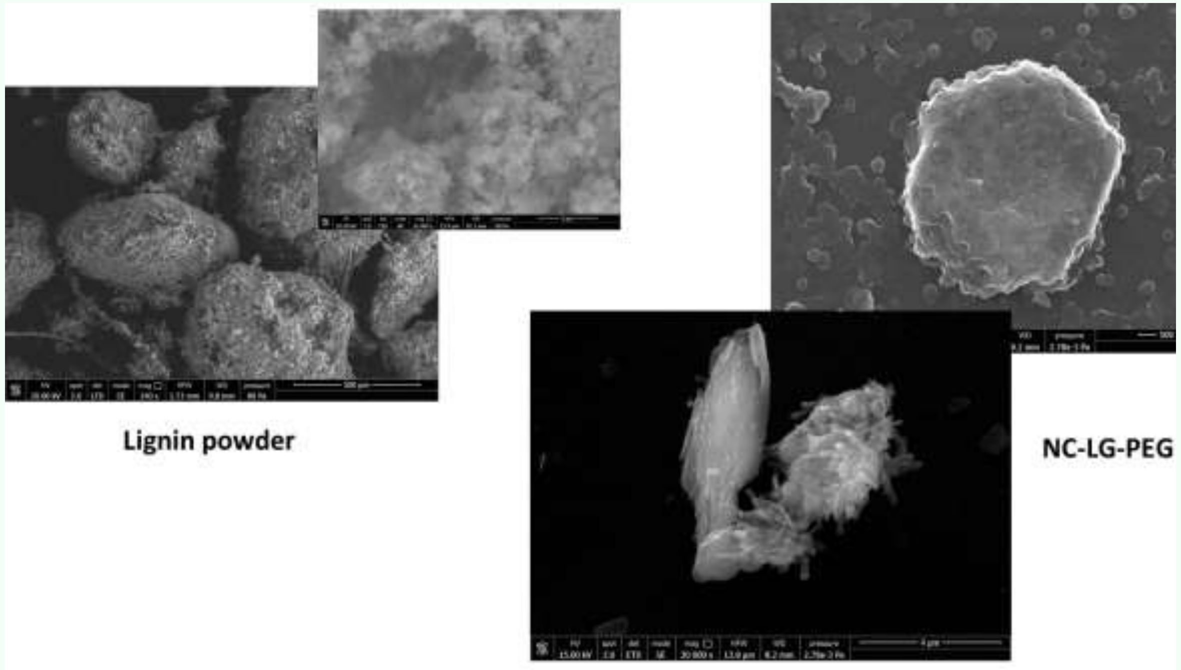
Test anti-microbici preliminari hanno mostrato che non c’è una attività anti-microbica, probabilmente perché CN è scarsamente presente sulla superficie del film → COATING

Modifiche funzionali grazie ad una piattaforma di biopolimeri e complessi



Pure chitin nanofibrils

NC-PEG



Lignin powder

NC-LG-PEG

Composti attivi possono essere intrappolati nei complessi chitina/lignina

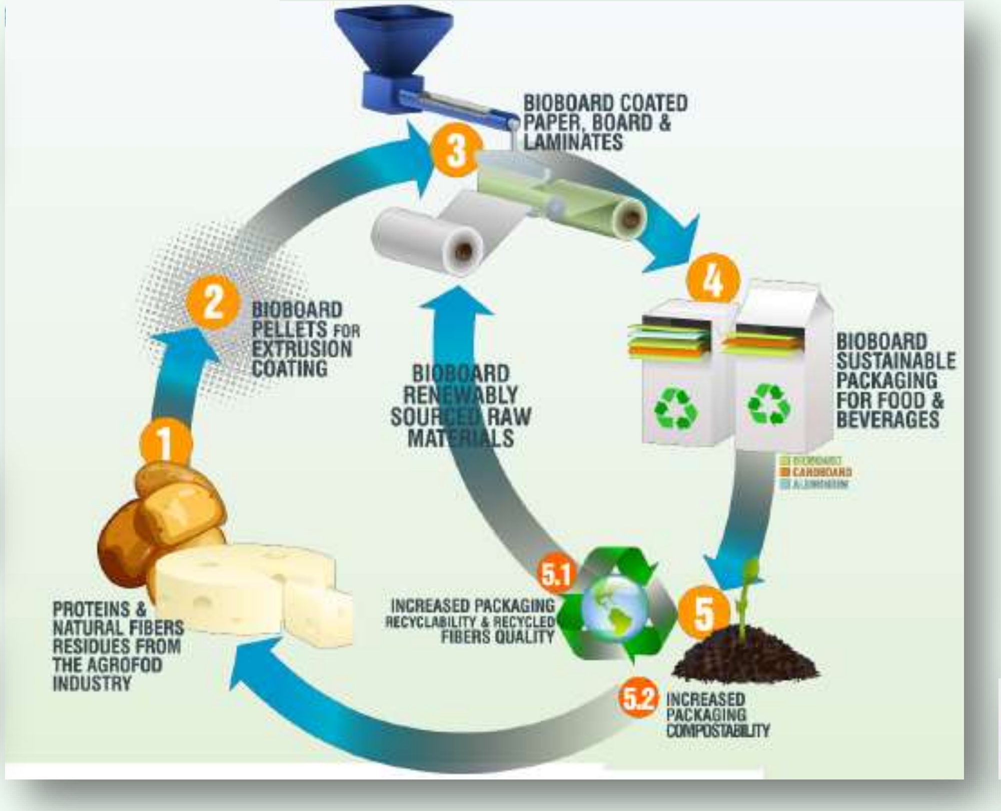
Prove di stabilità termica e compatibilità con i cheratinociti hanno portato alla selezione dei migliori trattamenti per le tre diverse applicazioni del progetto POLYBIOSKIN

Proprietà barriera: il progetto BIOBOARD

BIO-BOARD deals with the "Development of sustainable protein-based paper and paperboard coating systems to increase the recyclability of food and beverage packaging.

