Wet Lamination of Micro-Fibrillated Celluloses on Board

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Outline

- Introduction
  - Packaging and Microfibrillated Celluloses
- The MFC wet lamination concept
  - MFC used
  - First results
- Performances of MFC wet laminated board
  - Adhesion of the MFC layer
  - Barrier performances
- Conclusions

OUTLINE 1/4

Introduction

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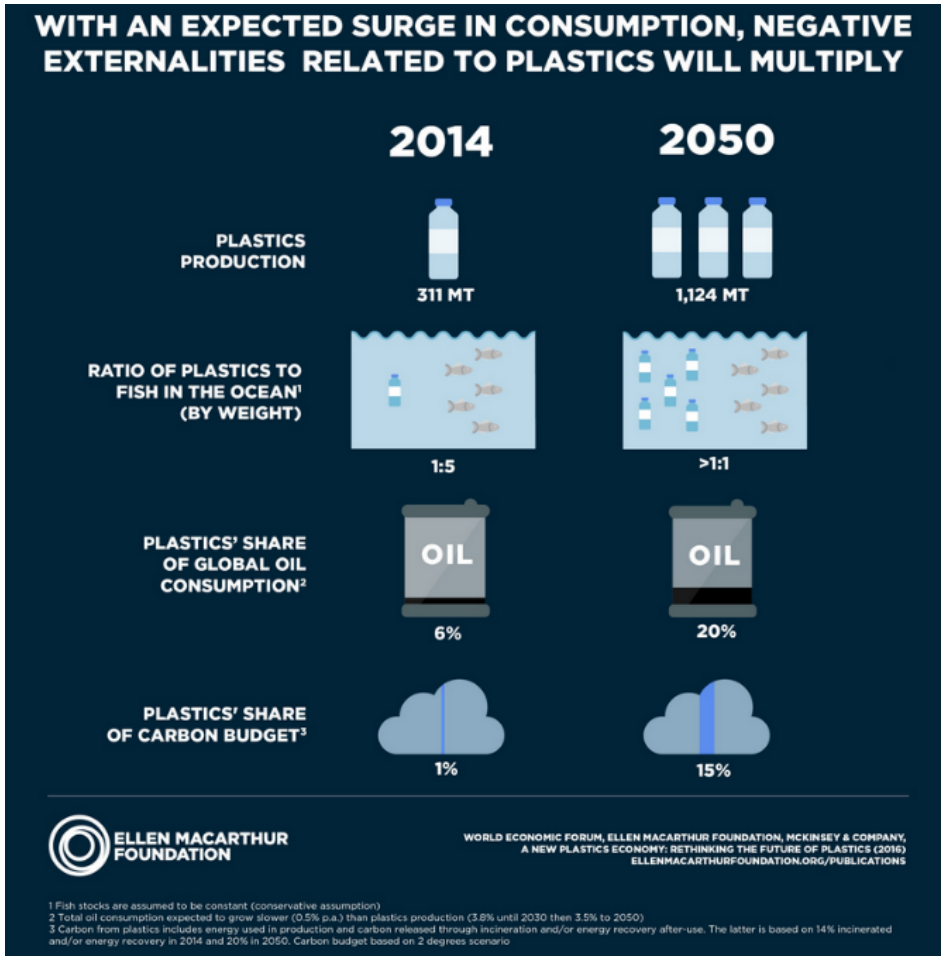
Main issues with Packaging Materials

- **Advantages**
  - Protection
  - Transport/Storage
  - Information/Use
  - Marketing
- **Drawbacks**
  - Depletion of resources
  - Costly/Misleading
  - Might be harmful
  - End of life



CTP’s strategy for packaging materials

Develop ligno-cellulosic materials

Better Barrier

Lighter Stronger

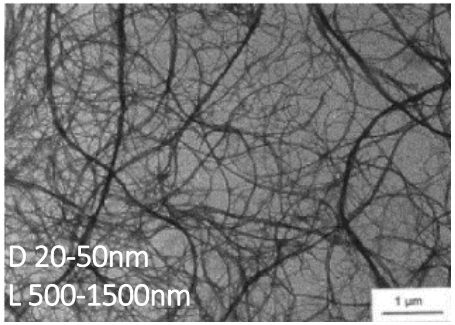
3D shapes

Safety and Food Contact

LCA / Recyclability / Biodegradability

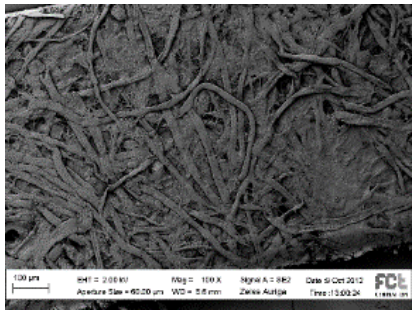
Microfibrillated Celluloses

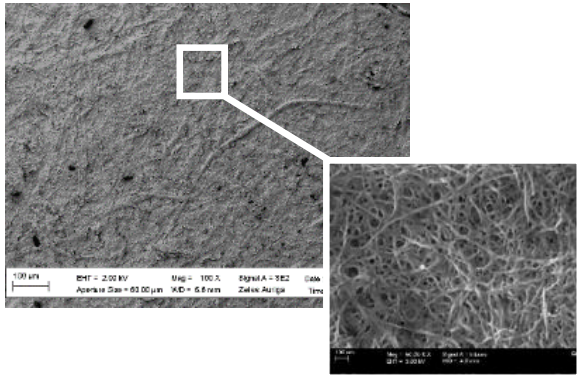
Produced from Cellulosic fibres



Sources :
Guézénnec C., PhD Thesis, CTP-LGP2, 2012
Raynaud S., PhD Thesis, CTP-LGP2, 2017

