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# Molded pulp packaging for chilled dairy products - challenges and opportunities

Alexander Leo Bardenstein
*Danish Technological Institute, Plastics and Packaging Technology*


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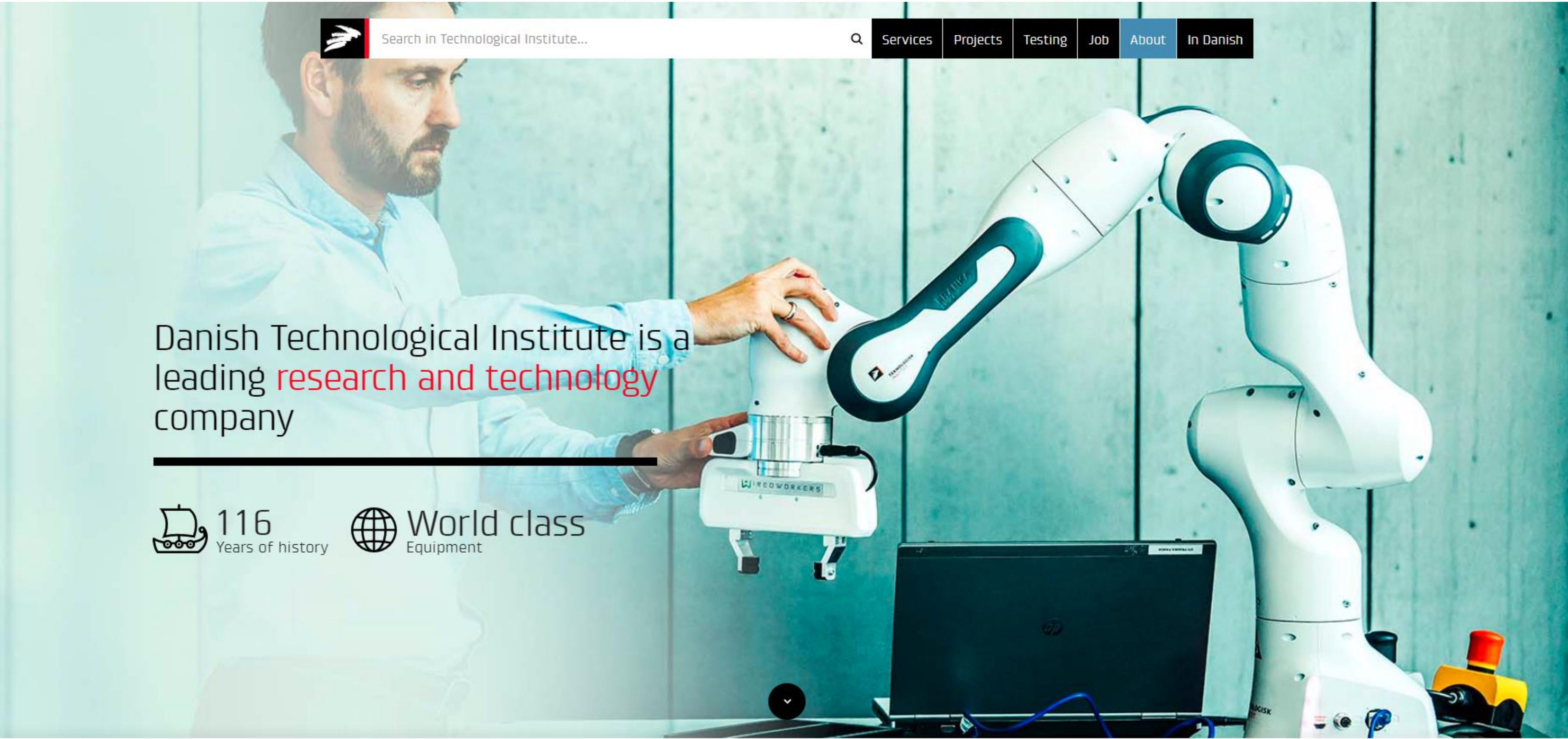

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Mrs. Beatriz Chambel
*Biorefinery*

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# Why molded pulp? What barriers are needed? What are challenges?

➢ Chilled dairy cups and bottles need up to 1-2 months of storage, very low WVTR, moderate or higher OTR, water, oil & grease repellency, wet strength

➢ The European Commission by its Regulation 1616/2022 authorises the applicable recycling technologies and from 10 July 2023 only recycled plastic obtained from authorised recycling technologies may remain on the market of packaging food contact materials, except for recycled plastics obtained from the development of novel (recycling) technologies

➢ At the time the Regulation comes into force, the Commission has authorised two recycling technologies:

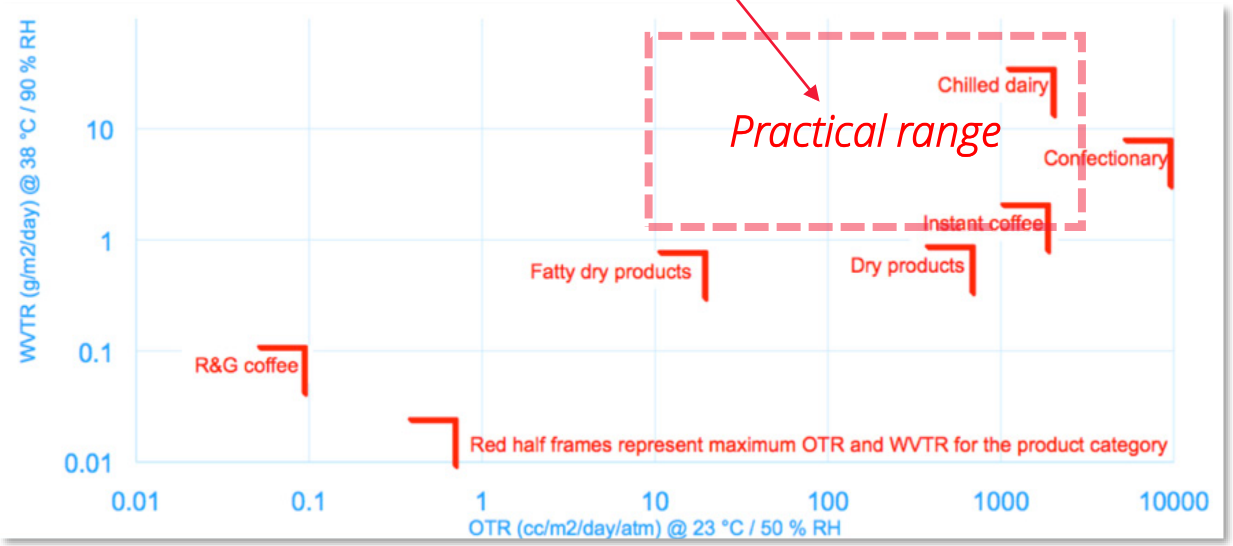
1. Post-consumer mechanical PET recycling
2. Recycling from product loops which are in a closed and controlled chain, e.g. PET, HDPE or PP bottles

➢ Fibers must constitute minimum 83-88% of molded pulp packaging weight to provide for 80-85% repulpability of cellulose

➢ The better barriers are needed, the more difficult it is to recover (repulp and recycle) cellulosic fibers

➢ The challenge is thus to find recyclable cellulose-based solutions with required barrier performance and as low content of polymer plastics as possible

Barrier requirement estimates for different food product categories (courtesy of Y. Wyser & A. Vishtal)



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# Short introduction to pulp molding technologies

- Thermoforming of air-laid dry pulp
- Thermoforming of wet-laid/hot-pressed dry pulp/paper/board sheets
- Wet pulp molding

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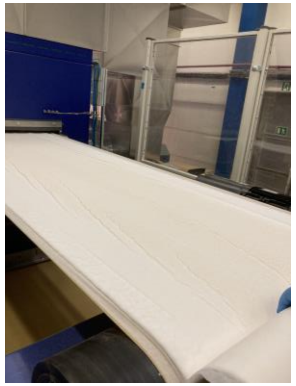
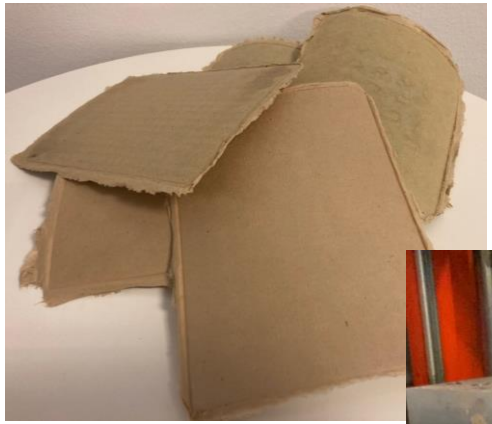
# Thermoforming of air-laid dry pulp



- Molding of a tray from air-laid pulp
- Modified air-laid Kraft fibers
- Air-laid process parameters:
  - top and bottom tissues,
  - 400-700 gsm,
  - compressed at 0.5-1.5 bar,
  - sprayed with water and dried at ~90°C
- Hot-pressing at 50-100 tons, 120-130 °C for 1-3 sec
- Smooth and glossy surface, resembles thermoformed plastic items
- Extremely hydrophilic (water-absorbing)

# Thermoforming of dry pulp/paper/board sheets

- Both trays and cups can be molded from sheets but special tools are required
- Both calendared and uncompressed pulp sheets can be thermoformed
- Sheets have to be wetted and pre-formed at low pressure
- Final compression at 20-150 tons 110-120 °C results in decent quality wrinkle-free trays and plates
- Surface and bulk properties influencing coating deposition, such as roughness, porosity, hydrophilicity etc. would mostly depend on the pulp composition



# Molding of wet pulp -

## Categorization of the International Molded Fiber Association (IMFA)

1. **Thick wall:** formed in an open fiber collection mold and then oven dried; the surface in contact with the mold is relatively smooth, and the other side is very rough; applications - support packaging, cushioning.

