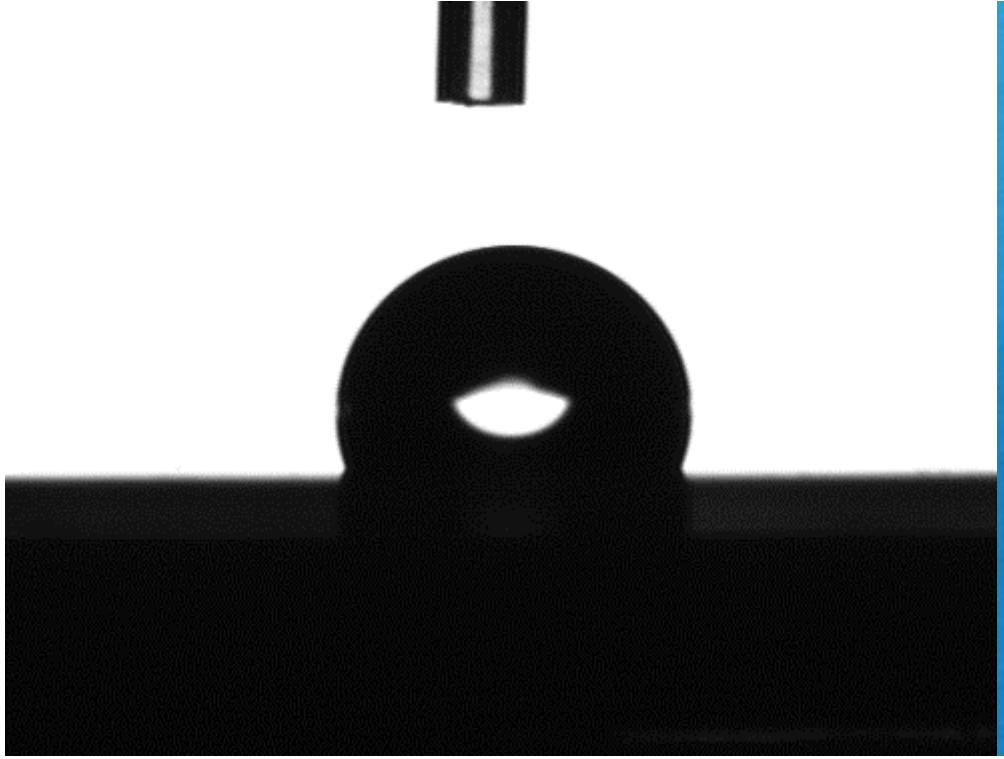
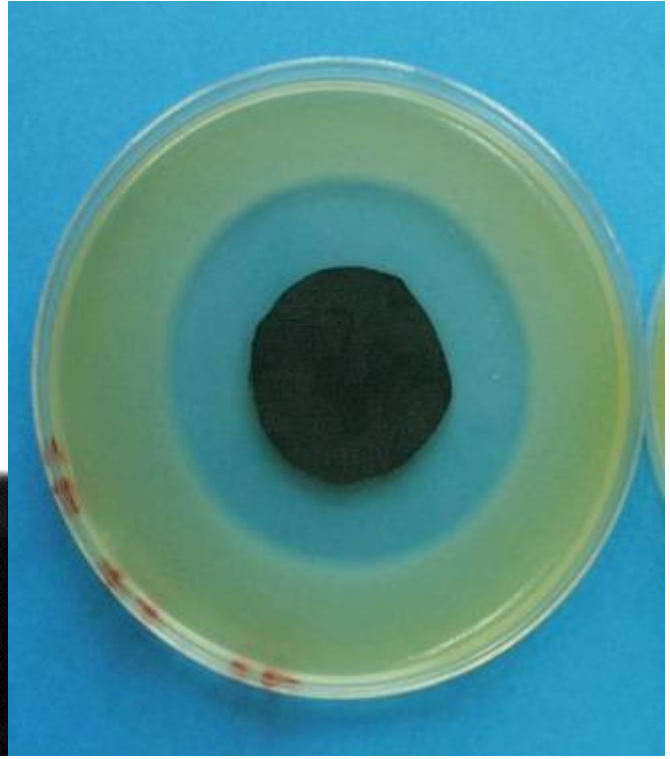




DURATEX

Interreg
France-Wallonie-Vlaanderen

GoToS3
DURATEX

Avec le soutien du Fonds européen de Développement Régional
Met de steun van het Europees Fonds voor Regionale Ontwikkeling

Results of biobased antimicrobial products used for the diffusion process on polyester woven fabric

Pauline GRESSIER, Nemeshwaree BEHARY, Christine CAMPAGNE

David De Smet, Myriam VANNESTE

ensait ROUBAIX
ÉCOLE D'INGÉNIEURS TEXTILE

DURATEX

ensait
ROUBAIX
ÉCOLE D'INGÉNIEURS TEXTILE

150 students graduated /year
Master level textile ENGINEERING

50 Ph D students enrolled/year

Objectifs / Objectives

Interreg France-Wallonie-Vlaanderen

GoToS3
DURATEX

DURATEX

Main goal: Development of **sustainable polyester** textiles with **antimicrobial** properties for **construction** and **architecture**

AIM OF THIS WORK

Test the use of some **bio-based antimicrobial products**

→ Functionnalisation of PET fiber **DIFFUSION METHOD**

→ Test the ANTIMICROBIAL **properties** of the functionalised textiles

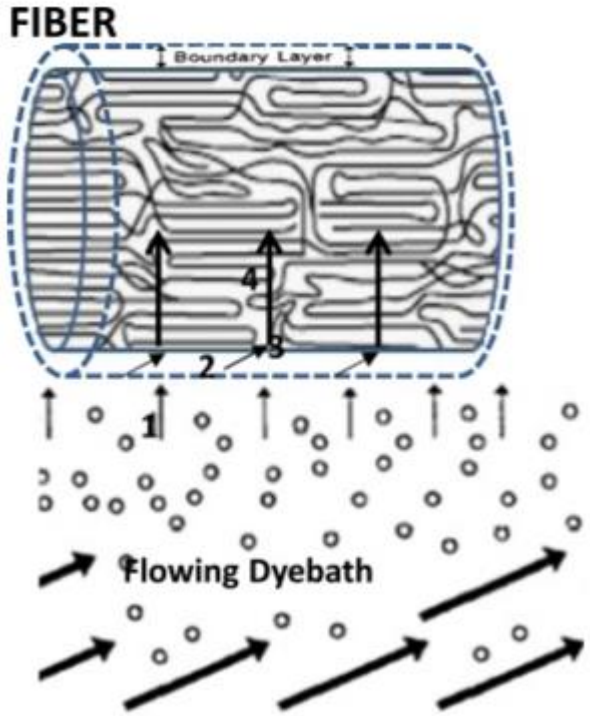
3

Diffusion method (theory)

Slark A.T., Hadgett P.M., 1999. The effect of specific interactions on dye transport in polymers above the glass transition. Polymer 40, 4001–4011.

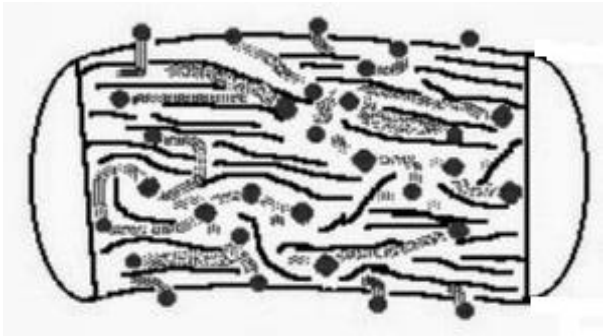
Diffusion

Adsorption



Dyeing and more particularly **dye transport** through PET fibres is **function of both diffusivity and solubility** of the dye in the PET fibre[Slark, 1999].