Building block for Materials





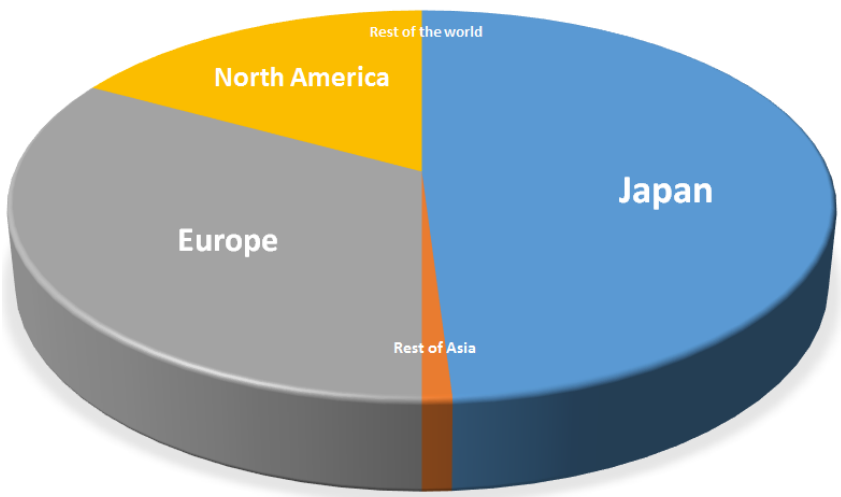
Advantages

Bio-based, Biodegradable, Recyclable in conventional paper recycling streams

Reported to bring excellent barrier to grease, oxygen contaminants, aromas

Microfibrillated Celluloses

- **Availability of CNF/MFC**
  - Ab. 20 000t/y



Source FUTURE MARKETS Tomorrow's technology, Today

The Global Market for Cellulose Nanofibres to 2030, Nov. 2018

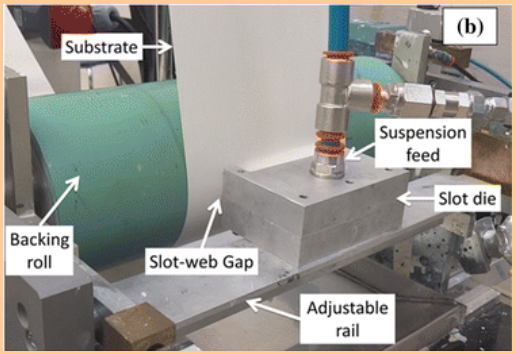
- **Some issues to overcome to make materials**

MFC suspension at 2% solids

Sources :
Raynaud S., PhD Thesis, CTP-LGP2, 2017

  - Low solids
  - Highly viscous
  - Cost

Processes reported to deposit a layer of MFC

	Coating	Wet end deposition	Extrusion coating / Lamination
	 *Picture Source : Cellulose, March 2017, Volume 24, Issue 3, pp 1247–1260 | Substrate role in coating of microfibrillated cellulose suspensions| V. Kumar, V. Koppolu, D. Bousfield, M. Toivakka*	 *Picture Source : Final Publishable Summary Report, Flexpakrenew EU Project, NMP3-SL-207810, D. Guérin, August 2011*	 *Picture Source : Vaha-Nissi, M., Vartiainen, J., Koivula, H., Räisänen, H., Lampi, A-M., Ragni, P., ... Harlin, A. (2017). Cellulose nanofibrils in bio-based multilayer films and pouches. Flexible Packaging Conference, FlexPackCon, Tampa, United States*
+	• Easy to use • Process adjustments are needed	• Use of the dewatering capability of the fourdrinier	• Excellent barrier
-	• CW>1gsm overpasses coater drying capabilities • Poor barrier at low coat weight • Drying costs	• Filtration through the wet mat • Adhesion to the press rolls • Decrease of the press section dewatering?	• Brittleness of thick M/NFC films • Use of a specific process for MFC film/deposition • Material/Energy cost • % of Cellulose in the multilayer