Research for the benefit of SME AGs, *01.11.2012-31.10.2015*

Research activity: Production of sustainable plastic films containing protein derived by whey and potato by products.
Whey protein coating can provide bio-degradable plastic layers that can replace existing plastic coatings in multilayer packaging and enhance their recyclability –by separating the individual fractions of the multilayer packaging.


1
PROTEINS & NATURAL FIBERS RESIDUES FROM THE AGROFOD INDUSTRY
2
BIOBOARD PELLETS FOR EXTRUSION COATING
3
BIOBOARD COATED PAPER, BOARD & LAMINATES
4
BIOBOARD SUSTAINABLE PACKAGING FOR FOOD & BEVERAGES
CARDBOARD
BIOBOARD RENEWABLY SOURCED RAW MATERIALS
5
5.1
INCREASED PACKAGING RECYCLABILITY & RECYCLED FIBERS QUALITY
5.2
INCREASED PACKAGING COMPOSTABILITY

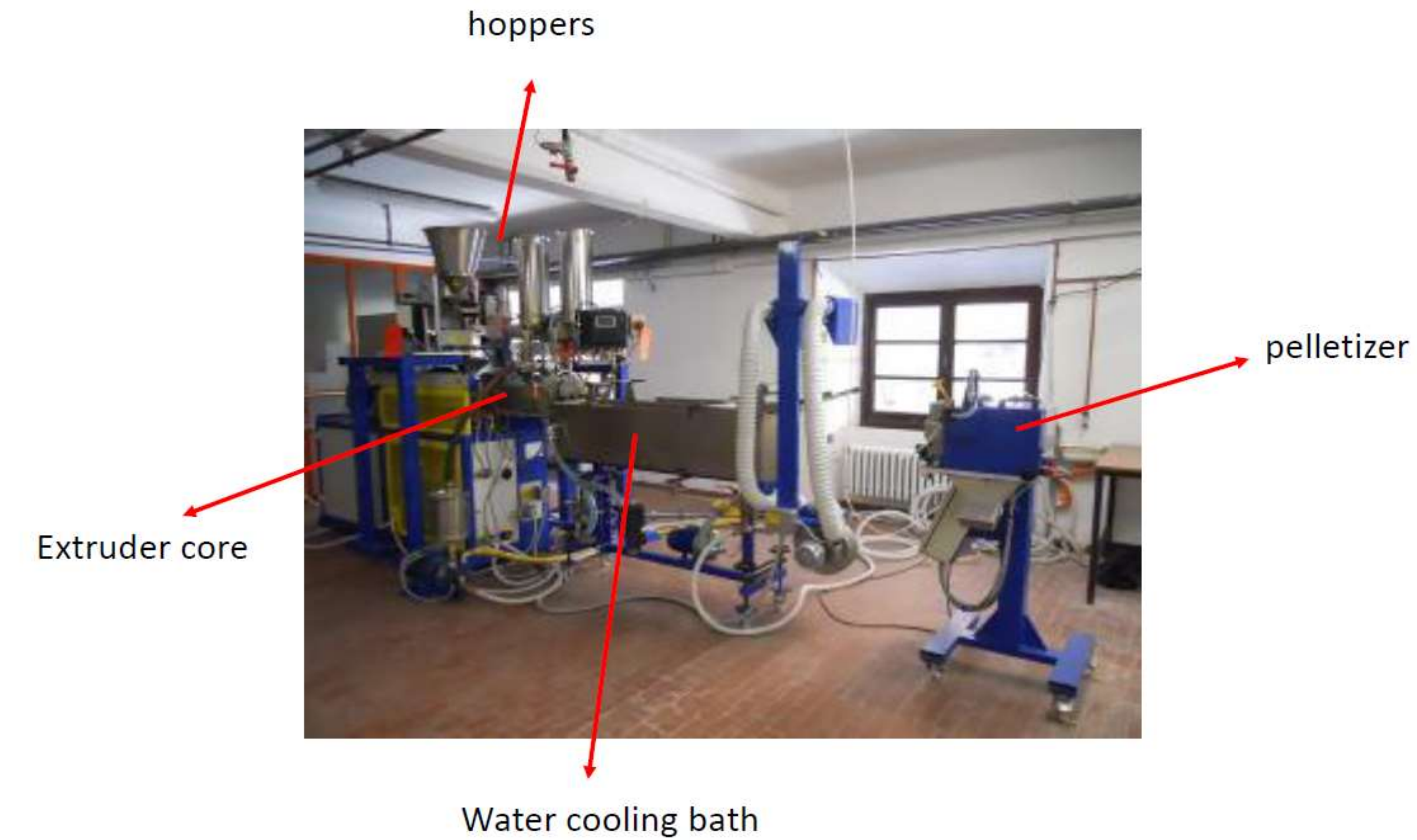
Whey proteins

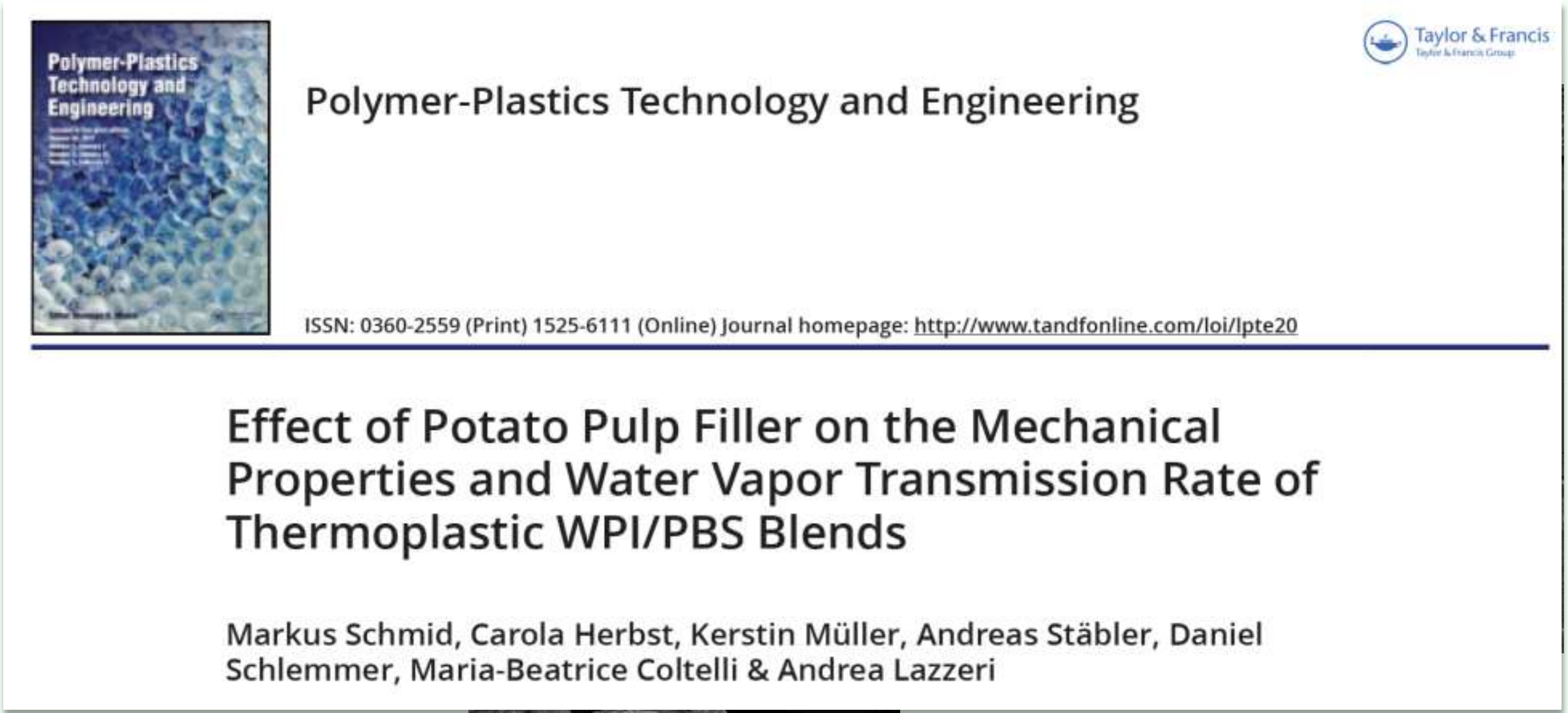