δ PET Fiber = 21.4 MPa$^{1/2}$



● = **Active molecule**

✓ Solubility parameter δ
δ fiber ≈ δ active molecule

✓ High temperature for a good mobility of PET chains (amorphous zone)
T ≈ Tg

✓ (glass transition temperature)

Active ingredients from essential oils used

Active ingredient alone (10%)	
	Ortho-vanilline / Orthovanillin
	Para-vanilline / Paravanillin
	Cinnamaldehyde / Cinnamaldehyde
	Géraniol / Geraniol

Active ingredient alone (10%)	
	Thymol / Thymol
	Carvacrol / Carvacrol
	Brewtan F / Brewtan F
	Tanal 04 / Tanal 04

(10%) means % by mass relative to the mass of the sample

Formulation de textiles antimicrobiens par diffusion / Formulation of antimicrobial textiles by diffusion

Interreg France-Wallonie-Vlaanderen

GoToS3 DURATEX

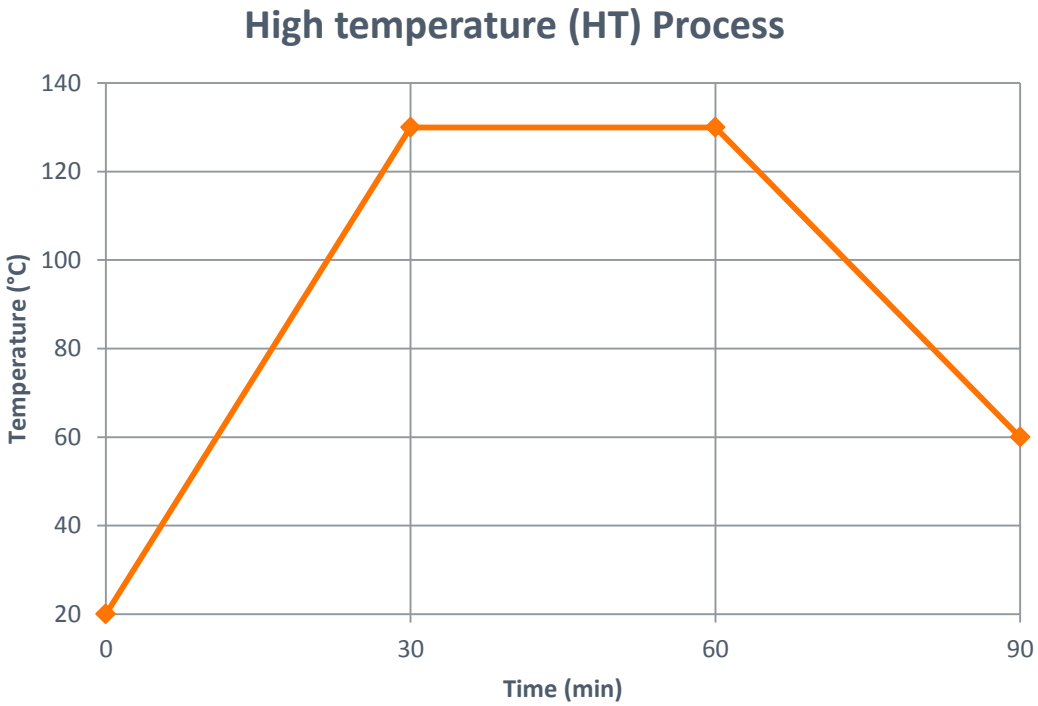
DURATEX

» Procédé de diffusion étudié / Diffusion process

> Haute température (130°C) / High temperature (130°C)

> Autres paramètres / Other parameters
  - pH / pH
  - % éthanol / 5 % of ethanol
  - Rapport de Bain / liquorratio 1/20

> Molécules testées seules et combinées / molecules are tested alone and combined

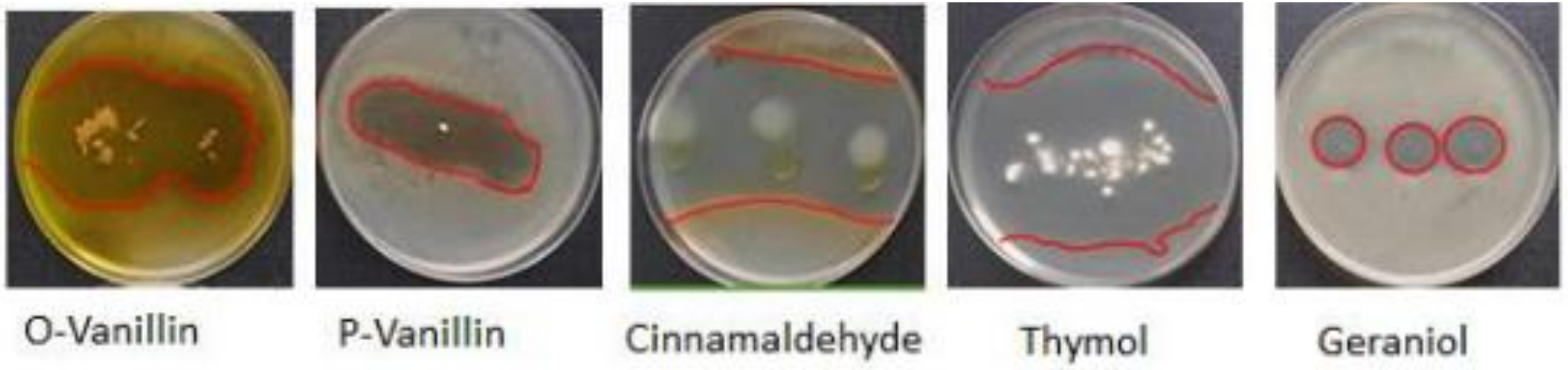


(HTP)Beaker Dyeing Machines used for diffusion process

Thermal stability of the active agents determined from TGA thermographs

Active agent	Temperature range of degradation	Max. Stability Temperature (at 5% mass loss)
O-Vanillin	80°C-200°C	142°C
P-Vanillin	80°C-200°C	162°C
Thymol	80°C-160°C	115°C
Cinnamaldehyde	80°C-200°C	136°C
Geraniol	80°C-215°C	153°C

Antibacterial test (qualitative) against *S. Epidermis (at ENSAIT)*



Spectrophotometric study: Color intensity evaluation before and after removal of physi-sorbed molecules

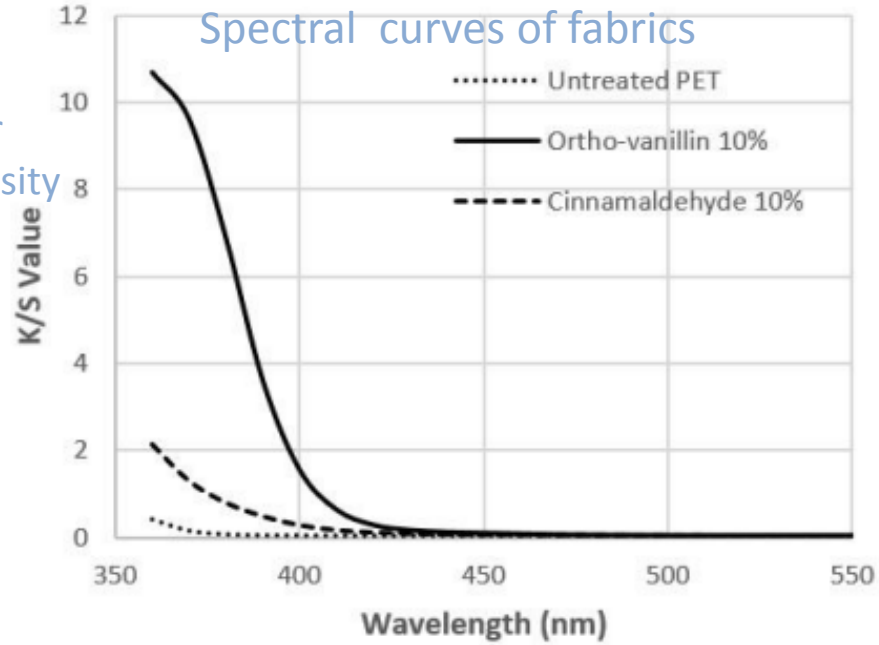
Pale yellowish coloration

Color intensity

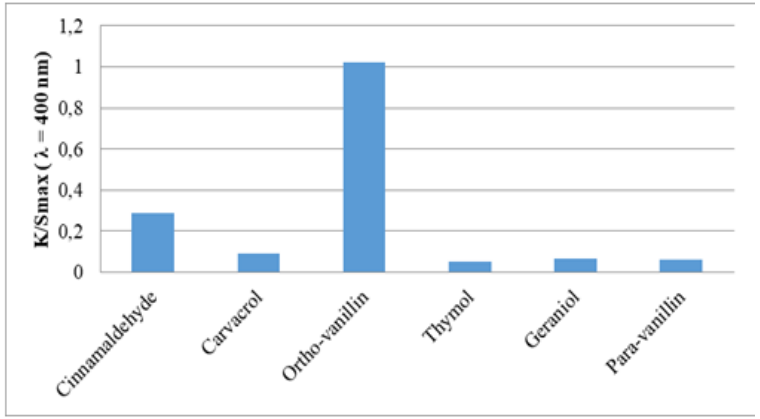
$$\frac{K}{S}(\lambda) = \frac{(1 - R(\lambda))^2}{2R(\lambda)}$$