OUTLINE 2/4

The MFC wet lamination concept

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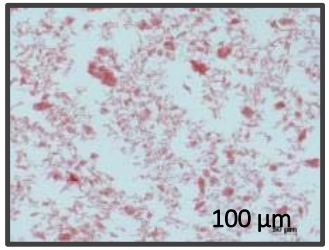
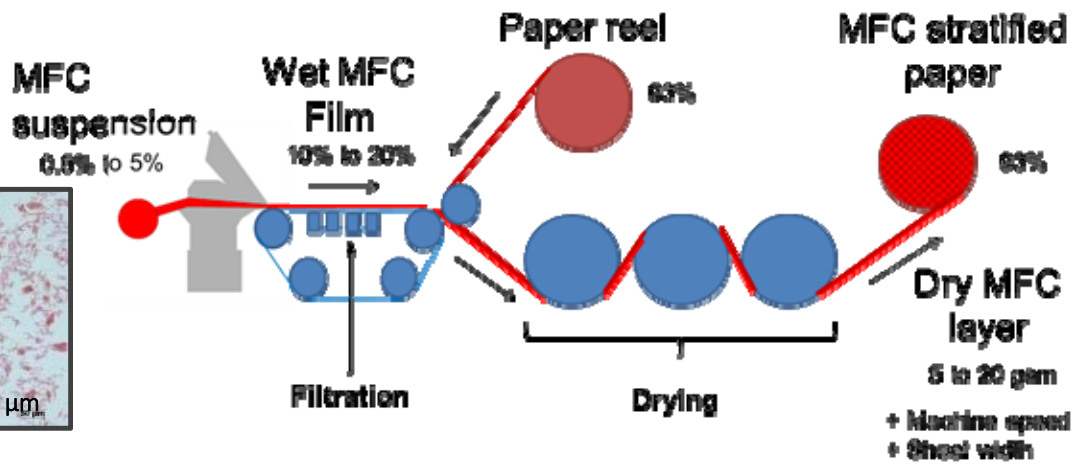
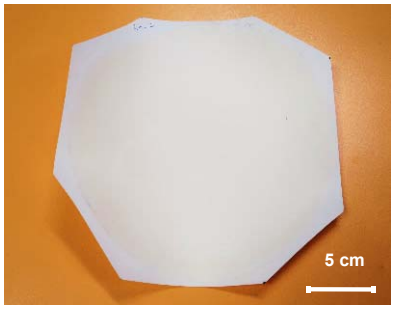
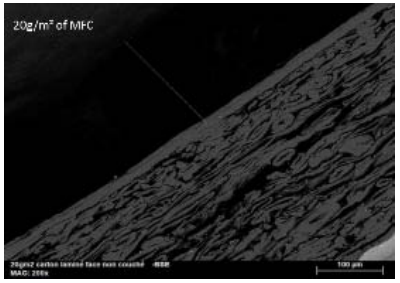
The MFC wet lamination concept

- Process and device for manufacturing a laminated material comprising a fibrillated cellulose layer
  - WO 2016174348 A1
  - D. Guerin, Y. Rharbi, P. Huber, V. Meyer - CTP/CNRS

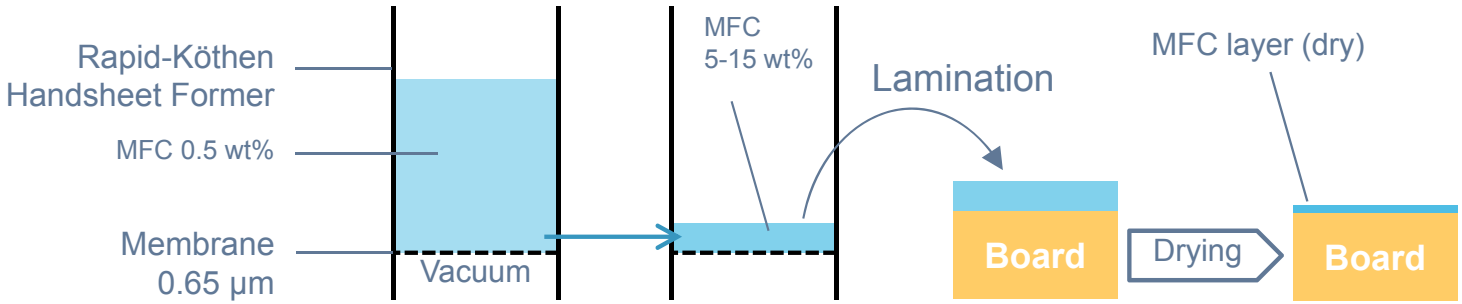




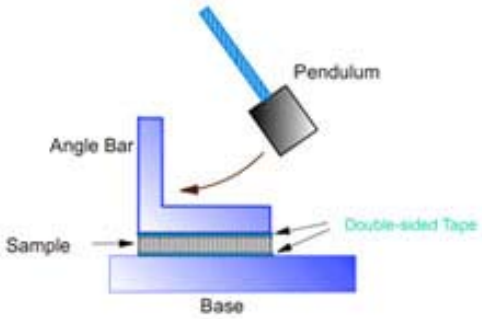




First Results



- Wet laminated on a several substrates
- Use of MFC produced @CTP from BBK
- Cohesion and Adhesion tests (Scott Bond)