2. **Transfer molded:** both collection and transfer (take-off) molds are used; relatively smooth surfaces on both sides and better dimensional accuracy; molded product is oven dried; applications are egg trays, packaging for electronics, plant pots etc.

3. **Thermoformed (or thin-wall):** the most recent and advanced technology; formed product is captured in a heated mold where it is pressed, densified and dried; no oven drying is needed; high quality, thin-walled items with high dimensional accuracy, smooth rigid surfaces; resembles thermoformed plastic items; 3D-analog of paper calendaring

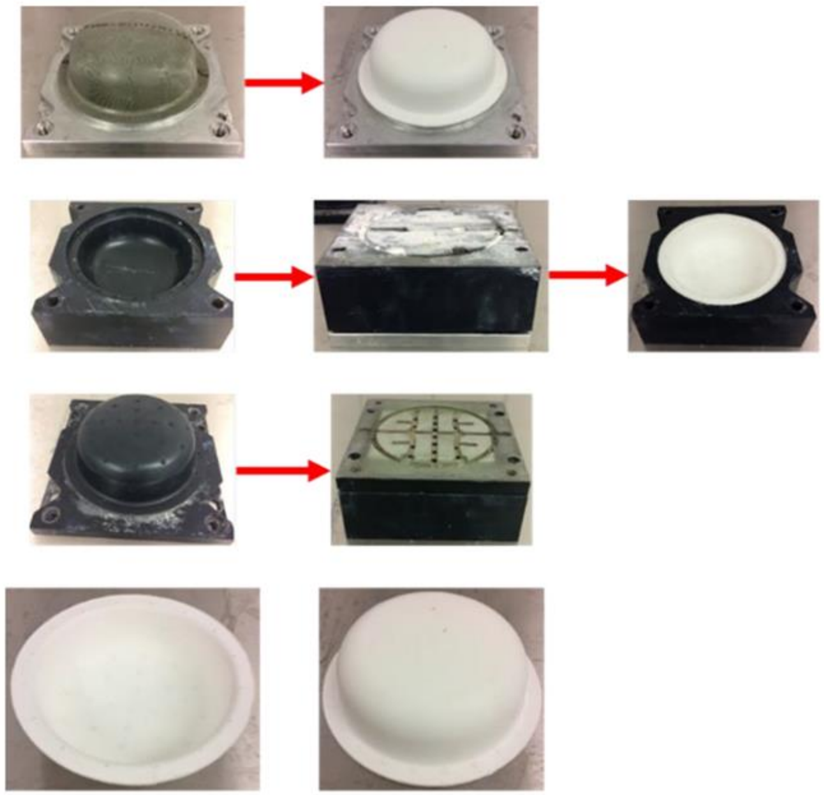
4. **Processed:** molded pulp products, mostly thermoformed/thin-wall, with further or special treatments like surface processing, coating deposition, printing etc.








# Thermoforming = forming + transfer + hot pressing between two molds



Pulp preparation - cellulosic fibers are dispersed in water (0.1-1.0 % of solids)

The forming mold is submerged in the pulp. By application of vacuum, a wet cellulosic object is formed on the metal mesh → drying→ready as "thick-wall object" – Category 1

The wet formed object is transferred from the forming mold to the concave drying mold → drying→ready as a "transfer-molded object" – Category 2

The wet object is dried between the concave and convex molds by application of heat and pressure → ready as a "thermoformed object"– Category 3

The object is removed from the molding machine and is ready for subsequent processing (e. g. surface processing, barrier coating deposition etc.) – Category 4

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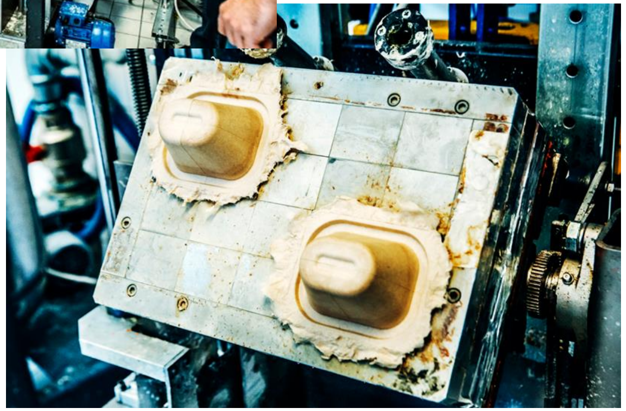
# Mini-Paper Factory

- MPF is a compact paper molding machine designed at Danish Technological Institute
- Primary application area is lab prototyping and pilot manufacturing of customer-specific fibre-based packaging containers – flat sheets, trays, cups, bowls, plates etc.
- Covers all wet-molded packaging categories



## Main features:

- Fast and convenient mold interchange
- Full controllability of process parameters
- Ideal for research or piloting
- Easy  to upscale
- Extremely low initial pulp volume of only 50 L

## Technical specifications

- Overall dimensions: 1600 (H)×1500 (W)×1200 (L) mm
- Footprint: 1.8 $m^2$
- Power consumption: 14 kW
- Maximum overall dimensions of a molded object:
  150 (H)×200 (W)×300 (L) mm
- Average capacity: 30 molded objects per hour

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# Wet molded pulp-based packaging with adequate diffusion barriers suitable for fiber recycling should be <u>thermoformed</u> and then <u>processed</u> by deposition of coatings

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# Examples of liquid barrier coatings, their functionalities, and methods of deposition onto 3D molded pulp packaging at DTI

| Coating liquid | Basic functional properties | Function | Deposition method |
|---|---|---|---|
| EXCEVAL AQ-4104 (Kuraray)<br>Hydrophobically modified PVOH, water solution, viscosity of 3.6-4.4 mPa·s | Hydrophilic, excellent oxygen barrier, moderately water-absorbing within a short time, requires moisture protection, heat sealable, oven drying above 120 °C | Primer, oxygen barrier | Spraying, dipping, washing, spinning of rotationally symmetric items |
| REEF-1 (CelluComp Ltd.)<br>Acrylic-based, water dispersion (37% of solids), contains Curran® MFCP, viscosity of 300-800 mPa·s @ 23 °C | Hydrophobic, excellent moisture barrier, vacuum flash drying or oven drying at ~ 110 °C | Moisture barrier | Spraying, dipping, washing, spinning of rotationally symmetric items |
| REEF-2 (CelluComp Ltd.)<br>Acrylic-based, water dispersion (37% of solids), contains Curran® MFCP; viscosity of 300-800 mPa·s @ 23 °C | Hydrophobic, excellent moisture barrier, good oxygen barrier, heat sealable, vacuum flash drying or oven drying at ~ 110 °C | Moisture and oxygen barrier | Spraying, dipping, washing, spinning of rotationally symmetric items |
| ICO 020 sol-gel (DTI)<br>SiO-sol-gel;~ 5% solids; viscosity of 1.5-2 mPa·s | Hydrophobic, hard coating, requires a primer, excellent moisture barrier, oven sintering above 120 °C | Moisture and oxygen barrier | Spraying, dipping, washing, spinning of rotationally symmetric items |
| ICO 060 sol-gel (DTI)<br>SiO-sol-gel; ~ 5% solids; viscosity of 1.5-2 mPa·s | Hydrophilic, waterproof, oven sintering above 120 °C | Primer for water-based barrier coatings | Spray/ mist deposition |

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# Interaction of water-based barrier coatings with molded pulp substrates

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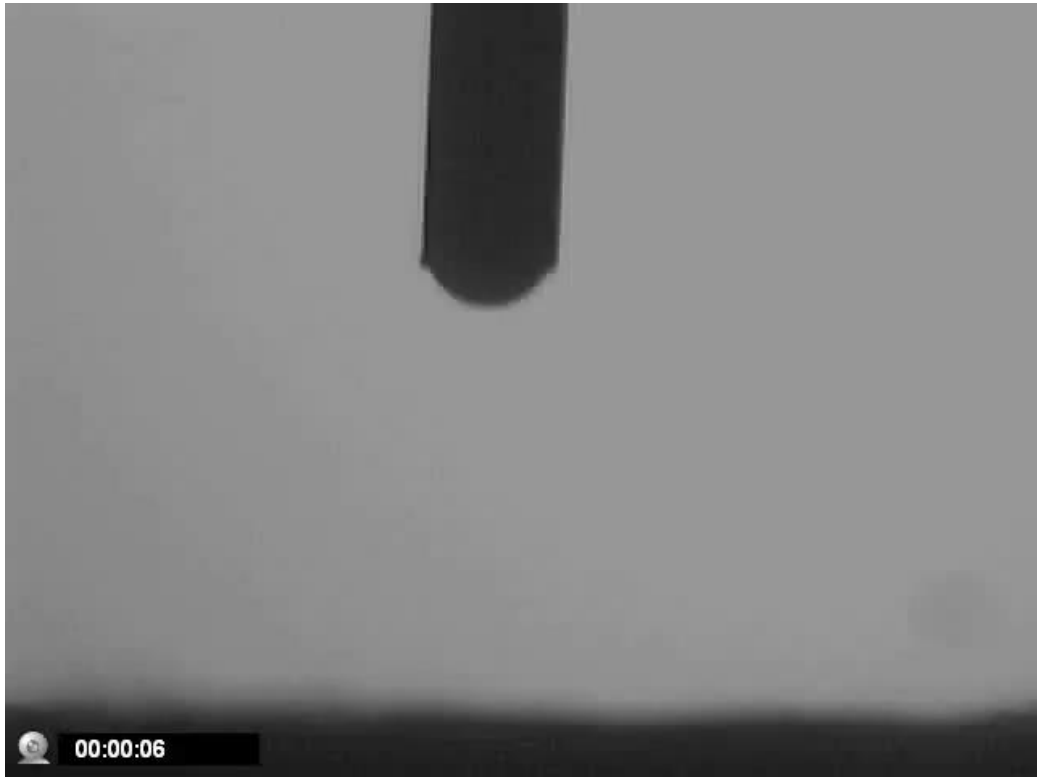
# Dramatic impact of hydrophilicity, water absorption and fiber uplift