Color intensity

Spectral curves of fabrics



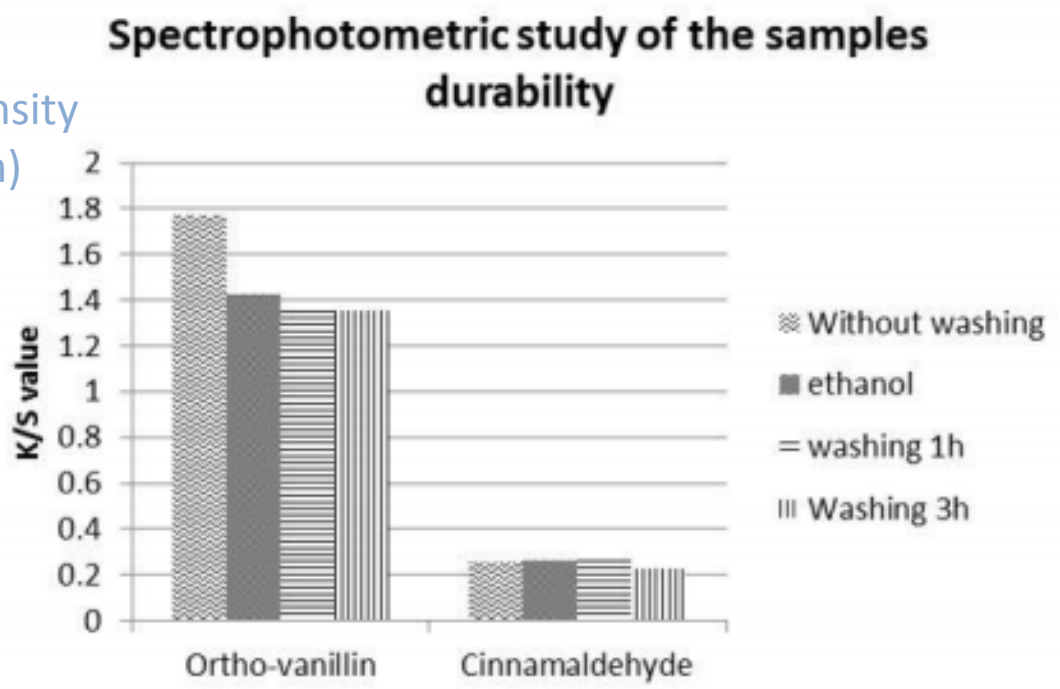
Color intensity(at 400 nm) for various agents



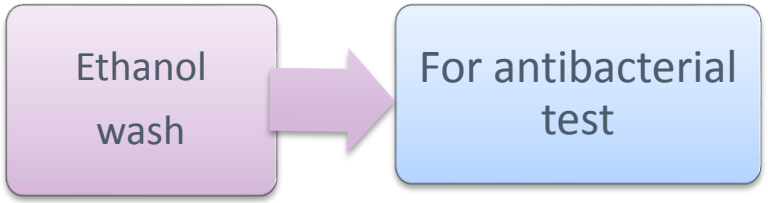
Quantify amount of active agent diffused inside the fiber

Removal of physi-sorbed O. Vanillin (washing with water v/s ethanol)

Color intensity
(at 400 nm)



Choice of method for removal of physi-sorbed active agent :
ethanol wash

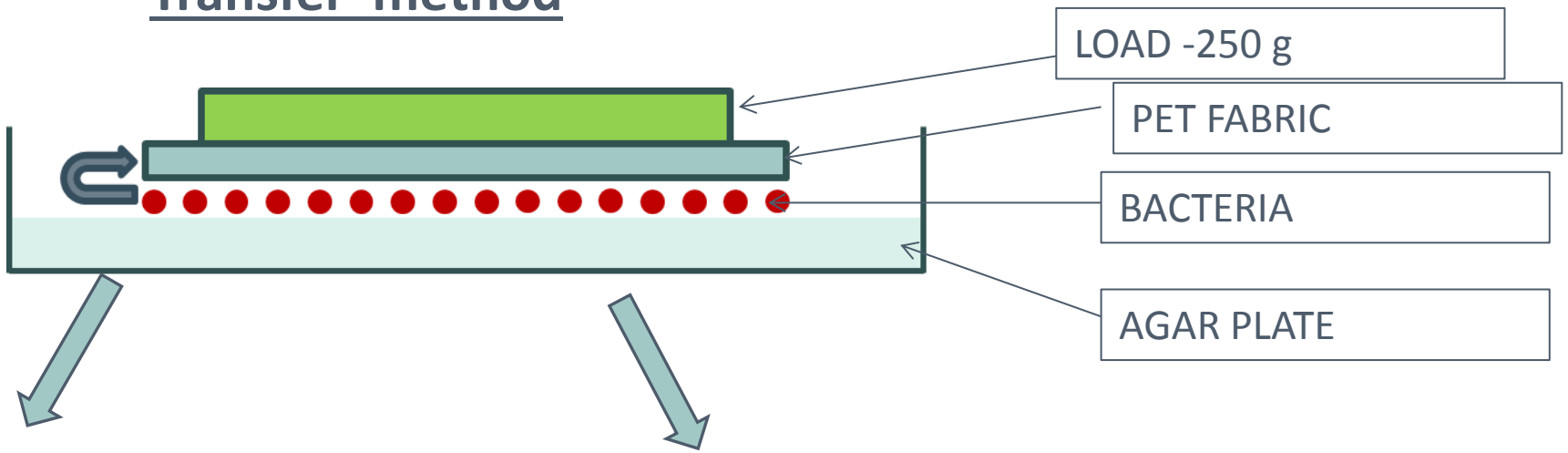


Antibacterial tests

ISO 20743 (2013): QUANTITATIVE

Determination of the antibacterial activity of texile products

- **Transfer method**



LOAD for 1 minute

No incubation

Counting of bacteria

LOAD for 5 minutes

18 – 24 hours of incubation

Counting of bacteria

Bacteria tested

Staphyloccocus aureus

Klebsiella pneumoniae

Calculation of antibacterial activity value

$$A=(\lg C_t-\lg C_0)-(\lg T_t-\lg T_0)=F-G$$

- where
- *A* is the antibacterial activity value;
- *F* is the growth value on the control specimen ($F = \lg C_t - \lg C_0$);
- *G* is the growth value on the antibacterial testing specimen ($G=\lg T_t - \lg T_0$) ;
- $\lg T_t$ is the common logarithm of arithmetic average of the numbers of bacteria, or the amount of ATP, obtained from three antibacterial testing specimens after an 18 h to 24 h incuba tion;
- **lg To** is the common logarithm of arithmetic average of the numbers of bacteria, or the amount of ATP, obtained from three antibacterial testing specimens immediately after inoculation.

« A » value which characterizes antimicrobial activity:

- ✓ **If A > 3 → Strong antibacterial activity**
- ✓ **If 2<A<3 → Significant antibacterial activity**
- ✓ **If A<2 → Insufficient activity**

Testing of washfastness and resistance to ageing (in climatic conditions) of antibacterial samples

Interreg France-Wallonie-Vlaanderen

GoToS3

DURATEX

DURATEX

Wallonie

west-vlaanderen

CENTEXBEL

certech

CETI PRODUCT INSIGHT

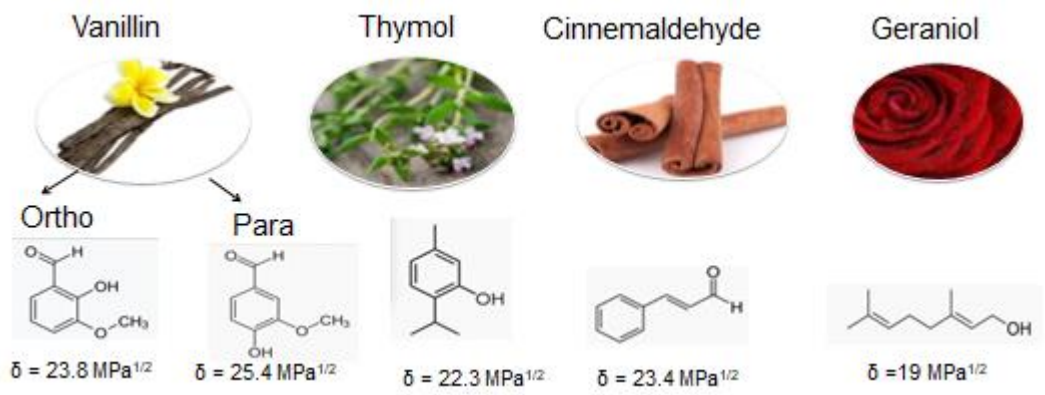
ensait

UCL Université catholique de Louvain

Samples exposed to outdoor climatic conditions

Samples washed during 3 hours at room temperature (to simulate effect of intense raining)