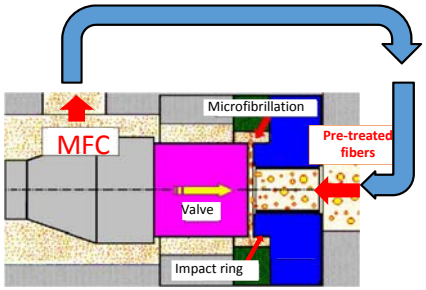
MFC production

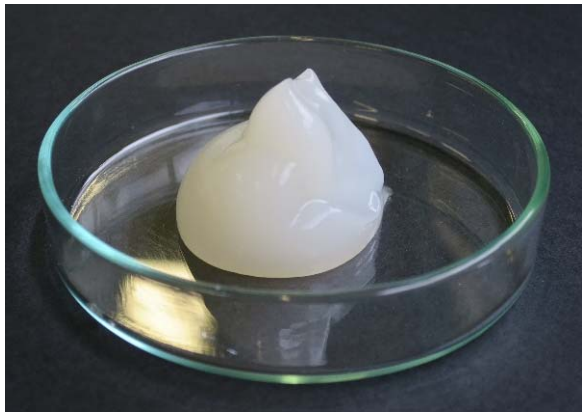
1- Pre-treatment
Endoglucanase + 2 refining steps

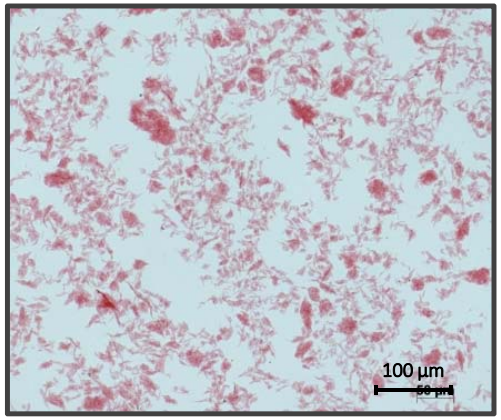
2- Homogenization at high pressure
6 passes at 2-3% consistency