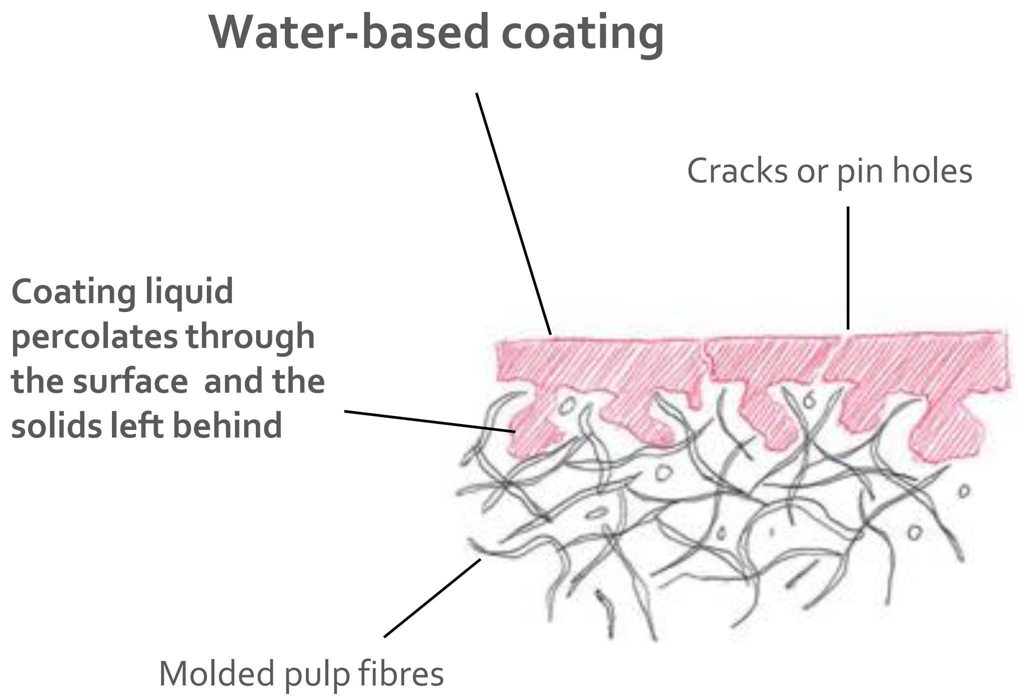
Category 3 molded tray, NBSK pulp

Deposition of a drop of REEF-2 coating (video)



# Peculiarities of wet molded pulp that affect performance of water-based barrier coatings

- Surface roughness
- Detachment (uplift) and dusting of fibers - fibers protrude from the coating layer of a regular thickness
- Pulp porosity
- Hydrophilicity of and water absorption by Kraft fibers
- 3D (concave/convex) geometries – inhomogeneity of coating thickness




➢ In the issue, conventional Category 3 molded pulp would typically need coating of higher grammage than calendared paper with special additives to attain similar barrier performance

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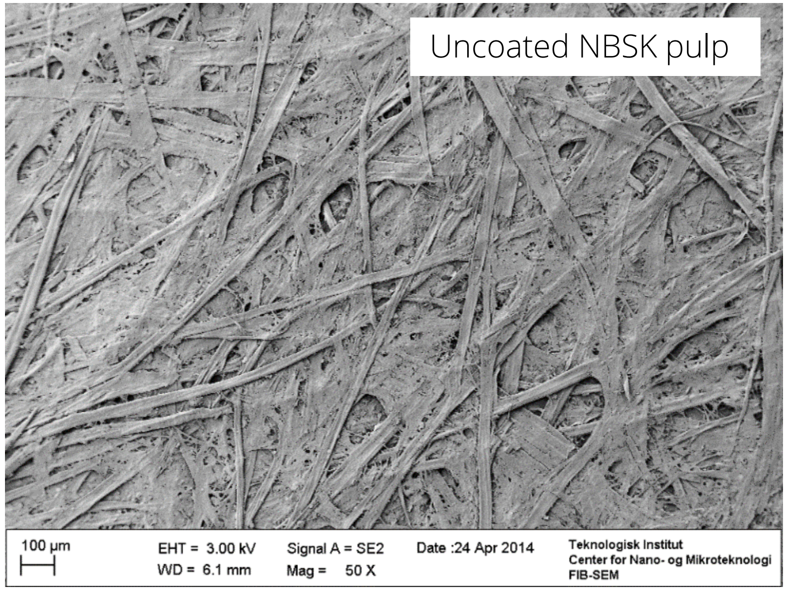
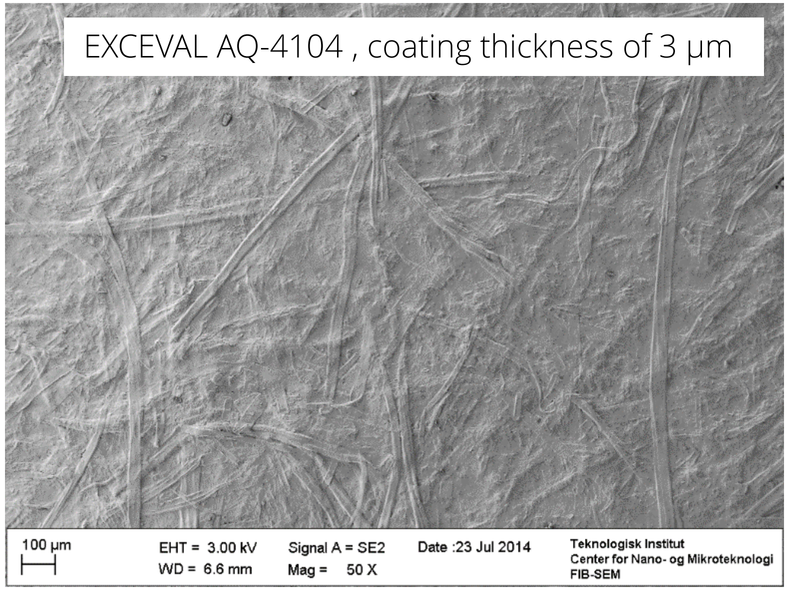
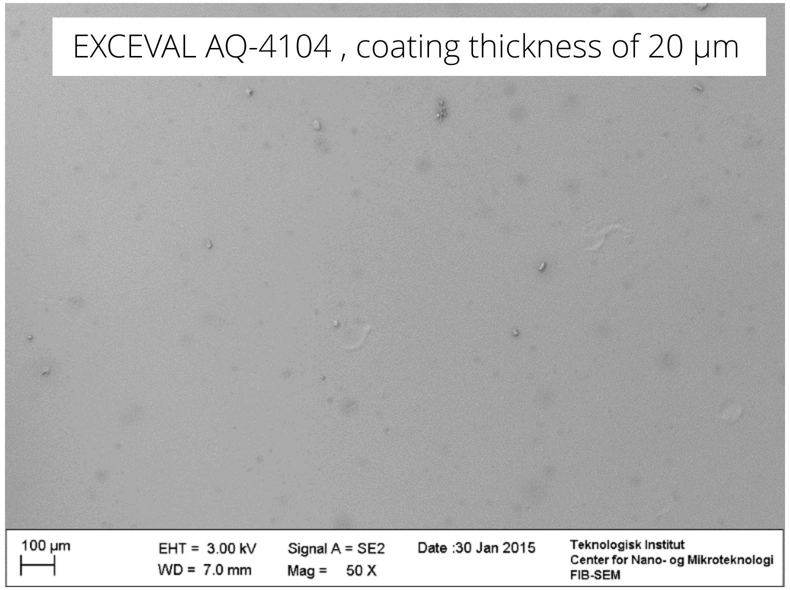
# How thick coating is needed to conceal a Category 3 molded Kraft pulp fiber network?



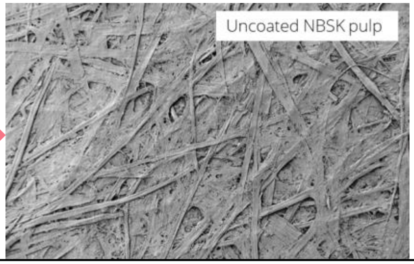



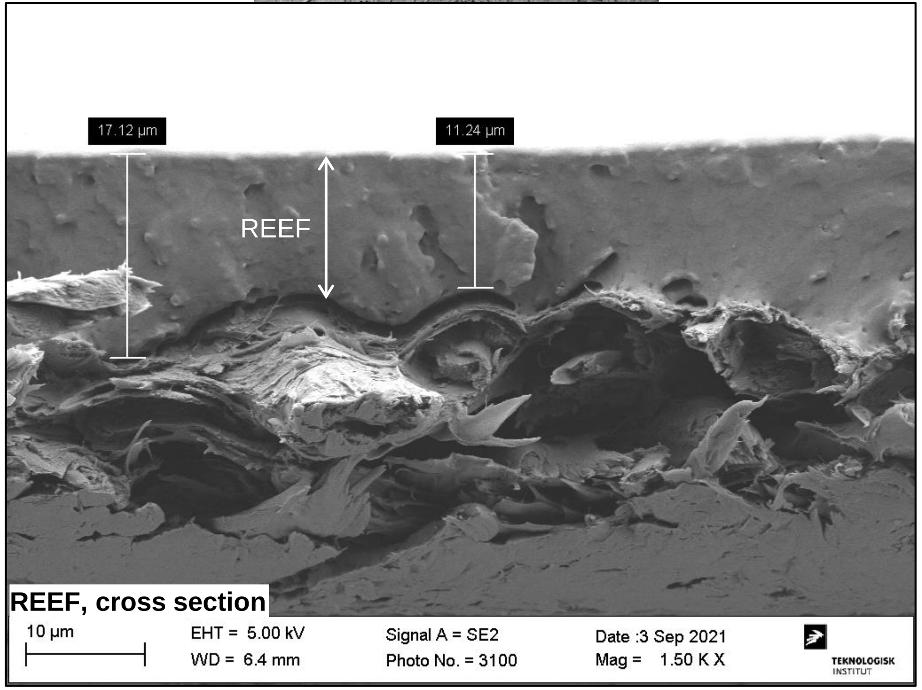




- Coating thickness of min. ~ 20 µm is needed to obtain a microscopically smooth surface