By-product of cheese production containing 7% dry matter of which 13% proteins, 75% lactose, 8% minerals, 3% organic acids and 1% fat

Whey protein can be separated and purified by membrane filtration process followed by spray drying

- 40-50% of the 50 million tons of whey produced annually in Europe is discarded
- Finding a new value added use is of interest for dairy producers

Preparation of biobased blends and composites


hoppers
pelletizer
Extruder core
Water cooling bath



Taylor & Francis
Taylor & Francis Group

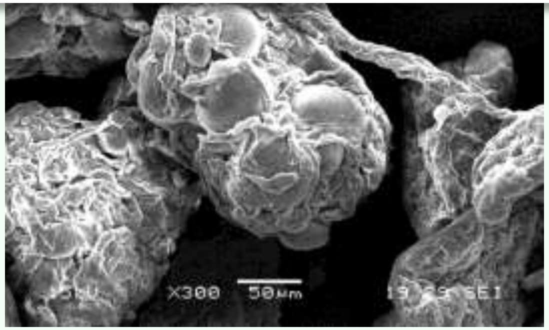
Polymer-Plastics Technology and Engineering

ISSN: 0360-2559 (Print) 1525-6111 (Online) Journal homepage: http://www.tandfonline.com/loi/lpte20

Effect of Potato Pulp Filler on the Mechanical Properties and Water Vapor Transmission Rate of Thermoplastic WPI/PBS Blends

Markus Schmid, Carola Herbst, Kerstin Müller, Andreas Stäbler, Daniel Schlemmer, Maria-Beatrice Coltelli & Andrea Lazzeri