Bleached Birch Kraft Pulp

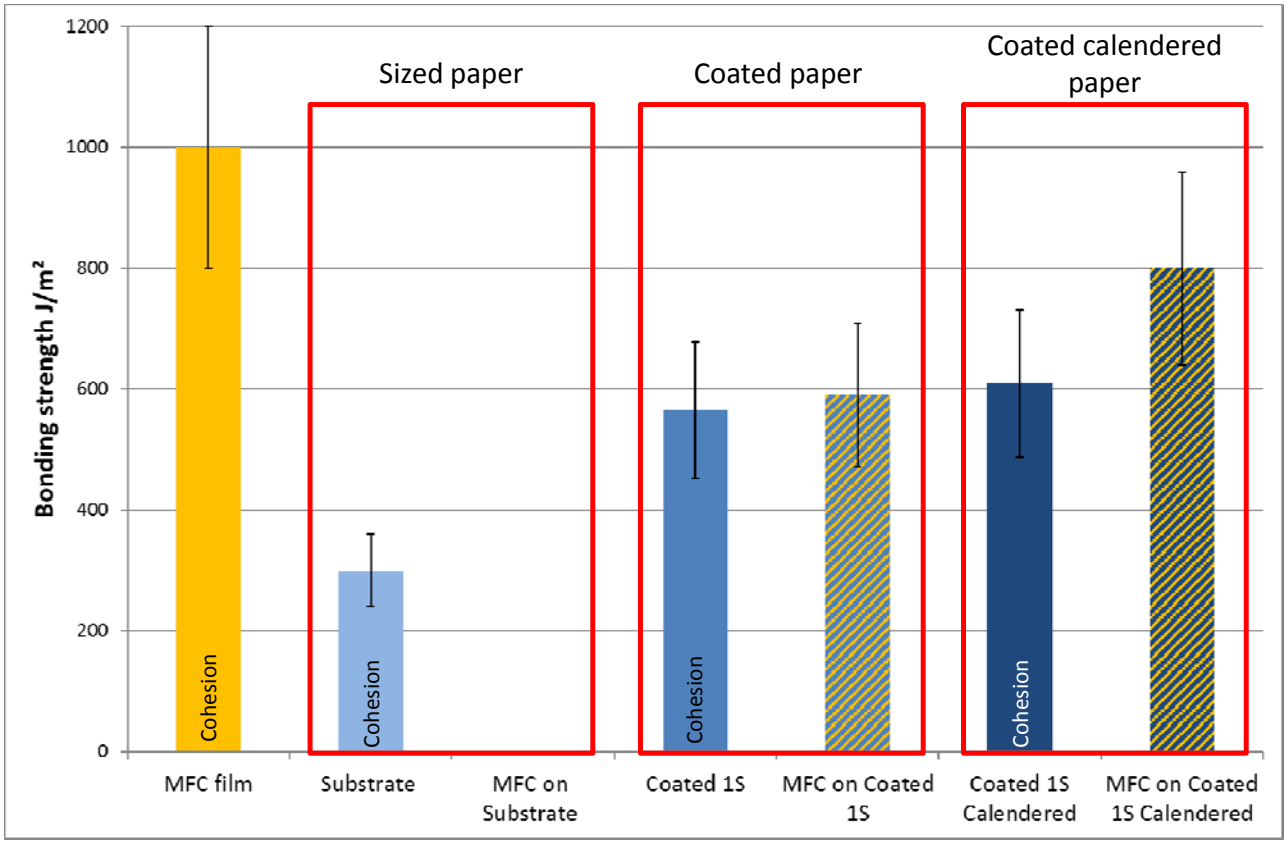
Pre-treated fibres







First results of MFC wet lamination

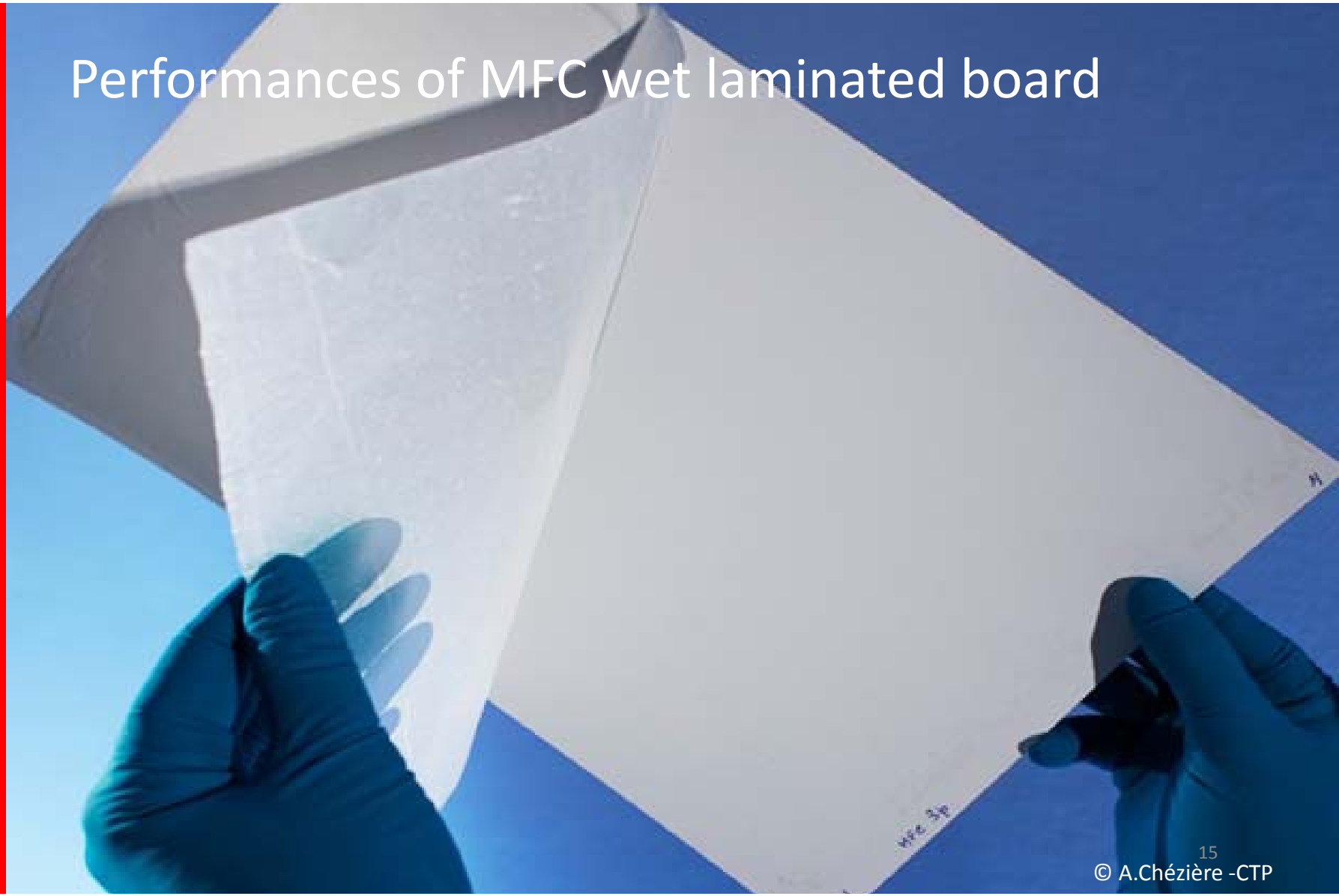


Preliminary conclusions

- Cohesive energy of MFC film is extremely high
- MFC films adhesion strongly depends on paper substrate
  - Surface energy and roughness matters
- In most of the cases the failure occurs in the bulk of the paper and not at the interface
  - Cohesive failure of the material and not really adhesion
- The highest adhesions are obtained on coated and calendered papers
  - Other interactions than hydrogen bonds prevail
  - Presumably Van der Waals and Coulombian forces