# Moisture barrier improvement due to reduced roughness, porosity, and water absorption

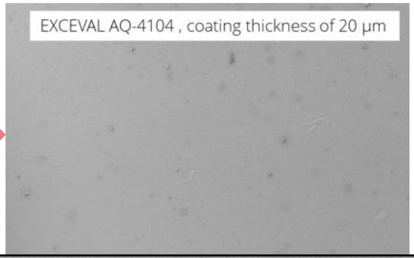
REEF-2, spraying, ~ 15 gsm

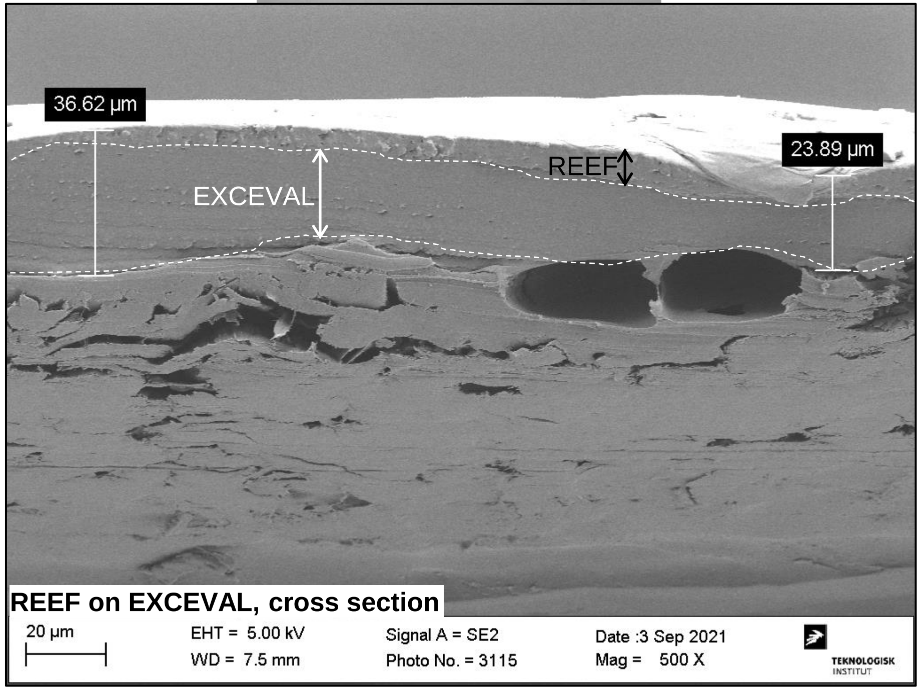



- WVTR (23 ºC, 50% RH) = 2.36 g/m²/day

REEF-2, spraying, ~ 7 gsm




- WVTR (23 ºC, 50% RH) = 0.29 g/m2/day (BIF = 8.1)

# Controlling hydrophilicity with Alkyl ketene dimers (AKDs)?

A drop of REEF-2 coating on the surface of molded NBSK pulp with AKD (1 g dry/kg)


- Adhesion is too low, and it would be difficult to deposit a continuous film of a water-based coating
- Fiber uplift, dusting and porosity issues remain
- No barrier improvement can be expected
- Molded pulp surface ideal for deposition of water-based barrier coatings would be *hydrophilic but watertight*

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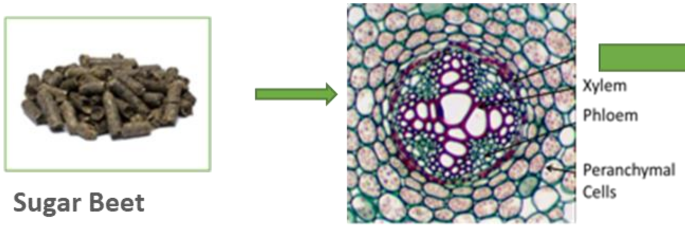
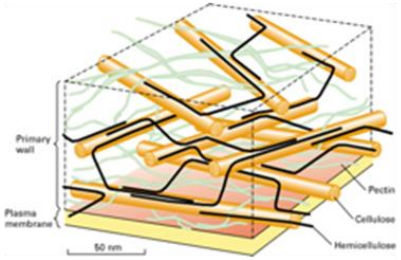
# Molded pulps with alternative fiber materials – introducing Curran®, Micro-Fibrillated Cellulose Platelets derived from root plants

Courtesy of CelluComp Ltd.



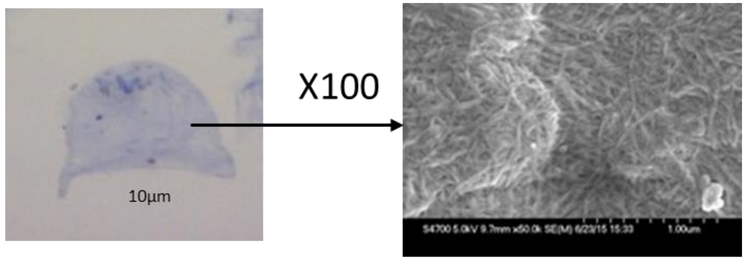

Sugar Beet
Pellet or Pulp

Source material rich in Parenchymal Cells

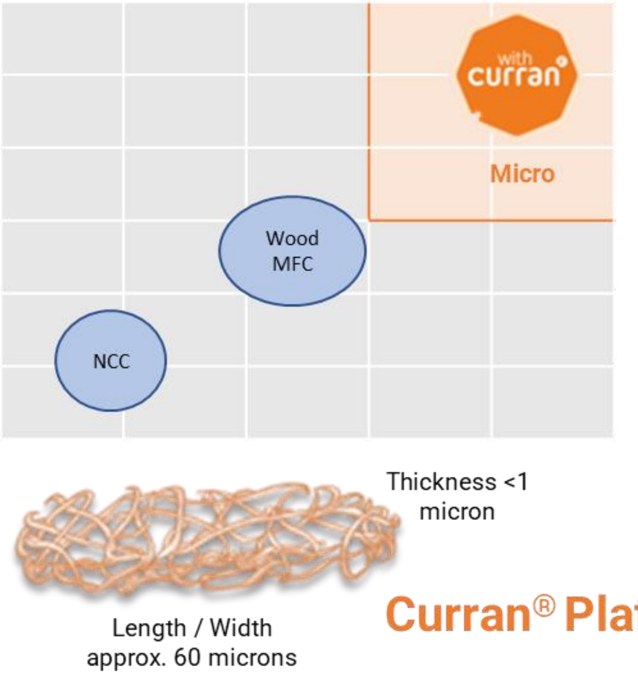


Cell wall structure

Extraction of pectin and hemicellulose




Curran® Platelet
Optical Microscopy




Curran® Platelet

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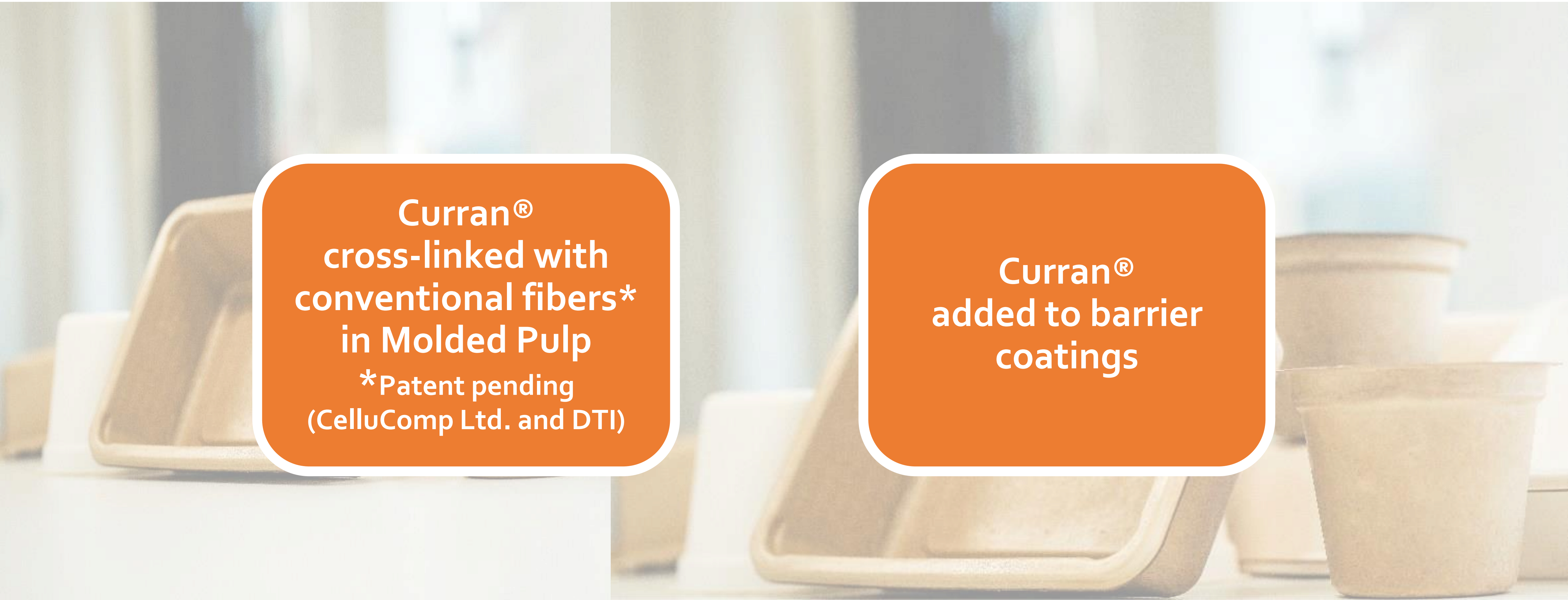
# Molded pulp and coatings with Curran®