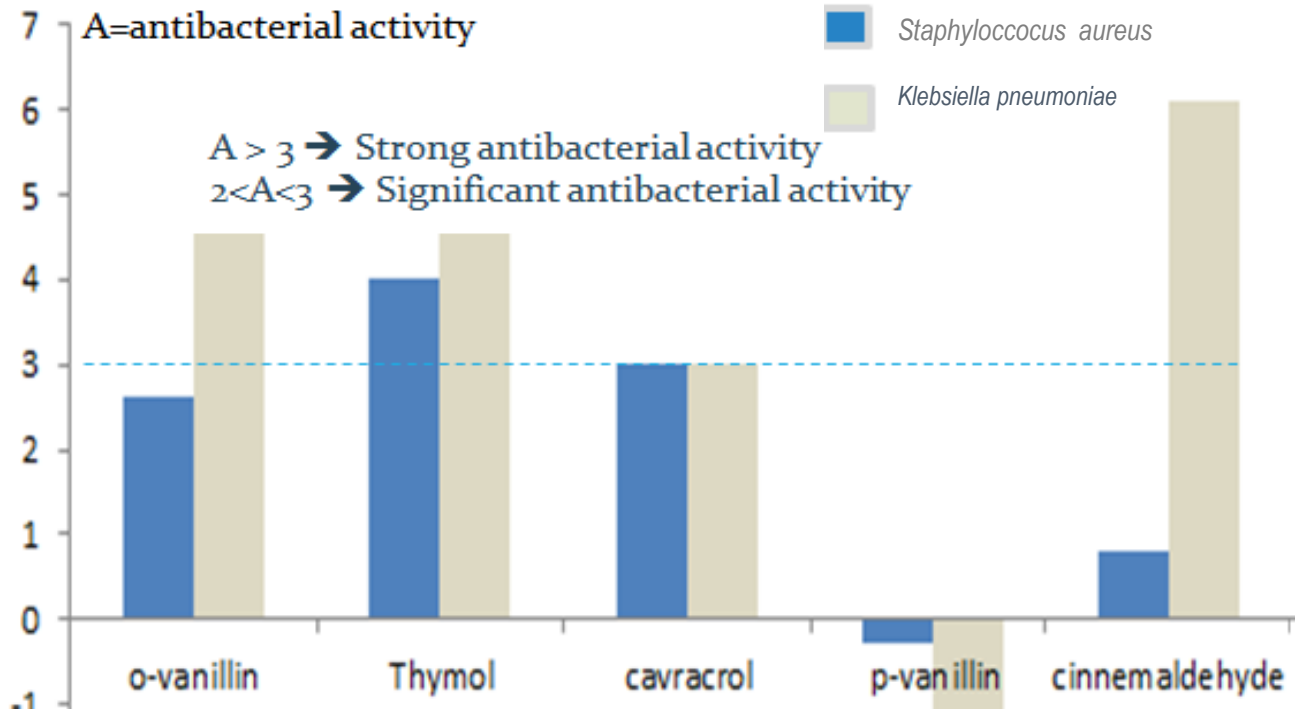
Active ingredients from essential oils used alone



Renewable, Bio-based

δ PET Fiber = 21.4 MPa$^{1/2}$

QUANTITATIVE ANTIBACTERIAL TEST ISO 20743 (2013) on Textiles:



Geraniol

Brewtan

Tanal

A<1


Industrial Crops & Products 136 (2019) 11–20

Contents lists available at ScienceDirect

Industrial Crops & Products

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

Antibacterial polyester fabrics via diffusion process using active bio-based agents from essential oils

Pauline Gressier[a], David De Smet[c], Nemeshwaree Behary[a,b,*], Christine Campagne[a,b], Myriam Vanneste[c]

EFFECT OF WASHING (Antibacterial results)

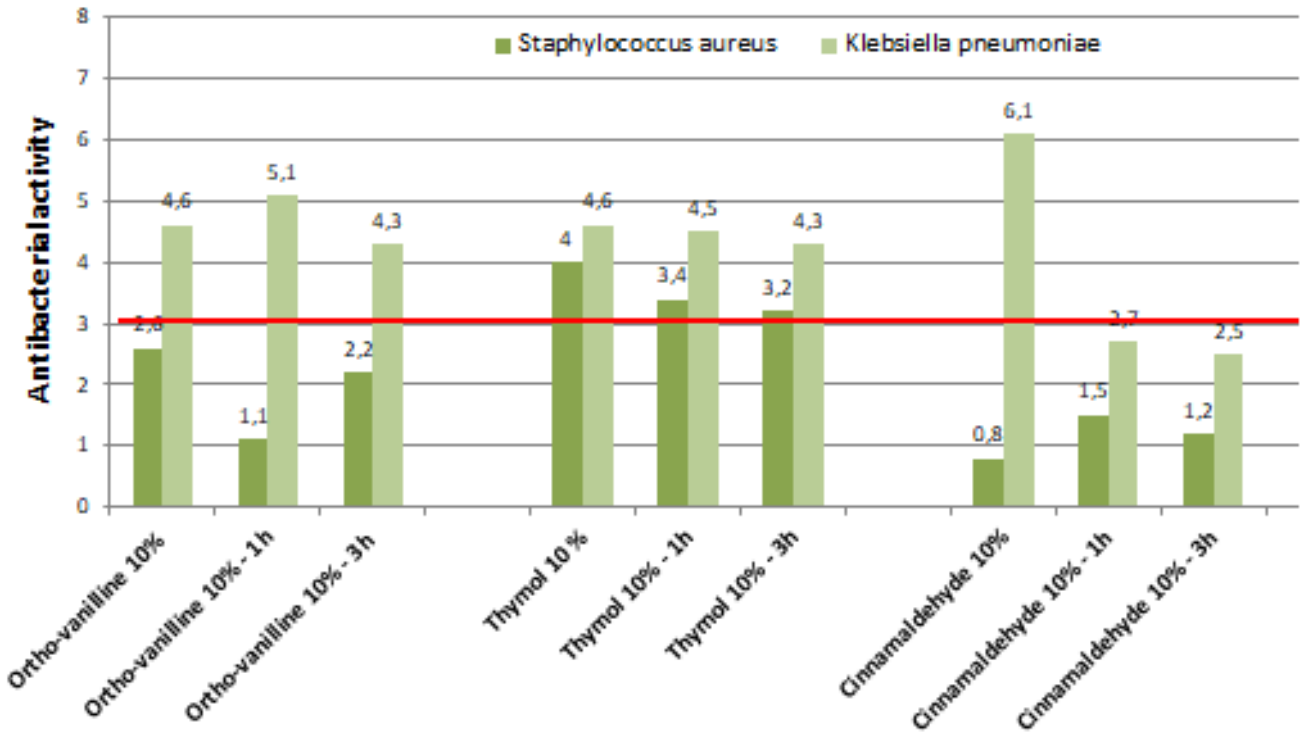
Active ingredients from essential oils used alone