OUTLINE 3/4

Performances of MFC wet laminated board



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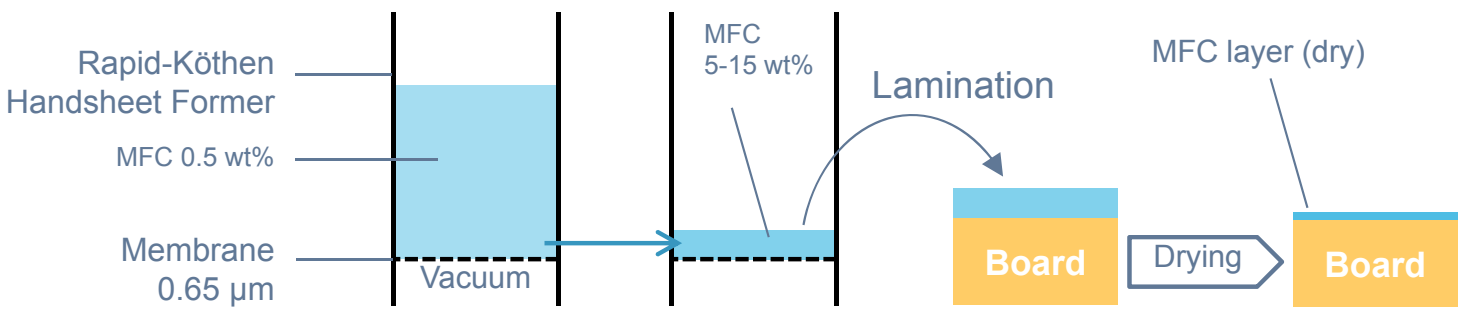
Influence of MFC thickness

- Work achieved by S. Raynaud during his PhD
  - Use of MFC produced @CTP from BBK
  - Wet laminated on a 190gsm SBB
    - 10gsm, 20gsm, 30gsm, 40gsm
  - Adhesion and Barrier performances



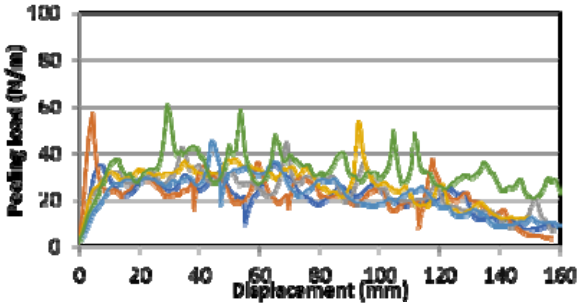
Influence of MFC thickness on the adhesion

Scott Bond test

Tappi method T 569

MFC deposit	Delamination energy (J/m²)	Comment
0g/m²	158	Failure in the board middle ply Adhesion of Film>Board cohesion
10g/m²	167	
20g/m²	146	
30g/m²	156	
40g/m²	147	

180° Peeling Test

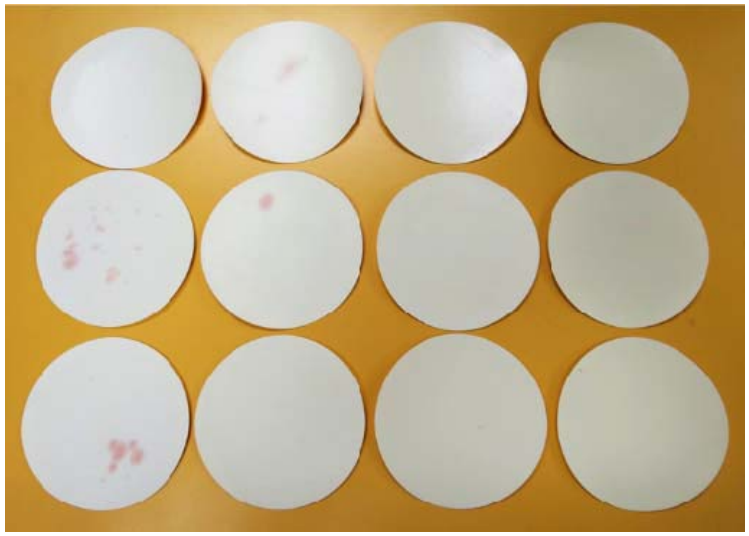


MFC deposit	Peeling force N/m
10g/m²	36 ±10
20g/m²	29 ±6
30g/m²	25 ±7
40g/m²	30 ±6

MFC thickness vs barrier performance

Red oil Cobb test

- 30 minutes exposure
- 3 replicates (shown here)