Potato pulp

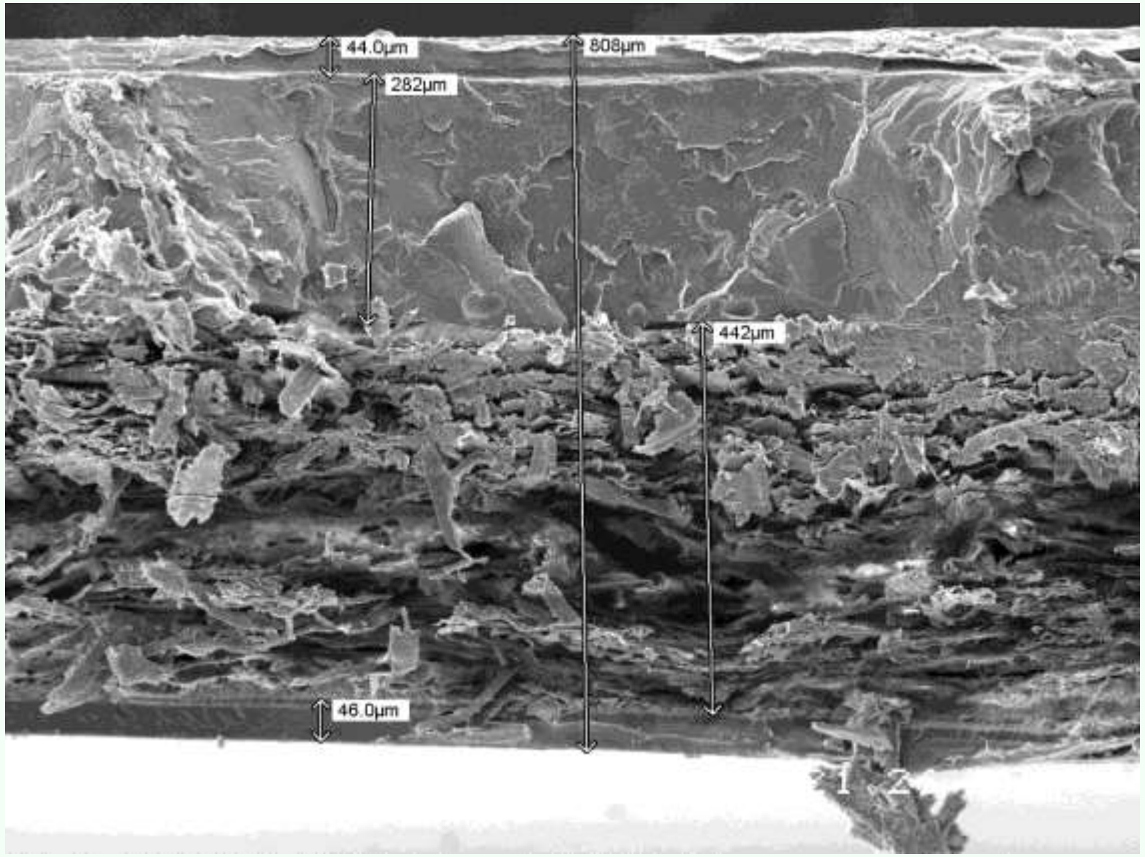


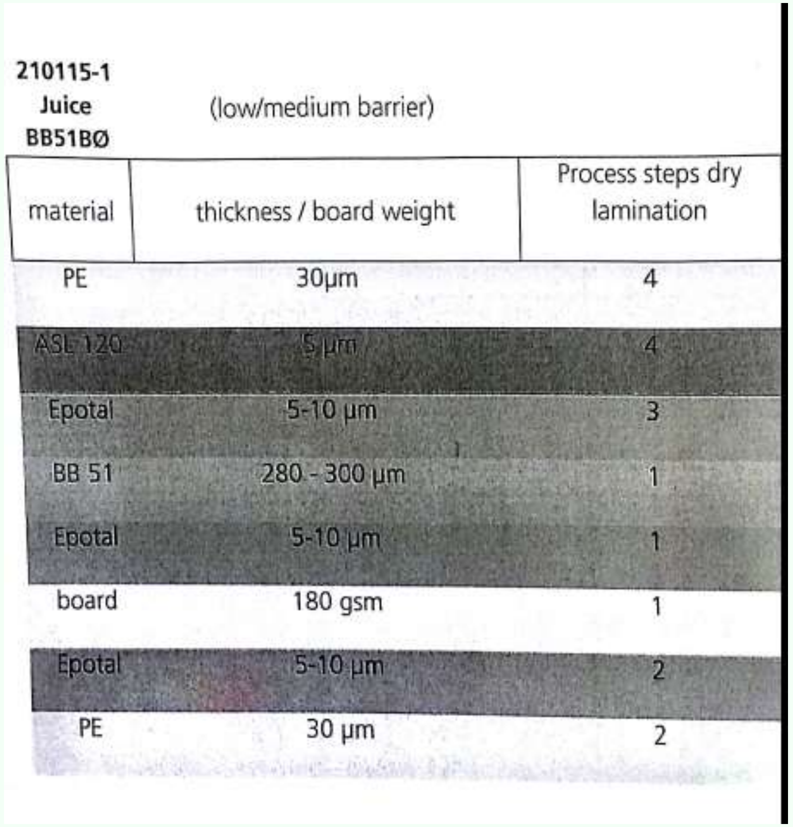
The potato pulp forms aggregates

paper

BB51BΦ

juice

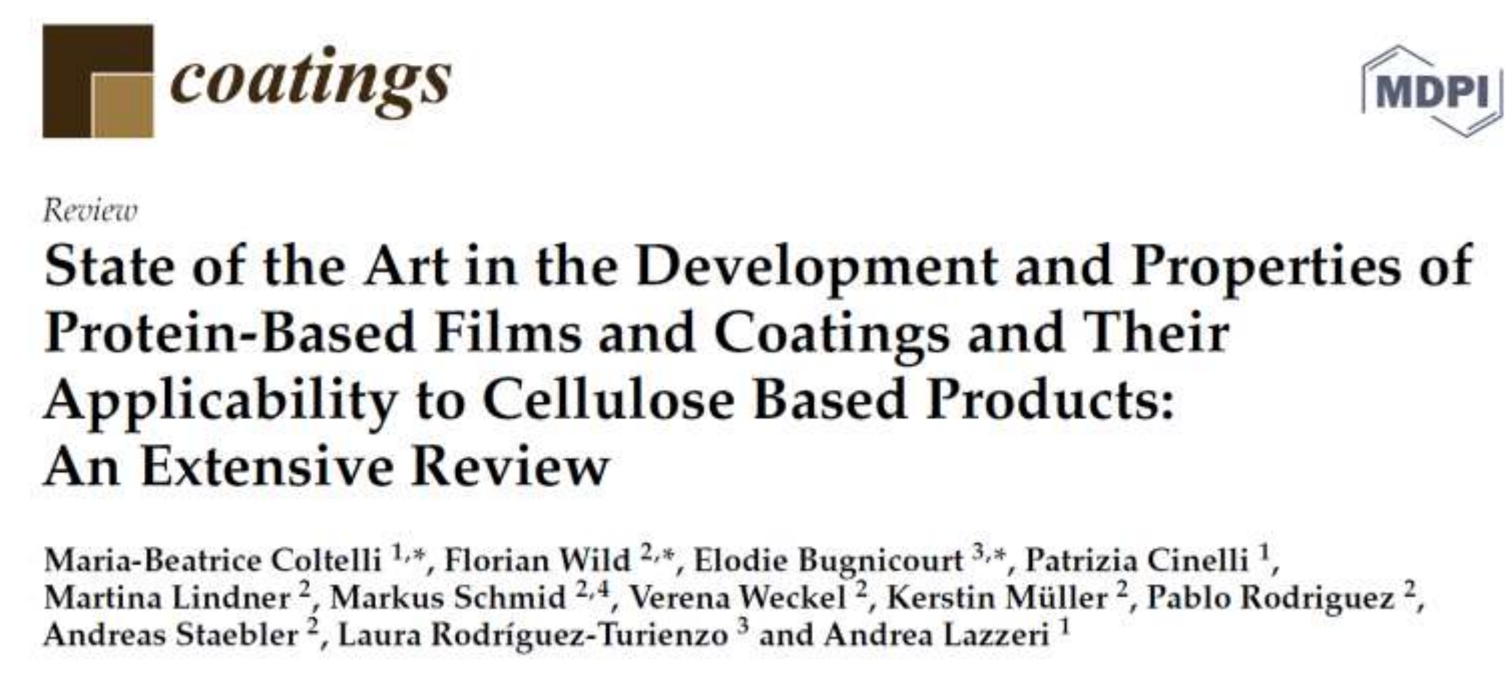




210115-1
Juice
BB51B0

(low/medium barrier)

material	thickness / board weight	Process steps dry lamination
PE	30µm	4
ASL 120	5 µm	4
Epotal	5-10 µm	3
BB 51	280 - 300 µm	1
Epotal	5-10 µm	1
board	180 gsm	1
Epotal	5-10 µm	2
PE	30 µm	2



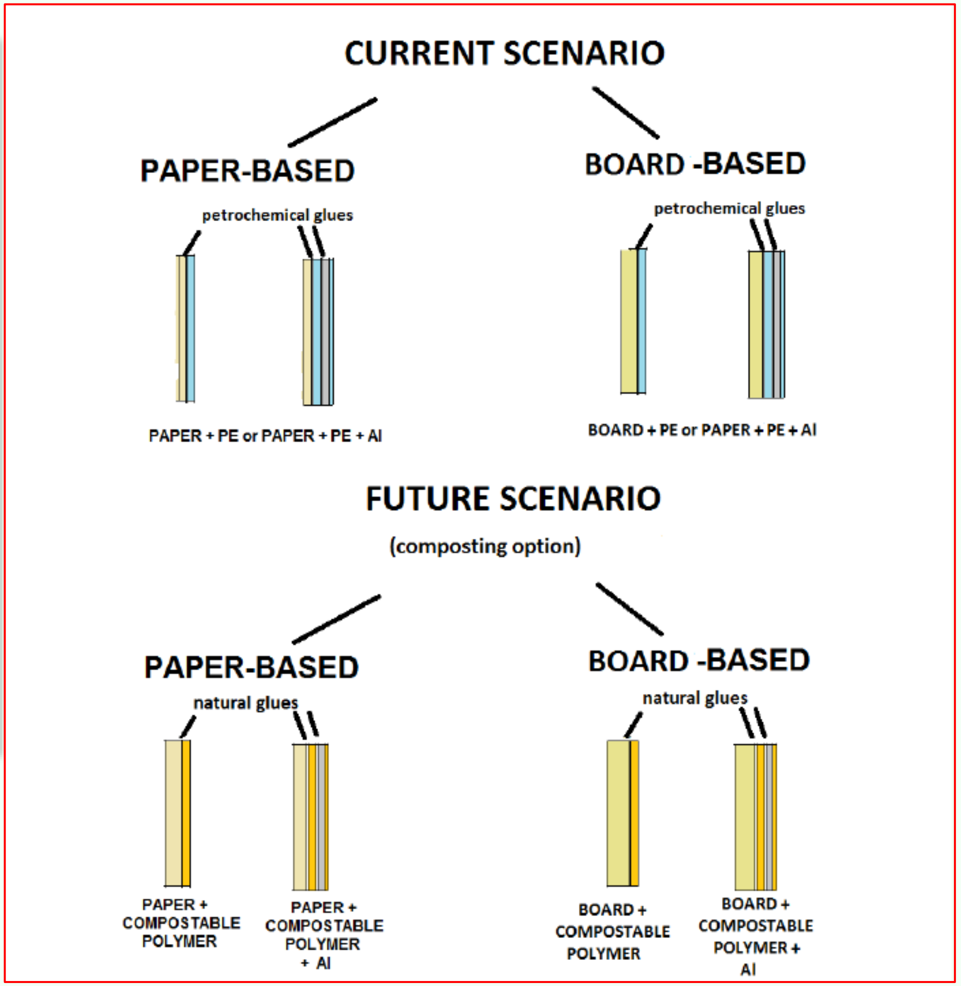
coatings

MDPI

Review

State of the Art in the Development and Properties of Protein-Based Films and Coatings and Their Applicability to Cellulose Based Products: An Extensive Review

Maria-Beatrice Coltelli [1,*], Florian Wild [2,*], Elodie Bugnicourt [3,*], Patrizia Cinelli [1], Martina Lindner [2], Markus Schmid [2,4], Verena Weckel [2], Kerstin Müller [2], Pablo Rodriguez [2], Andreas Staebler [2], Laura Rodríguez-Turienzo [3] and Andrea Lazzeri [1]



CURRENT SCENARIO

PAPER-BASED

petrochemical glues

PAPER + PE or PAPER + PE + Al

BOARD -BASED

petrochemical glues

BOARD + PE or PAPER + PE + Al

FUTURE SCENARIO

(composting option)