Antibacterial activity

Thymol>Orthovanillin>
cinnemaldehyde

A~3 ⟷ **A~2**

AFTER 3 hour washing



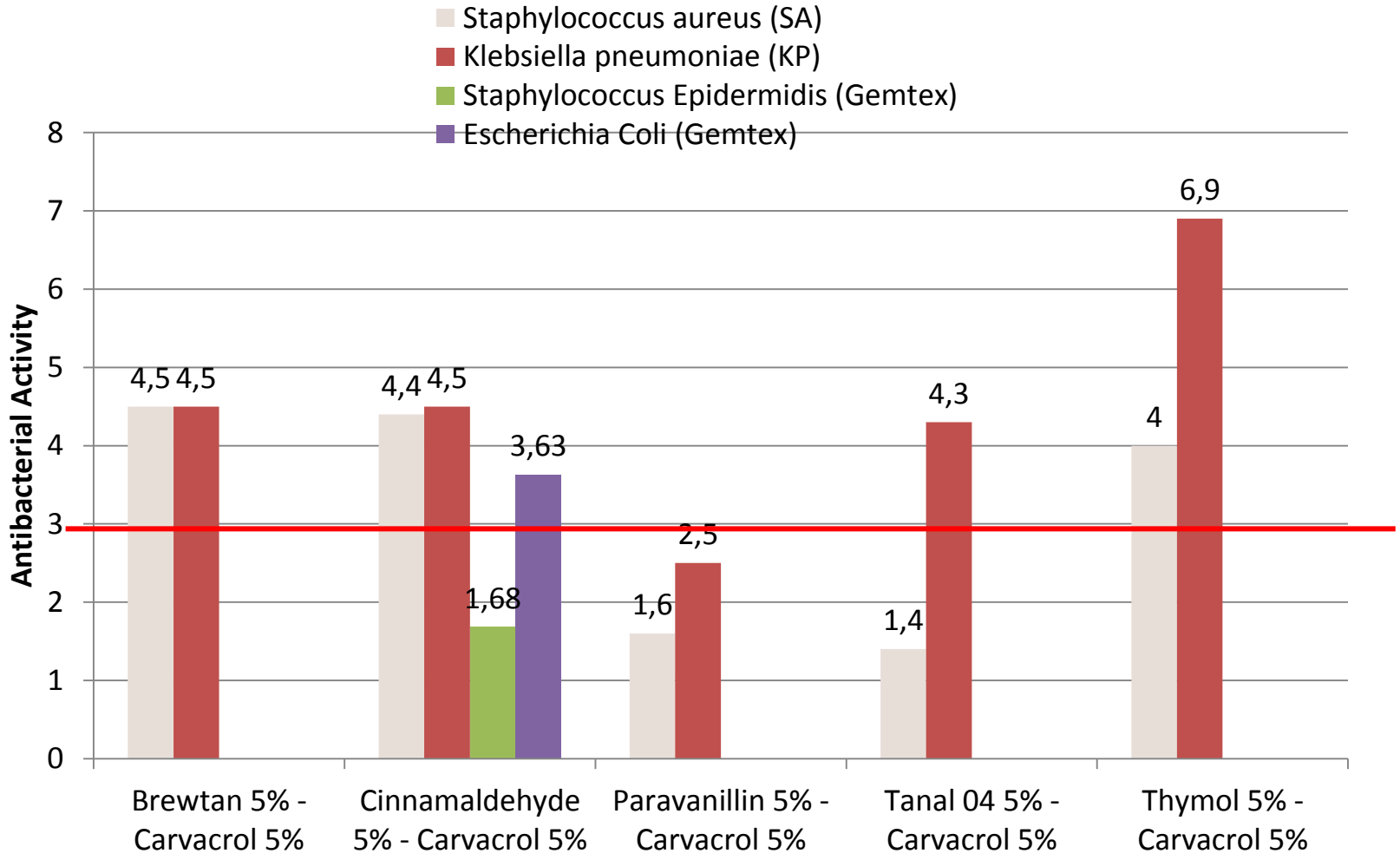
Bilan des principes actifs utilisés en synergie/
Summary of active ingredients used in synergy

Active ingredients used in synergy (5%/5%)
Para-vanilline - Carvacrol
Cinnamaldehyde – Carvacrol
Thymol - Carvacrol
Brewtan F – Carvacrol
Tanal 04 - Carvacrol

Bilan des principes actifs utilisés en synergie/
Summary of active ingredients used in synergy

(Brewtan, Cinnamaldehyde and Thymol)+ cavracrol

A>3 ➔ Strong activity



Essais antibactériens réalisés par CENTEXBEL / Antibacterial tests performed by CENTEXBEL

Washfastness of antibacterial textiles / - Active agent used alone or with synergy wih 5% cavracrol (Synergy study)

Interreg France-Wallonie-Vlaanderen

GoToS3 DURATEX

A = antibacterial activity

Active agent used alone

- **Thymol 10%,**
- **Géraniol 10%**
- **Cinnamaldehyde 10%**
- **Ortho-vanilline 10%**
- **Para-vanilline 10%**
- **Carvacrol 10%**
- **Brewtan F 10%**
- **Tanal 04 10%**

A~3

5% active agent mixed with 5% cavracrol

- Brewtan F - Carvacrol (5%/5%)
- Tanal 04 - Carvacrol (5%/5%)
- Cinnamaldehyde - Carvacrol (5%/5%)
- Para-vanilline – Carvacrol (5%/5%)
- Thymol – Carvacrol (5%/5%)

A->6

AFTER 3 hour washing

NEW RESULTS

A~2

A<1

Antibacterial activity

Thymol>Orthovanillin> cinnemaldehyde

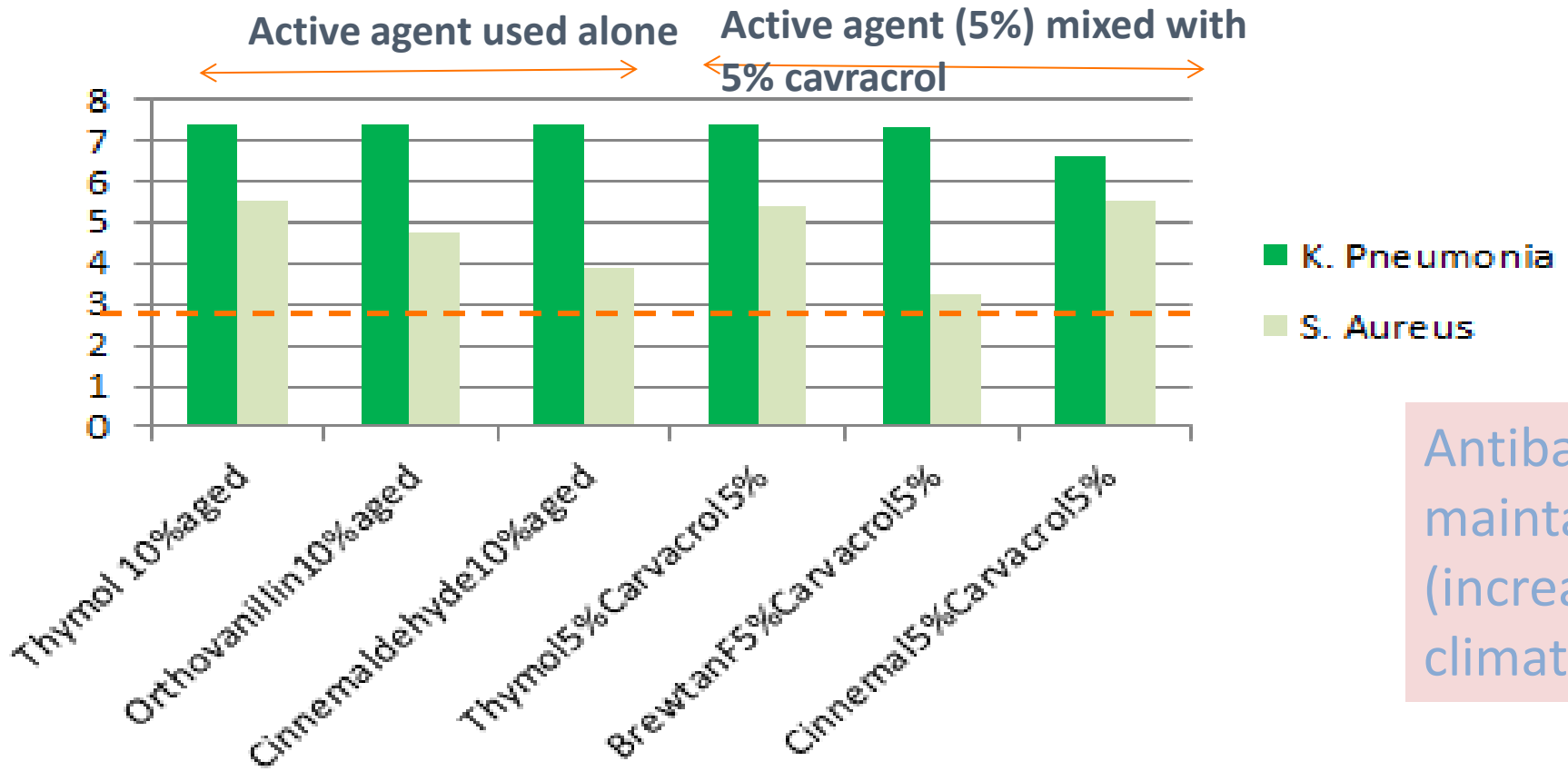
Carvacrol A~1: does not resist to washing

Antibacterial tests performed by CENTEXBEL

EFFECT OF CLIMATIC AGEING

3 months ageing from July to sept 2019.