**Curran®
cross-linked with
conventional fibers*
in Molded Pulp**

*Patent pending
(CelluComp Ltd. and DTI)

**Curran®
added to barrier
coatings**

# Impact of Curran® on the molded pulp surface quality

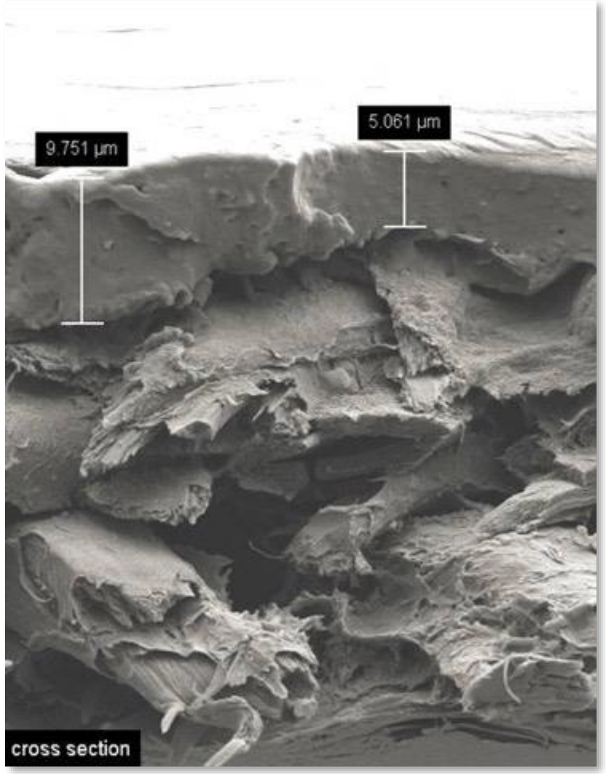
Category 3 molded tray, NBSK pulp

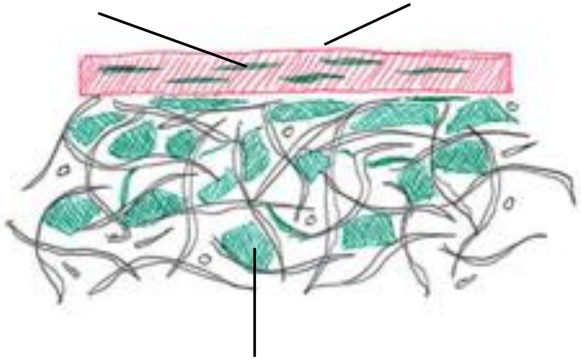
90% NBSK pulp + 10% Curran®, cross-linked

# Impact of cross-linked Curran® on the coating deposition process, resulting thickness and quality

Deposition of a drop of REEF-2 coating comprising Curran® onto molded pulp of 90% NBSK cross-linked with only 2% of Curran® (video)


~ 15 gsm w/o Curran® → ~7 gsm with Curran®



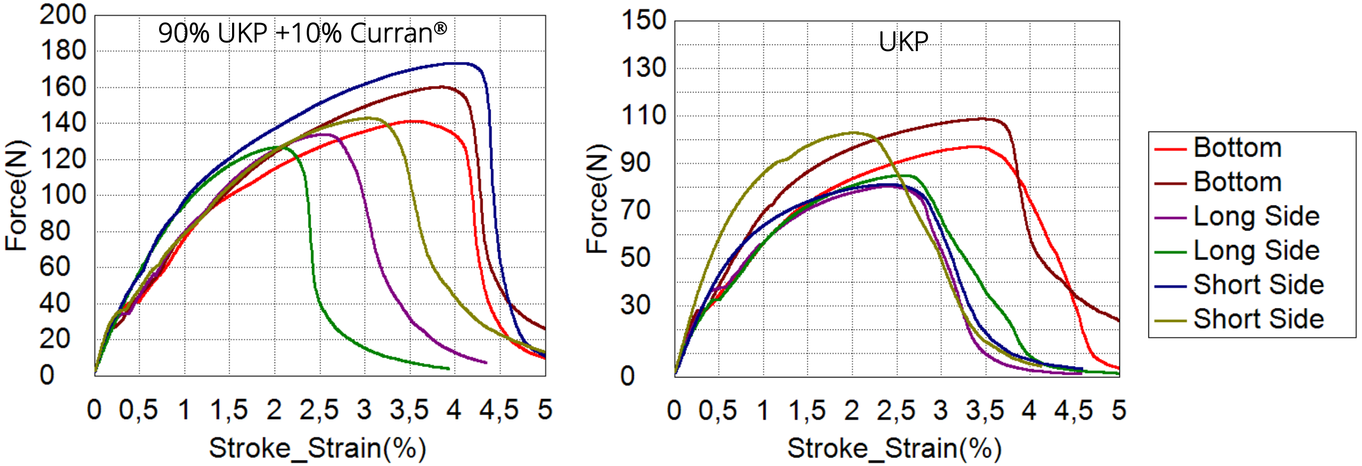

- Curran bridges gaps between pulp fibres and prevents coating liquid penetrating into the matrix.
- It also increases the wet strength of the pulp by crosslinking between fibres
- Eliminates re-roughening after wetting

# Impact of cross-linked Curran® on the molded pulp wet strength

Test samples were cut out from 6 different areas on the sides and bottoms of molded trays

| Pulp | Maximum tensile stress, MPa | |
|---|---|---|
| | Dry test | Wet test |
| Nordic unbleached softwood Kraft (UKP) | 7.8 ± 1.8 | 0.088 ± 0.012 |
| NBSK | 2.9 ± 0.1 | unmeasurable |
| 90% UKP + 10% Curran® | 11.0 ± 1.6 | 2.9 ± 0.4 |

Maximum tensile force vs strain



# Impact of cross-linked Curran® on the water absorption performance of coated molded pulp

- Spray coated Category 3 wet molded 1-mm-thick flat sheets

| *Pulp and coating* | <Cobb1800>, g/m$^2$ | | | |
|---|---|---|---|---|
| | single-sided | | double-sided | |
| | Avg. | SD | Avg. | SD |
| UKP; 15-20 µm of REEF-2 | 4.6 | 0.5 | 3.8 | 0.9 |
| 90% UKP +10% Curran®; 7-10 µm of REEF-2 | 2.8 | 0.7 | 1.2 | 0.5 |

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# Curran® in both coating and pulp makes a great impact on the barrier performance


- REEF coating (liquid) sprayed on both sides of a paper bottle wall molded of Unbleached Softwood Kraft (UKP) and 98% UKP + 2% Curran® pulps
- Bottle volume of 106 $cm^3$, surface area of 120.082 $cm^2$. Could be good packaging for cream.

| Pulp composition | WVTR (23°C, 50% RH), $g/m^2/day$ |
|---|---|
| UKP | 7.1 ± 0.1 |
| 98% UKP + 2% Curran® | 1.42 ± 0.02 |

BIF = 5

# Appearance of molded dairy cups spray coated by REEF-2


# Industry approved molded dairy cup

- 95% UKP cross-linked with 5% of Curran®
- REEF-2 coating sprayed on both sides, total grammage of ca. 20 gsm
- Heat sealable
- OTR (23 ºC, 50% RH) = 49.0 ± 5.4 cc/m$^2$/day
- WVTR (23 ºC, 50% RH) = 1. 6 ± 0.8 g/m$^2$/day
- Recyclable (PTS-RH 021:2012 – Category II: Paper and board for Recycling)
- Biodegradable (EN 13432 (2000), OECD 301B - $CO_2$ Evolution method )
- Passed food contact approval tests (EN1186-01:2002)

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# Food Contact Approval

## FCM•testing ApS

Test af materialer med fødevarekontakt

ilac-MRA DANAK



**Test report.** No.: 2021-840 Date:2022.04.04

Report template no. 001

| | |
|---|---|
| Customer: | Teknologisk Institut |
| Address: | Center for Plast og Emballage<br>Gregersensvej 1<br>2630 Taastrup<br>Att: Alexander Bardenstein |
| Product Name: | Paper Cup |
| Product Description: | Cup of paper |
| Sampled by: | Customer |
| Sample Receiving date: | 2021.12.01 |
| Testing Period: | 2022.01.01 - 2022.01.31 |

**Test Paper cup:**

**Test summary:**

| Type of analysis | Conclusion | Regulation or protocol |
|---|---|---|
| Total migration 10 % ethanol | Pass | (EC) no 10/2011 |
| Total migration acetic acid | Pass | (EC) no 10/2011 |
| Content of Pentachlorophenol (PCP) | Pass | LFGB |
| Content Formaldehyde | Pass | LFGB |

Test results: See next page.