PAPER-BASED

natural glues

PAPER + COMPOSTABLE POLYMER

PAPER + COMPOSTABLE POLYMER + Al

BOARD -BASED

natural glues

BOARD + COMPOSTABLE POLYMER

BOARD + COMPOSTABLE POLYMER + Al

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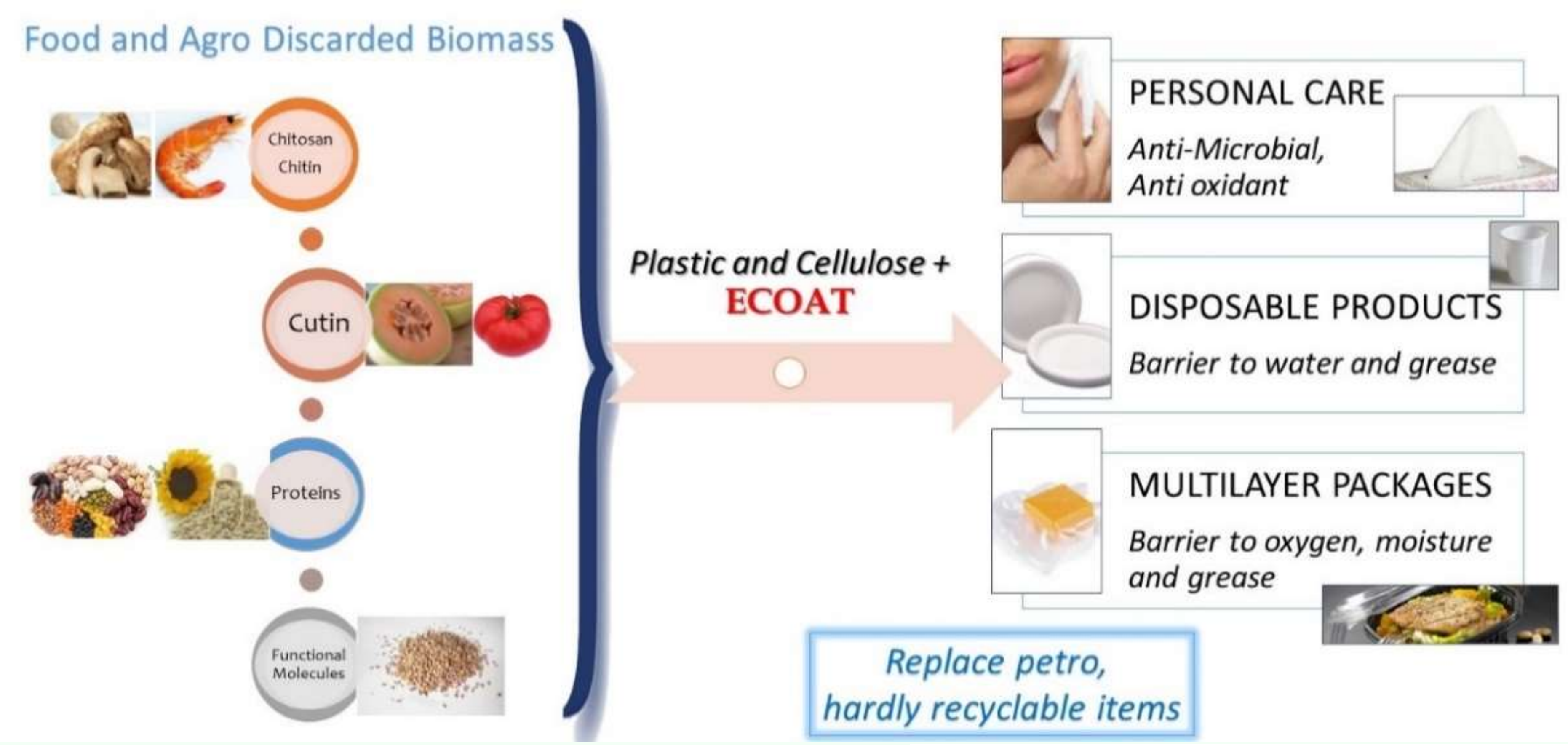
IMBALLAGGI ECO-SOSTENIBILI PER IL CONTATTO ALIMENTARE

ecoat

ECO sustainable multifunctional biobased COATings with enhanced performance and end of life options
GA 837863, BBI2018-RIA-A