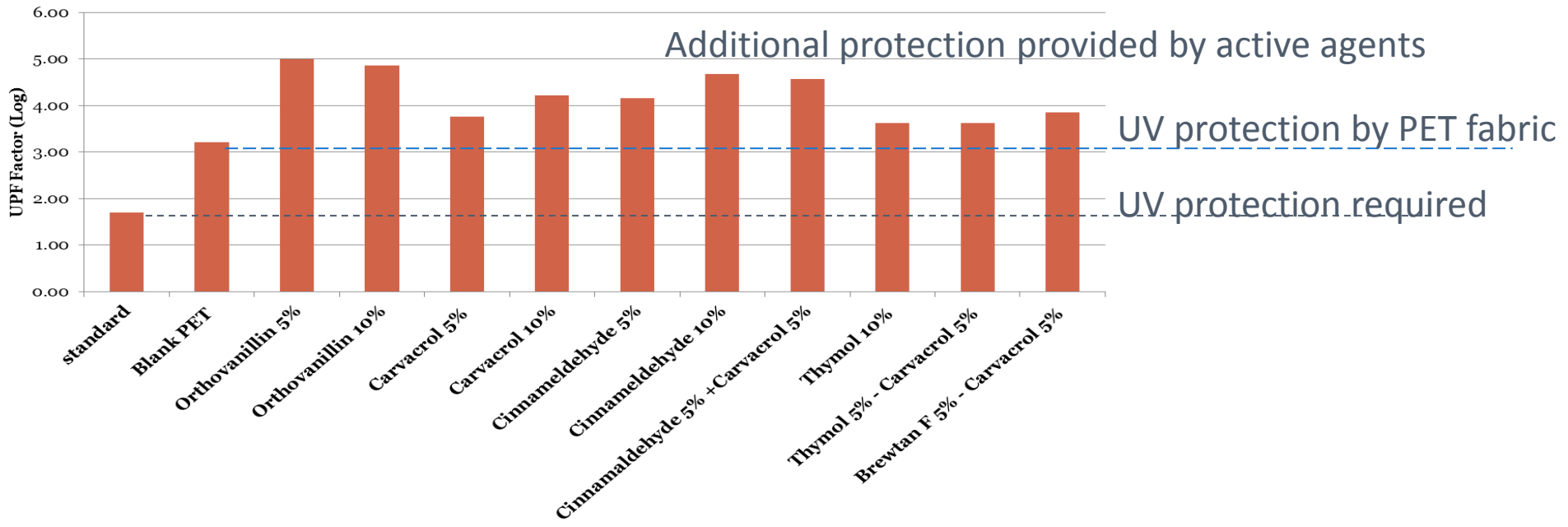
SAMPLES SUBJECTED TO CLIMATC AGEING FOLLOWED BY ANTIBACTERIAL TESTS



Antibacterial activity maintained (increased) after climatic ageing

Résistance aux Ultraviolets / *UV protection*

Résistance aux UV des échantillons / *UV resistance of samples*



Mesures faites par CENTEXBEL / Tests performed by CENTEXBEL

Plus le facteur de protection est grand, plus la résistance de l'échantillon aux UV est importante / *The higher the protection factor is, the more the UV resistance of the sample is important.*

Selecting active agent resistant to washing and climatic ageing- Toxicity?

Antibacterial activity

Thymol>Orthovanillin> cinnemaldehyde

(most resistant to washing and climatic conditions)

A~3 ⟷ **A~2** **(Thymol and orthovanillin)**

AFTER 3 hour washing

maintained after climatic ageing

Toxicity issue of each active agent.

- Corrosive to metals
- Skin corrosion
- Serious eye damage

- Acute toxicity
- Skin and eye irritation
- Skin sensitisation

Hazardous to the aquatic environment:
- Acute hazard
- Chronic hazard

Ortho vanillin	X	X	
Para vanillin		X	
Thymol	X	X	X
Cinnamaldehyde		X	
Geraniol	X	X	
Silver (Ag^+)			X Very toxic to aquatic life with long lasting effects

MOLECULE selected

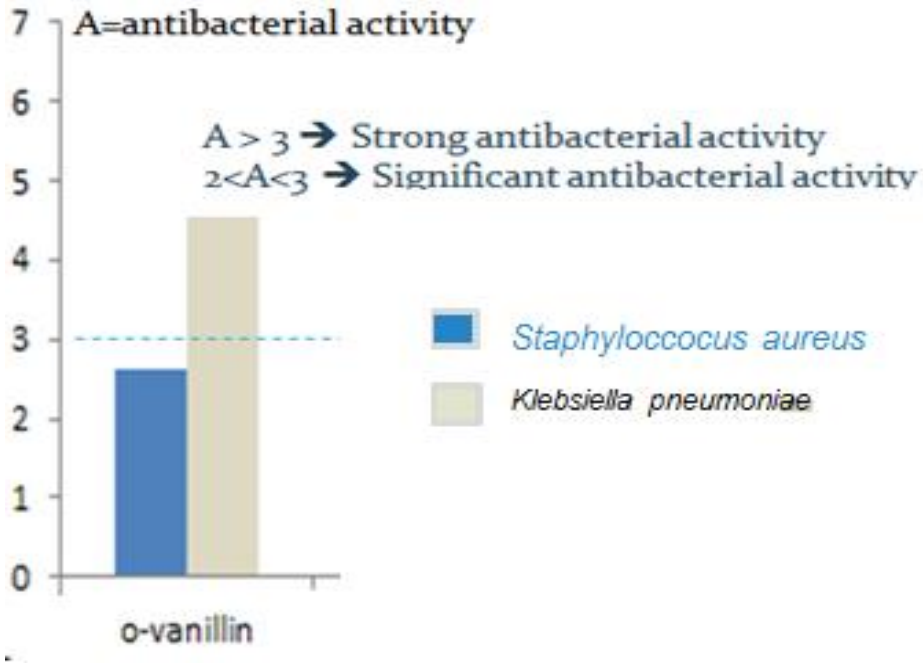
Ortho-vanillin

DIFFUSION PROCESS OPTMISATION BY MICROWAVES

O. Vanillin alone

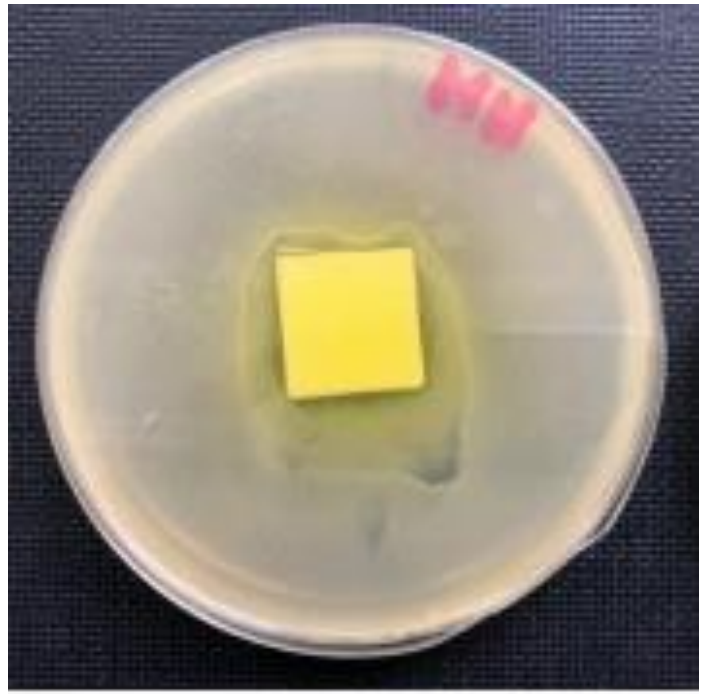
ISO 20645 (2004):
QUALITATIVE TEST –
Agar diffusion plate test