FCM•testing

Per Holst-Hansen
MSc/CEO



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E-mail: Info@fcmtesting.com; Website: www.fcmtesting.com

FCM•testing ApS
Test af materialer med fødevarekontakt



Test report. No.: 2021-840 Date:2022.04.04
Report template no. 001

**1: Total migration. Test methods and test conditions:**

DS/EN1186-01:2002, Guide to selection of conditions and test methods for overall migration
DS/EN1186-09:2002, Test methods for overall migration into aqueous food simulants by filling.

| Food Simulant | Test time | Test Temperature | Max. Permissible Limit* | Method uncertainty | Method LOQ |
|---|---|---|---|---|---|
| 10% v/v ethanol | 30 days ± 5 hours | 5°C ± 1°C | 10 mg/dm² | 10% | 0,5 mg/dm² |
| 3% w/v acetic acid | 30 days ± 5 hours | 5°C ± 1°C | 10 mg/dm² | 10% | 0,5 mg/dm² |

* According to regulation (EC) 10/2011

**Total migration Test results, repeat:**

| Test | Rep. A | Rep. B | Rep. C | Average | Pass/Fail* |
|---|---|---|---|---|---|
| 10% v/v ethanol (mg/dm²) | <0,6 | <0,6 | 1,5 | 0,9 | Pass |
| 3% w/v acetic acid (mg/dm²) | <0,6 | <0,6 | <0,6 | <0,6 | Pass |

* According to regulation (EC) 10/2011 incl. Amendments
Ratio Surface to Volume: 9,3 $dm^2$/l

**2: Total migration. Test methods and test conditions:**

DS/EN1186-01:2002, Guide to selection of conditions and test methods for overall migration
DS/EN1186-09:2002, Test methods for overall migration into aqueous food simulants by filling.

| Food Simulant | Test time | Test Temperature | Max. Permissible Limit* | Method uncertainty | Method LOQ |
|---|---|---|---|---|---|
| 10% v/v ethanol | 10 days ± 5 hours | Start 40 °C ± 1°C<br>ramp down 24 hours<br>9 days 5°C ± 1°C | 10 mg/dm² | 10% | 0,5 mg/dm² |
| 3% w/v acetic acid | 30 days ± 5 hours | Start 40 °C ± 1°C<br>ramp down 24 hours<br>9 days 5°C ± 1°C | 10 mg/dm² | 10% | 0,5 mg/dm² |

* According to regulation (EC) 10/2011

**Total migration Test results, repeat:**

| Test | Rep. A | Rep. B | Rep. C | Average | Pass/Fail* |
|---|---|---|---|---|---|
| 10% v/v ethanol (mg/dm²) | <0,6 | <0,6 | <0,6 | <0,6 | Pass |
| 3% w/v acetic acid (mg/dm²) | <0,6 | <0,6 | <0,6 | <0,6 | Pass |

* According to regulation (EC) 10/2011 incl. Amendments
Ratio Surface to Volume: 9,3 $dm^2$/l

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Test af materialer med fødevarekontakt



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**3: Content of Pentachlorophenol (PCP)**

Test method: Sample preparation according to EN ISO 15320:2011
Analysis vas performed by GC/ECD)

| Test | Unit | Result | Maximum Permissible limit* | Conclusion** |
|---|---|---|---|---|
| Content PCP (Cas.nr. 87-86-5) | mg/kg | <0,05 | 0,15 | Pass |

**4: Extractable of formaldehyde**

Test method: Sample preparation according to EN 645:1993, analysis with reference to EN 1541:2001

| Test | Unit | Result | Maximum Permissible limit* | Conclusion** |
|---|---|---|---|---|
| Extractable Formaldehyde (Cas.nr. 50.00-0) | mg/kg | <3 | 15 | Pass |

Picture of sample.

*** End of Report***

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# Molded pulps with alternative fiber materials – introducing SinProPack grass pulp for molded packaging

- SinProPack is a development project funded by the Danish Green Development & Demonstration Program (GUDP)
- Fibers is a waste of grass processing for isolating proteins
- DTI developed a deep processing/refining technology of grass pulp for wet molding resulting in a moisture proof fiber material with unusually low surface energy and high abrasive strength
- Blending SinProPack (SPP) grass fibers, Curran® and conventional cellulose fibers enables wet thermoformed pulps with outstanding performance: surface quality, abrasive strength, wet strength, low porosity etc.

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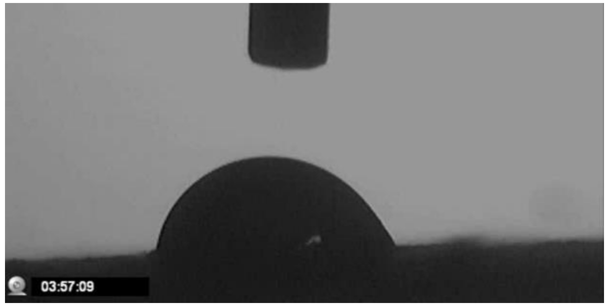
# Surface properties of wet molded pulps with SPP grass

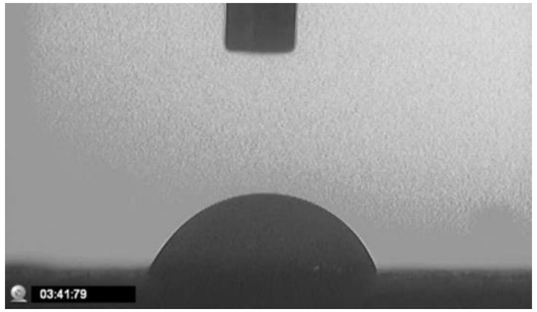
| Pulp composition | Contact angle | | Surface energy (mJ/m²) | |
|---|---|---|---|---|
| | Water | Glycerol | Dispersive | Polar |
| SPP grass | 86.0 | 86.2 | 3.5 | 21.4 |
| 70% SPP grass, 30% UKP | 69.9 | 75.7 | 1.1 | 39.0 |
| 50% SPP grass, 50%UKP | 55.9 | 65.9 | 0.4 | 57.1 |

- Drop of REEF-2 coating on molded pulps with SPP grass, after ca. 3-4 min after deposition

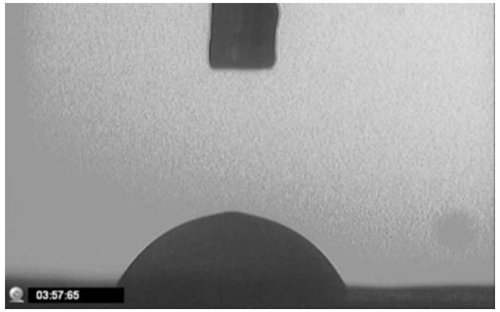
SPP grass




70% SPP grass, 30% UKP




50% SPP grass, 50% UKP




- Molded pulp with SPP grass fibers would be an excellent substrate for water-based barrier coatings

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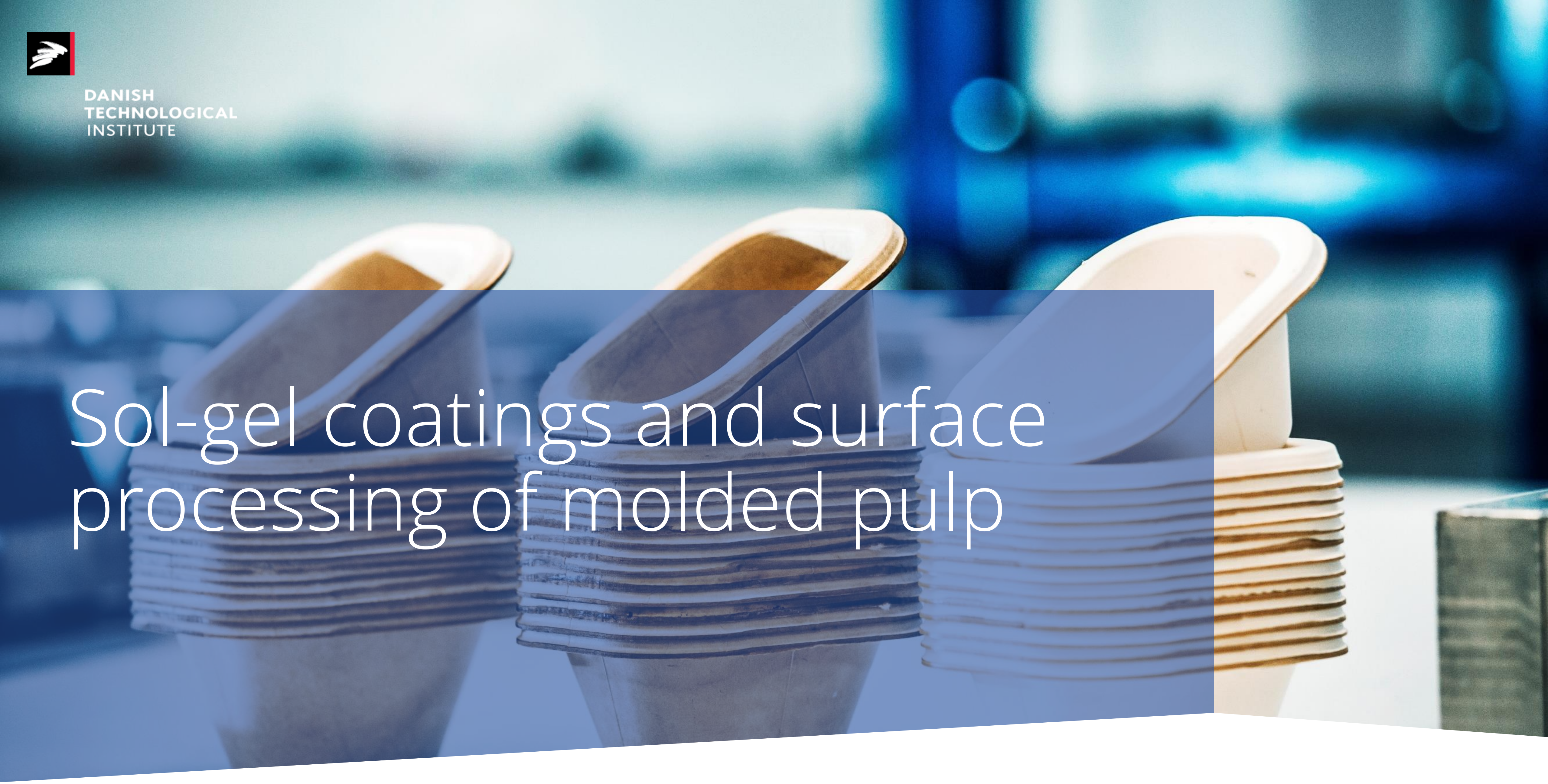