Food and Agro Discarded Biomass

Chitosan
Chitin

Cutin

Proteins

Functional Molecules

Plastic and Cellulose +
ECOAT

PERSONAL CARE

Anti-Microbial,
Anti oxidant

DISPOSABLE PRODUCTS

Barrier to water and grease

MULTILAYER PACKAGES

Barrier to oxygen, moisture and grease

Replace petro, hardly recyclable items

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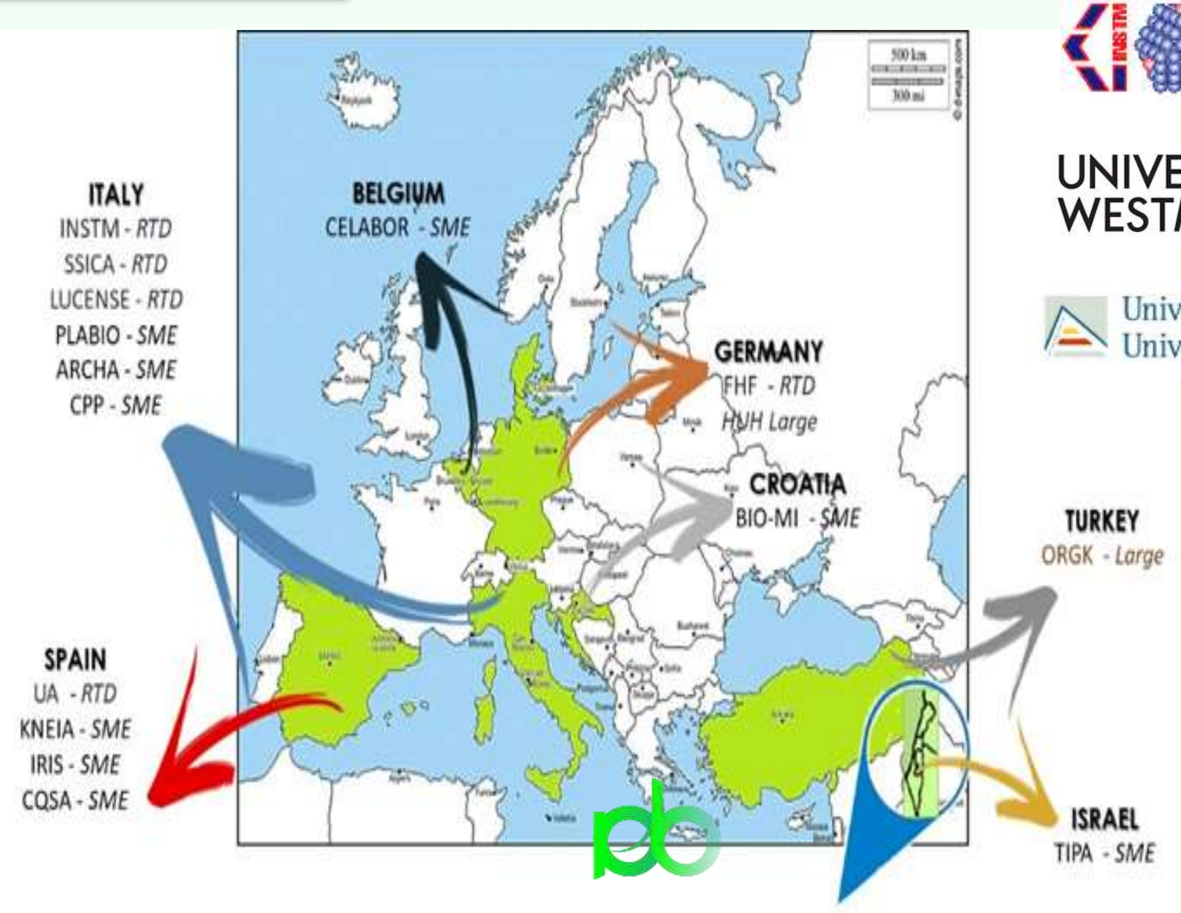
Bio-based Industries Consortium

Horizon 2020
European Union Funding
for Research & Innovation

LUCCA, 23 Maggio 2019


ITALY
INSTM - RTD
SSICA - RTD
LUCENSE - RTD
PLABIO - SME
ARCHA - SME
CPP - SME
BELGIUM
CELABOR - SME
GERMANY
FHF - RTD
HUH Large
CROATIA
BIO-MI - SME
TURKEY
ORGK - Large
SPAIN
UA - RTD
KNEIA - SME
IRIS - SME
CQSA - SME
ISRAEL
TIPA - SME

UNIVERSITY OF WESTMINSTER

SSICA
STAZIONE SPERIMENTALE PER L'INDUSTRIA DELLE CONSERVE ALIMENTARI

Universitat d'Alacant
Universidad de Alicante

Fraunhofer

celabor

condensia

iris

LUCENSE
RICERCA E SERVIZI PER L'INNOVAZIONE

ARCHA
Laboratori ARCHA S.r.l. unipersonale

bio-mi
SUSTAINABLE SOLUTIONS

TIPA

PARMA

ORGANIK KIMYA
the chemistry between us

Huhtamaki

Kneia

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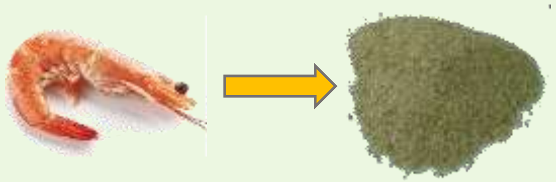
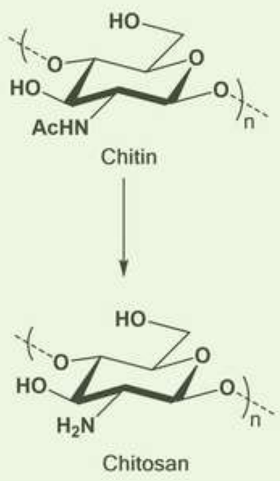
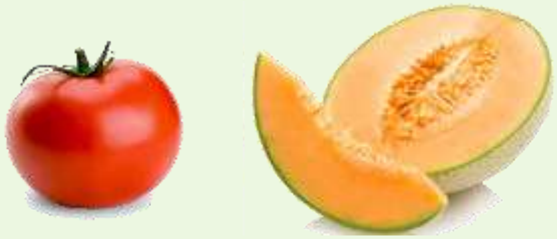
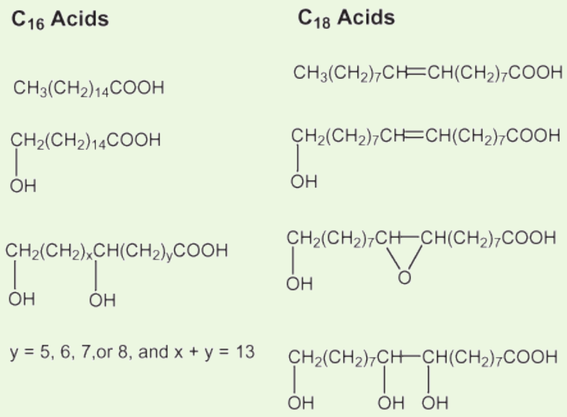
IMBALLAGGI ECO-SOSTENIBILI PER IL CONTATTO ALIMENTARE