Polyester with 10% owf Vanillin
using exhaustion method
K/S400nm = 1.6
K/S 360 nm= 11



ISO 20743 (2013): QUANTITATIVE

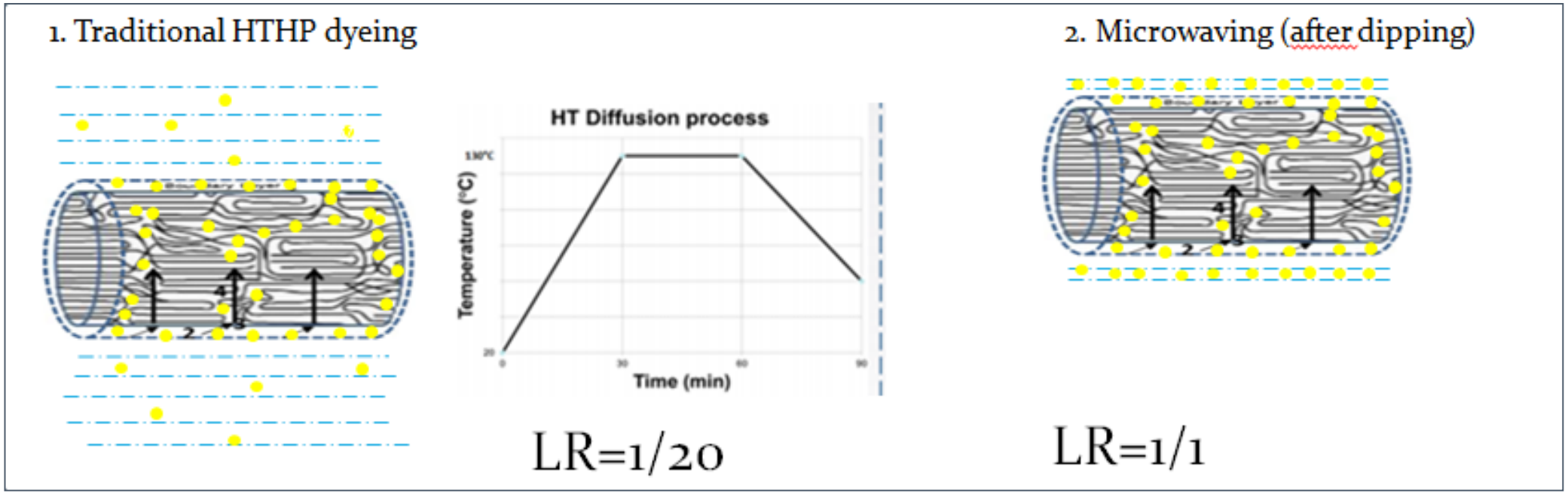
Polyester + Vanillin
using MICROWAVE
K/S400nm = 2
K/S 360 nm= 12



NO bacterial growth (*Staphyloccocus Epidermis*) under fabric

- Environmental consideration:

- O. vanillin diffuses inside the PET fber using the two methods



Liquor ratio : 1/20 and 1/1 : Water Consumption

Energy Consumption

Time : 90 min (HTP) v/s 3 min (MW)

LCA analysis in future work

Functionalisation of polyester fabric with bio-based antibacterial Ortho-vanillin using microwaves

Interreg France-Wallonie-Vlaanderen

GoToS3

DURATEX

A. Microwave

Electromagnetic energy → thermal energy

Boost the dye fixation process / Increase the diffusion.			
Excite	Water molecules	Polyester PET chains	O-V molecules

Type	7890 from SEVERIN for household cooking
Power(W)	125 / 250 / 400 / 550 / 700
Time(min)	0-10 / 15 / 20 / 25 / 30 / 35

Experimental condition	
Power(W)	250 / 400 / 550 / 700
Time(min)	0-8

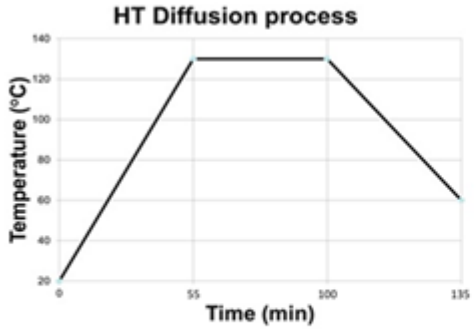


Figure 1a: Time-temperature diagram of the HTHP process

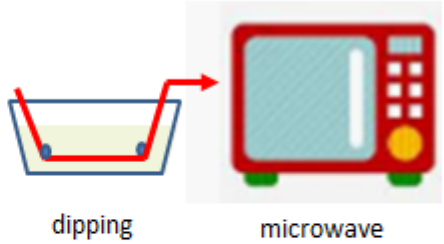
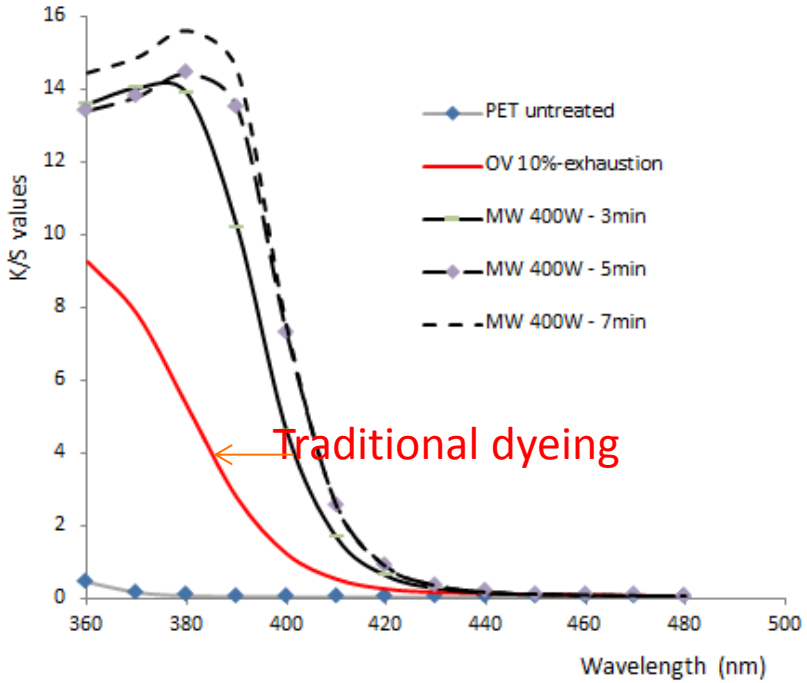