10 g/m² 20 g/m² 30 g/m² 40 g/m²

Coat weight of the MFC layer

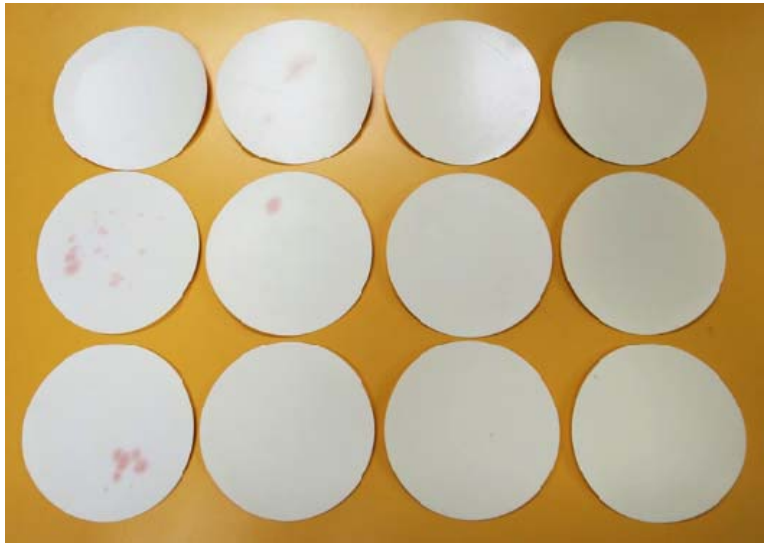
MFC deposit	Red dyed oil absorption	Kit Test	Water absorption
	$Cobb_{30min}$ in g/m²		$Cobb_{60s}$ in g/m²
10 g/m²	3,6 ±0,8	10+	38 ±1.2
20 g/m²	1,9 ±0,7	12	41 ±1.7
30 g/m²	1,2 ±0,1	12	47 ±1.2
40 g/m²	1,0 ±0,3	12	50 ±1.9

MFC thickness vs barrier performance

		Oxygen transmission rate (OTR) $cm^3/(m^2.d.bar)$			
Basis weight		10 g/m²	20 g/m²	30 g/m²	40 g/m²
Theoretical thickness[1]		8 µm	16 µm	23 µm	31 µm
Four Replicates	1	5,3	4,0	3,8	4,6
	2	4 700	1,5	1,6	2,5
	3	4 300	3,2	3,8	3,6
	4	293 800	290	400	2,7
Theoretical OTR[2]		1,04	0,52	0,35	0,26

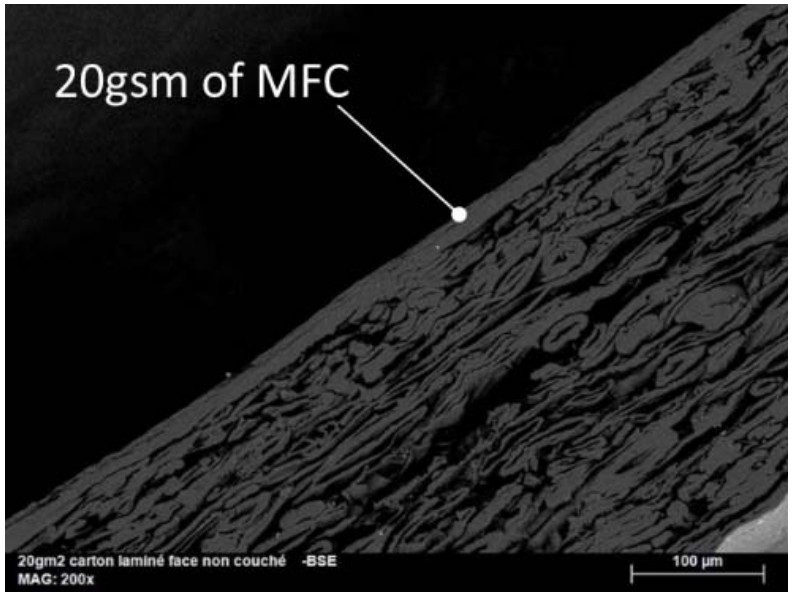
Defects

[1]Calculated from density and basis weight
[2]Calculated from permeability and theoretical thickness

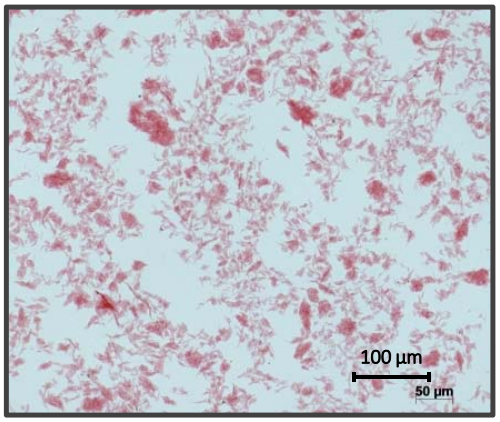


10 g/m² 20 g/m² 30 g/m² 40 g/m²
Coat weight of the MFC layer

Intermediate conclusions



- Very good adhesion on Boards
  - Coated side and uncoated side
  - Scott bond test is not adapted
    - Failure always in the middle-ply of the board
- Influence of MFC layer thickness on
  - Barrier performance
  - Defects linked to the residual coarse elements?
  - Finer grades vs cost ?



OUTLINE 4/4

Conclusions

Conclusion on MFC wet lamination

- A new process to cover papers and boards with MFC
  - Targeted MFC weights from 5 to 20 g/m²
    - To get the benefit of MFC for barrier
  - Combined filtration and drying
    - to decrease process costs
  - Adhesion without the use of glue
    - Possible both on mineral coated side or fibrous side of a board
    - Depends on paper surface chemistry, roughness, porosity and on MFC grade
    - True nature of the adhesion is not yet fully understood
      - Van der Waals, Hydrogen, Coulombian Forces? Mechanical Interlocking?