ECOAT STRATEGY




HO
O
O
HO
O
AcHN
n
Chitin
HO
O
O
HO
O
H_2N
n
Chitosan





C_{16} Acids

$CH_3(CH_2)_{14}COOH$

$\underset{OH}{CH_2}(CH_2)_{14}COOH$

$\underset{OH}{CH_2}(CH_2)_x\underset{OH}{CH}(CH_2)_yCOOH$

y = 5, 6, 7,or 8, and x + y = 13

C_{18} Acids

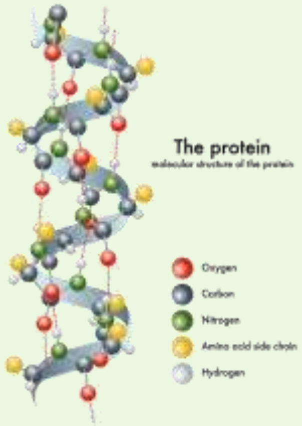
$CH_3(CH_2)_7CH{=}CH(CH_2)_7COOH$

$\underset{OH}{CH_2}(CH_2)_7CH{=}CH(CH_2)_7COOH$

$\underset{OH}{CH_2}(CH_2)_7\underbrace{CH{-}CH}_{O}(CH_2)_7COOH$

$\underset{OH}{CH_2}(CH_2)_7\underset{OH}{CH}{-}\underset{OH}{CH}(CH_2)_7COOH$

Cutin-forming acids


The protein
molecular structure of the protein
Oxygen
Carbon
Nitrogen
Amino acid side chain
Hydrogen

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BIO-BASED INDUSTRIES
Public-Private Partnership

LUCCA, 23 Maggio 2019

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Our Research Team

CISIM

Interdepartmental Center of Materials Science and Engineering

Full Professor

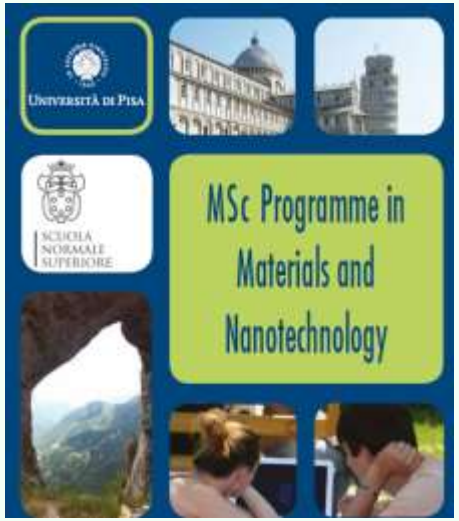
Dott. Ing. Andrea Lazzeri

Associate and Assistant Professors

Patrizia Cinelli – Maria Beatrice Coltelli – Serena Danti

MATERIALS AND NANOTECHNOLOGY

Corso di laurea magistrale



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SCUOLA NORMALE SUPERIORE

MSc Programme in Materials and Nanotechnology

Researchers, Post Docs, PhDs, Research Grants

Francesca Signori

Laura Aliotta

Vito Gigante

Luca Panariello

Bahareh Azimi

Alessandro Vannozzi

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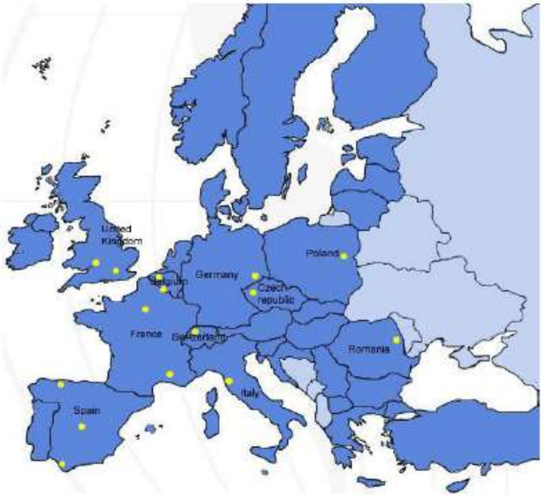
POLYBIOSKIN

ENMat

EUROPEAN NETWORK of MATERIALS RESEARCH CENTRES



www.enmat.eu

President of ENMat:
Dr. Maria Beatrice COLTELLI
Centre for Materials Science and Engineering (CISIM),
University of Pisa, Italy

Benefits for members

- **Contacts** with materials research centres all over Europe
- Platform for **partner search**: find academic and industrial project partners
- **Promotion** and **publicity** of activities and achievements on a European and global stage

Issue 5, March 2018

European Network of Materials Research Centres

ENMat

NEWSLETTER

Breakthrough of new Networking between European Materials Research Centres

Welcome

Welcome to the fifth issue of ENMat Newsletter. ENMat has been founded in September 2005, to create a powerful network of leading Materials Research Centres in Europe. We expect to stimulate beneficial interdisciplinary activities between members of the network as well as to increase the efficiency of the transfer of results from R&D to industry. We also expect to improve opportunities for participation in activities in the frame of EU supported projects.

Highlights in this Newsletter

Description of ENMat members: FRAUNHOFER IKTS (Germany) and IMT Mines Alès/C2MA (France) - *page 2*

CERAMIC MATERIALS RESEARCH IN EUROPE - *page 3*

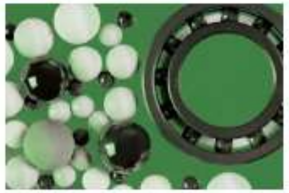
Members

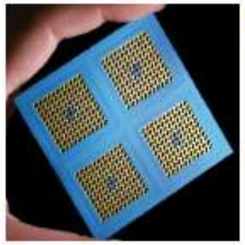
- Centre des Matériaux des Mines d'Alès (C2MA), École des Mines d'Alès, Alès Cedex, France
- Centre for Materials Science and Engineering (CMSE),

ENMAT is open to all multidisciplinary research centres in materials sciences in Western and Eastern Europe.

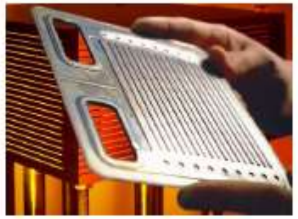
Candidate members have to meet the following criteria:

- Multidisciplinary research centre in materials science: academic or research centre or industrial network dealing with materials in a broader sense





- Having high quality & competences in materials science
- Being active in research & development
- Play a role in materials education (of students and/or industry)
- Being active in technology transfer



- Be prepared to participate in joint projects (also with industry)

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Grazie per la vostra attenzione!!!