# Sol-gel coatings and surface processing of molded pulp

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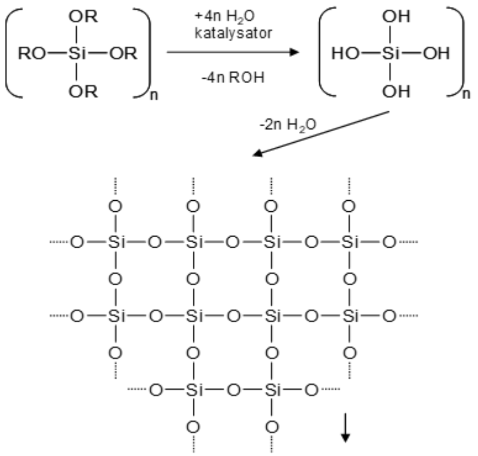
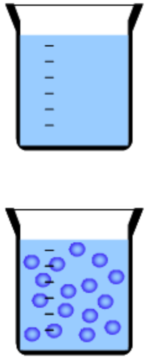
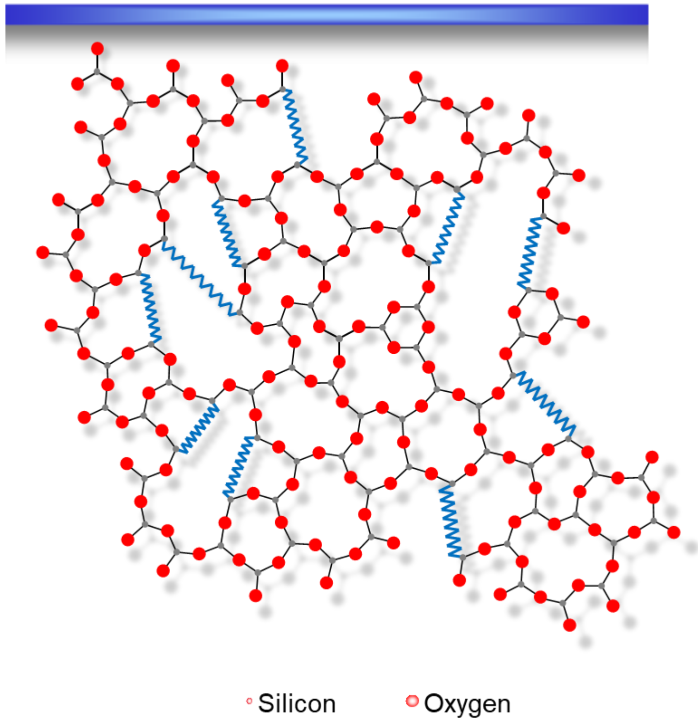
# Sol-gel. Or rather *sol→gel* ?

- The sol–gel process is a method for producing solid materials (e.g. coatings) from molecules in a liquid
- The method is widely used for the fabrication of silicon oxides (SiO)
- The process involves conversion of <u>silicon alkoxides monomers</u> into a colloidal solution (= **sol**) that acts as the precursor for an <u>integrated network</u> (= **gel**) of network organic-silicon polymers ($SiO_xC_yH_z$).
- Typical precursors are Hexamethyldisiloxane (HMDSO), 1,1,3,3-Tetramethyldisiloxane (TMDSO), 2,4,6,8-Tetramethylcyclotetrasiloxane (TMCTS), Hexamethylcyclotrisiloxane (HMCTS) etc.
- Depending on a precursor, SiO may have different organic side-chains. This allows to synthesize organic/inorganic amorphous structures where the organic components act as "springs" controlling the coating hardness, hydrophilicity, and the barrier performance
- After a sol→gel reaction reaches the equilibrium, and the "gel" is formed in a solvant, the liquid coating compound can be applied to a 3D surface by spraying, deeping, spinning etc.
- Curing of a sol-gel coating typically requires heating but other means of removing the remaining solvent , e.g. vacuum drying, may sometimes work





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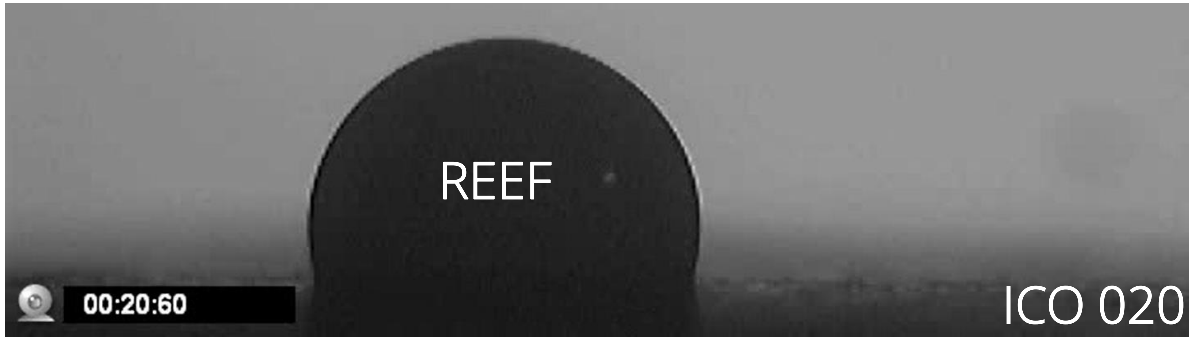
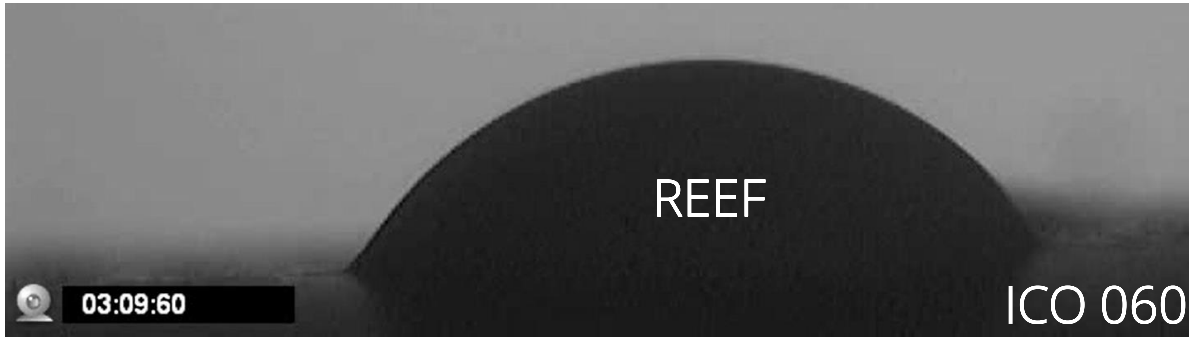
# Impact of molded pulp surface pre-processing (primering) in sol-gel spray mist on the coating deposition process

| | | | |
|---|---|---|---|
| ICO 020 sol-gel – contains a realatively small amount of silicon oxide, forms a hydrophobic water- and oil-repellent layer on a continuous "solid" surface, or percolates through the molded pulp surface and "jackets" the fibers, resulting in a hydrophobic surface | Hydrophobic, hard coating, requires an impermeable primer for barrier functionality, excellent moisture barrier, oven sintering above 120 °C | Excellent moisture and oxygen barrier | Spraying, dipping, washing, spinning of rotationally symmetric items with a primer, e. g. EXCEVAL or another sol-gel |
| ICO 060 sol-gel – contains a co-block hydrophilic polymer additive, forms a hydrophilic layer on a continuous "solid" surface, or percolates through the molded pulp surface and "jackets" the fibers, thereby reducing their hydrophilicity | Hydrophilic, waterproof, also requires an impermeable primer for barrier functionality, oven sintering above 120 °C | Excellent primer itself for water-based barrier coatings | Spray/mist deposition on the molded fiber substrate |

- Drop of REEF-2 coating on molded NBSK pulp treated with ICO 020 (left) and ICO 060 (right) sol-gel compounds by spray mist deposition (<1 gsm)



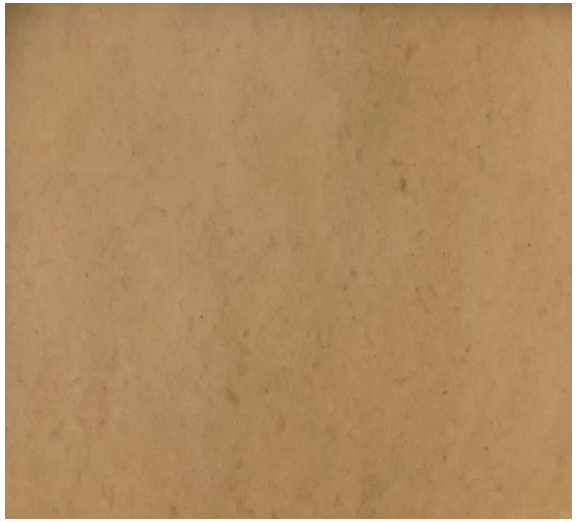



# Facilitation of a water-based barrier coating deposition on a super hydrophilic dry-molded air-laid pulp by ICO 060 sol-gel primer