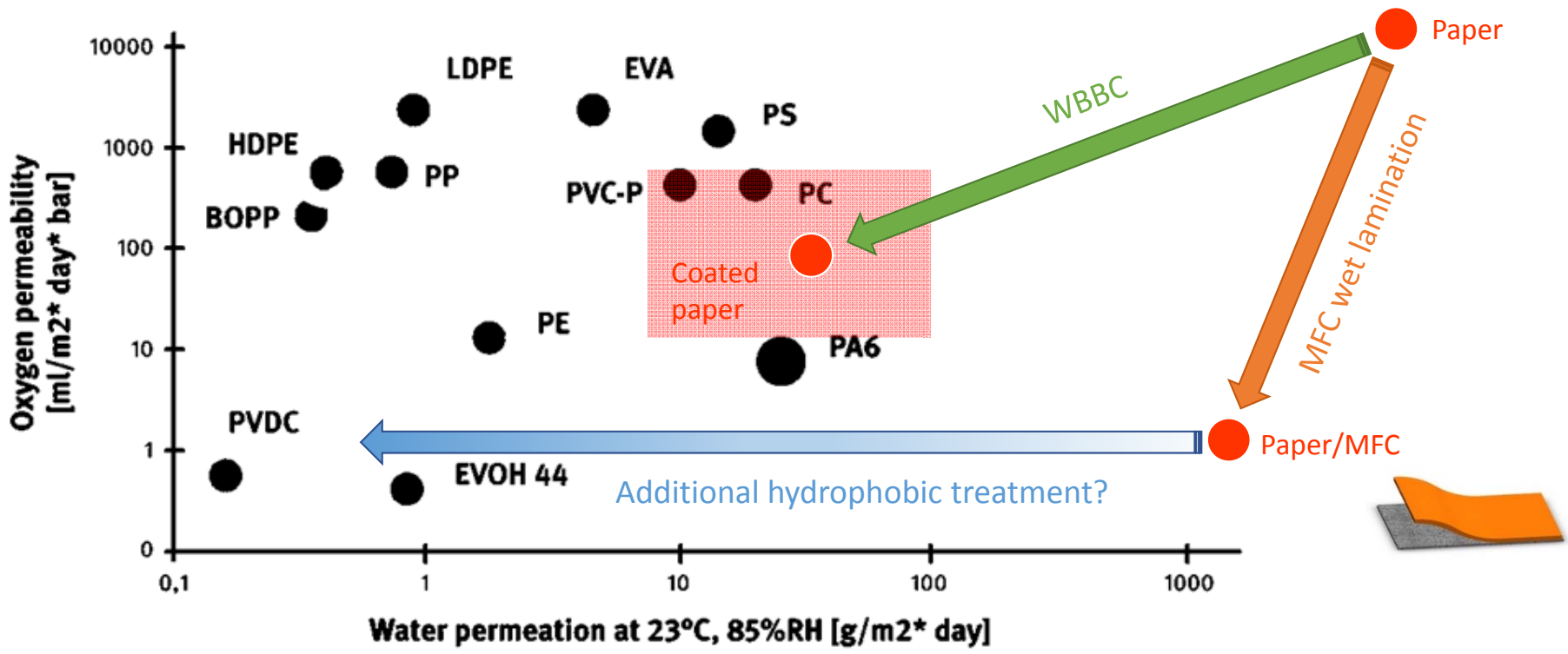
Summary of the barrier performances

- Data measured on a FBB (270g/m²) wet laminated with 20g/m² of MFC BBK

Grease barrier		Oxygen Barrier		Water barrier	Water vapour Barrier	
Oil absorption $Cobb_{30min}$	Kit test	23°C – 0% RH	23°C – 50% RH	Water absorption $Cobb_{60s}$ - ISO 535	23°C – 50% RH	38°C – 90% RH
~1 g/m²	12	<1 cm³/m².d.bar	3 cm³/m².d.bar	40 g/m²	130 g/m².d	2400 g/m².d

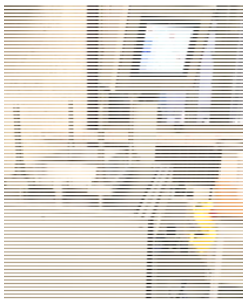
Not matching requirements of several markets

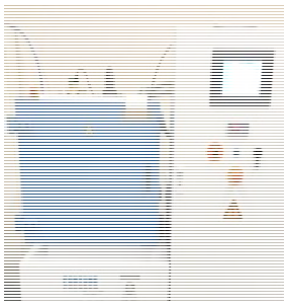
Towards a plastic-like all-cellulosic material for packaging markets



Perspectives on MFC wet lamination

- Main working axis
  - MFC grade (cost/thickness/size vs barrier)
  - Economics of the process
  - Get a better understanding of the process steps
    - Filtration, mechanics of the wet MFC films, adhesion, drying…
  - Development / Demonstration of the process
    - Dedicated laboratory equipment were commissioned in March 2018
    - Small scale pilot machine is expected for September 2019





MaLiCs
Une plateforme soutenue par
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Acknowledgements

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- https://tel.archives-ouvertes.fr/tel-01796806

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Thank you

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TAPPI