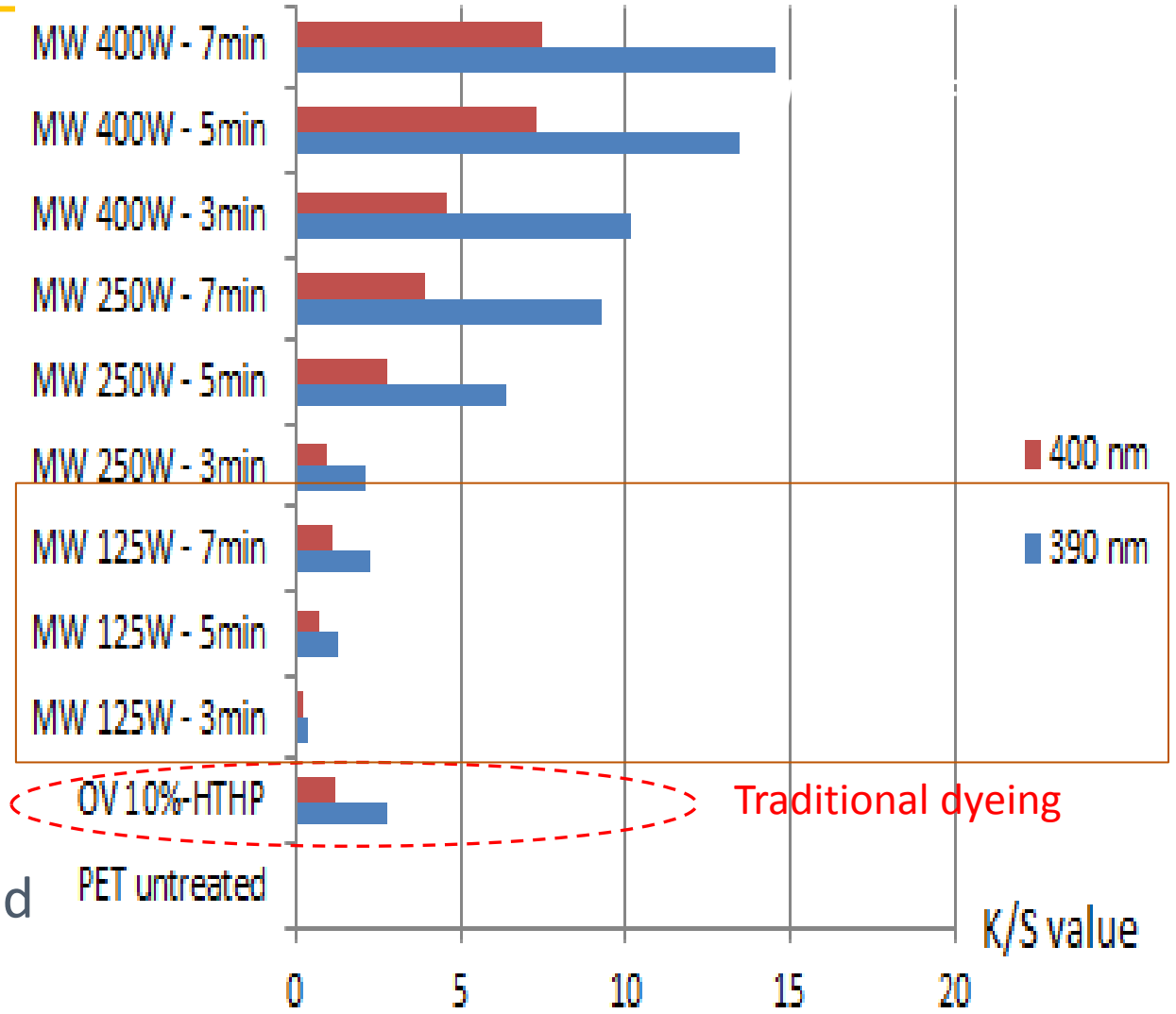
Figure 1b: Dip/microwave process

- Color evaluation after removal of physi-sorbed molecules

(Spectral Curves)

MICROWAVE AT 400 W





O. Vanillin diffusion greatly enhanced using low power microwave

Conclusions

Bio-based active agents used alone, or in combination with cavracol, yield antibacterial textiles resistant to climatic changes (A>3 in most cases)

Some did not resist to 3 –hour washing (A decreased)-only thymol and vanillin showed highest activity

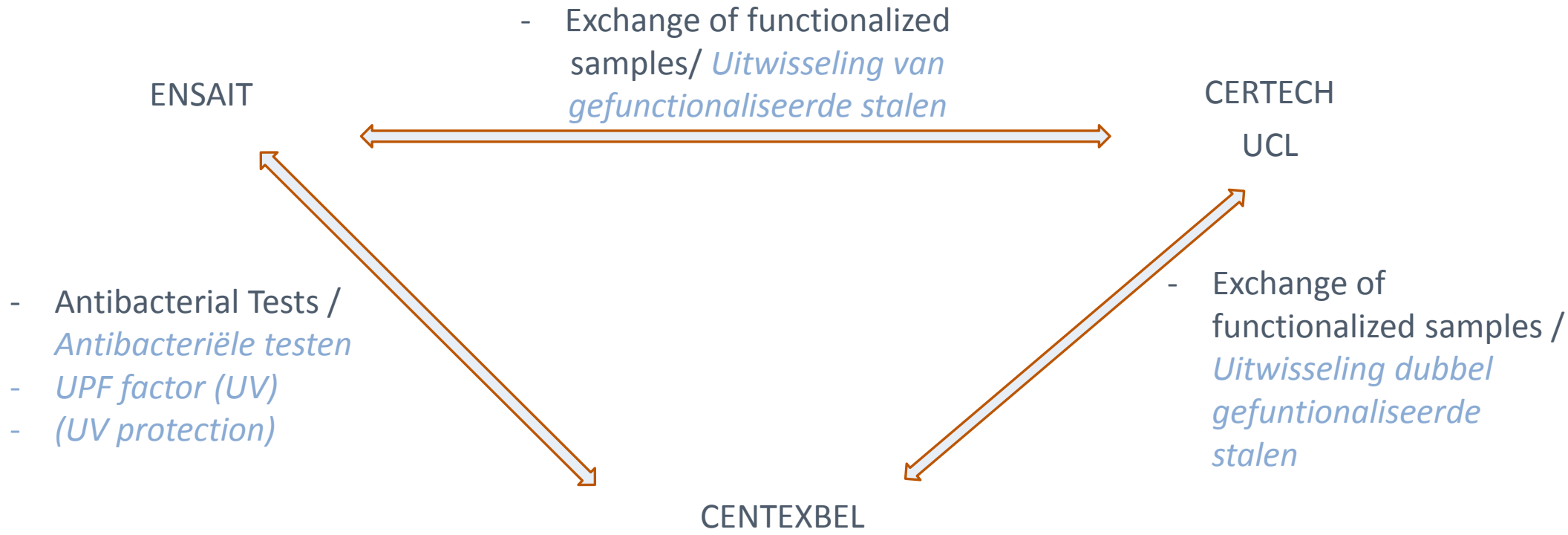
All of them improved UV protection

Some can cause skin sensitization, but can be used for architectural applications

Microwaves can be an alternative eco-friendly method to traditional diffusion method used for dyeing (water and energy used)

Interactions between DURATEX partners

Interacties tussen partners



- Mails réguliers/ *Regelmatig mails uitwisselen*
- Réunions entre les différents partenaires notamment Kick-off et réunion du 10.04.2018/ *Vergaderingen tussen partners en kick-off vergadering (10/04/2018)*

References

Slark A.T., Hadgett P.M., 1999. The effect of specific interactions on dye transport in polymers above the glass transition. Polymer 40, 4001–4011.

Hansen Solubility Parameters: A User's Handbook, Second Edition, Charles M. Hansen, June 15, 2007 by CRC Press, ISBN 978084937248

Vanillin, a potential carrier for low temperature dyeing of polyester fabrics », 27 octobre 2016. http://www.sciencedirect.com/science/article/pii/S095965261200688 9.

Yitzhak Marcus, The Properties of Solvents, vol. 4, England, John Wiley & Sons Ltd, 1999, 239 p. (ISBN 0-471-98369-1)

Microwave Irradiation and its Application in Textile Industries, Karima Haggag,, National Research Center, Dokki, Cairo, Egypt , Science Publishing Group 548 Fashion Avenue New York, NY 10018

Conférences internationales et publication/ *Internationale conferenties en publicaties*

Interreg France-Wallonie-Vlaanderen

GoToS3 DURATEX

DURATEX

Conférences internationales/ Internationale congressen

ORAL PRESENTATION –AUTEX 2019

Proceedings of the 19th World Textile Conference - Autex 2019

Home > Archives > 2019: Autex > Advanced dyeing/printing techn & systems

4B3_0259_ FUNCTIONALISATION OF POLYESTER FABRIC WITH BIO-BASED ANTIBACTERIAL ORTHO-VANILLIN USING MICROWAVES

P. Gressier
Nemeshwaree Behary
C. Campagne
Published Jul 10, 2019

https://ojs.ugent.be/autex/article/view/11501

POSTER PRESENTATION

Publication/ *Publicatie*


Industrial Crops & Products 136 (2019) 11–20

Contents lists available at ScienceDirect

Industrial Crops & Products

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

ELSEVIER

Antibacterial polyester fabrics via diffusion process using active bio-based agents from essential oils

Pauline Gressier[a], David De Smet[c], Nemeshwaree Behary[a,b,*], Christine Campagne[a,b], Myriam Vanneste[c]


ISSN: 2641-192X

DOI: 10.33552/JTSFT.2020.04.000597

Journal of Textile Science & Fashion Technology

Iris Publishers

Research Article

Copyright © All rights are reserved by Nemeshwaree Behary

Antibacterial and Multifunctional Polyester Textile Using Plant-Based Cinnamaldehyde

Nemeshwaree Behary[1,2]*, David De Smet[3], Christine Campagne[1,2] and Myriam Vanneste[3]

[1]ENSAIT, GEMTEX - Laboratoire de Génie et Matériaux Textiles, F-59056 Roubaix, France

[2]University of Lille, F-59000 Lille, France

[3]CENTEXBEL, Technologiepark 7, BE-9052 ZWIJNAARDE, Belgium