REEF-2 (~20 gsm, dry)

REEF's water instantaneously percolates through the surface and the solids left behind thus no continuous barrier coating layer is formed, however the surface is hydrophobic and in the short run watertight

ICO 060 (<1 gsm)/REEF-2 (<10 gsm)



REEF forms a continuous glossy film on top of the ICO 060 treated surface.
WVTR (23 ºC, 50% RH) = 1. 7 g/m²/day

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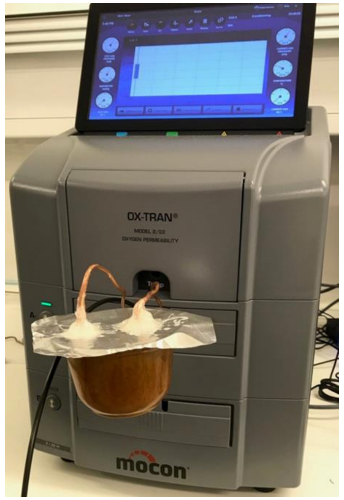
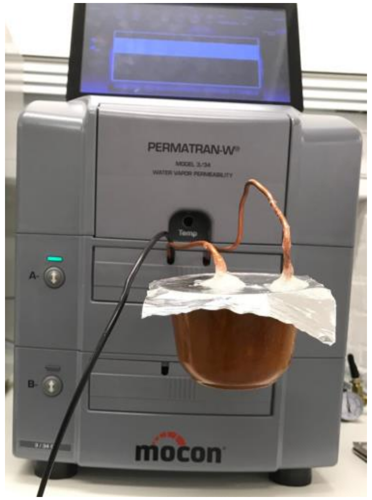
# Molded dairy cup with cross-linked Curran® in the pulp and ICO 020 sol-gel barrier coating

- 95% UKP cross-linked with 5% of Curran®
- EXCEVAL AQ-4104 **primer** sprayed on both sides, total grammage of ca. 30 gsm
- Sol-gel ICO 020 coating sprayed on both sides, total grammage of ca. 5-6 gsm
- OTR (23 ºC, 50% RH) = 2.4 ± 1.2 cc/m²/day
- WVTR (23 ºC, 50% RH) = 1.8 ± 0.6 g/m2/day
- Passed food contact approval tests (EN1186-01:2002)
- Microwaveable
- Repulpability of ~ 40%, not recyclable as paper packaging

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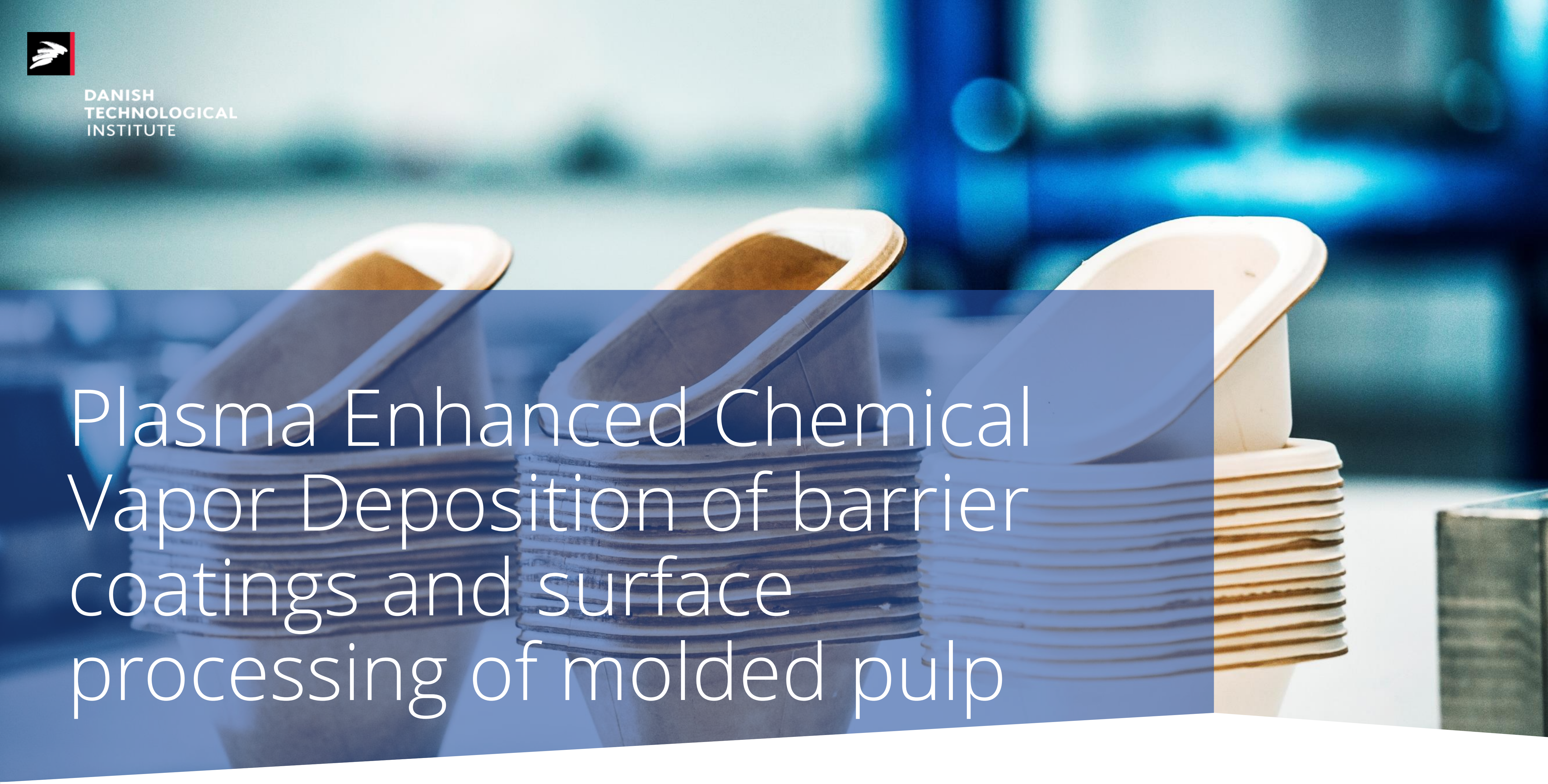
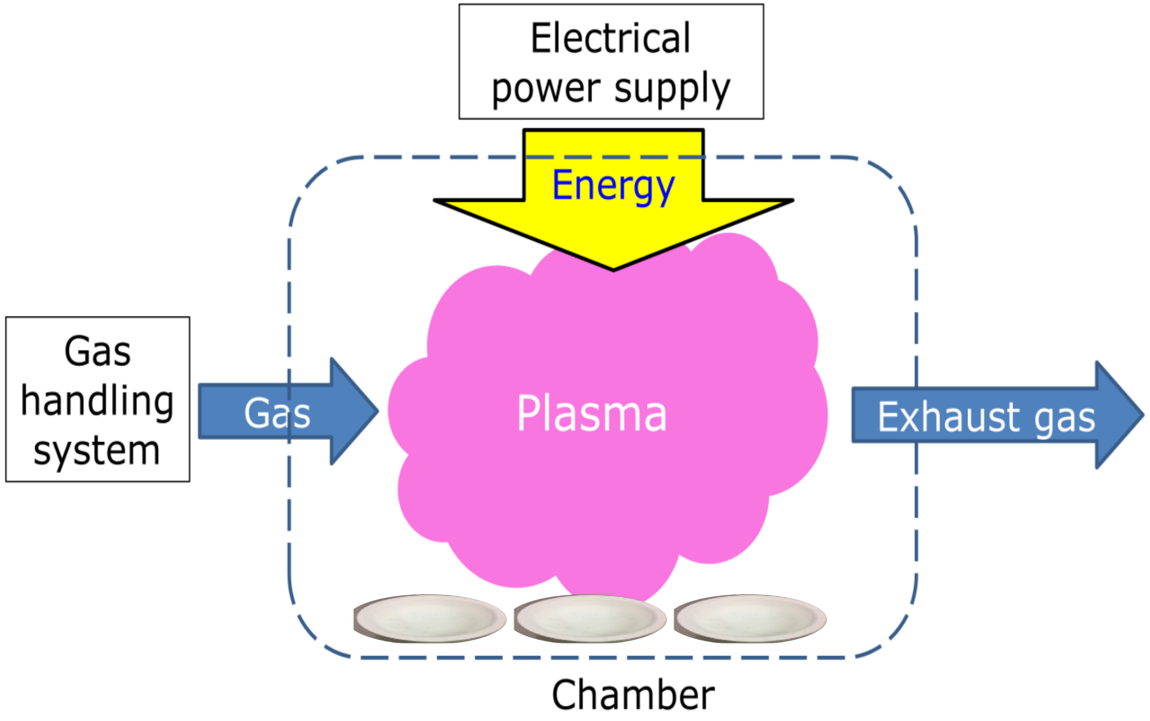


# Plasma Enhanced Chemical Vapor Deposition of barrier coatings and surface processing of molded pulp

# What's plasma?

- Plasma is ionized chemically reactive gas
- It is used both for surface modification and barrier coating deposition (solid film synthesis)
- PECVD process usually operated in **vacuum**, but can be also performed at **atmospheric pressure**



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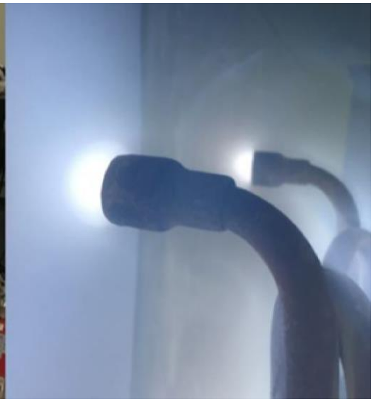
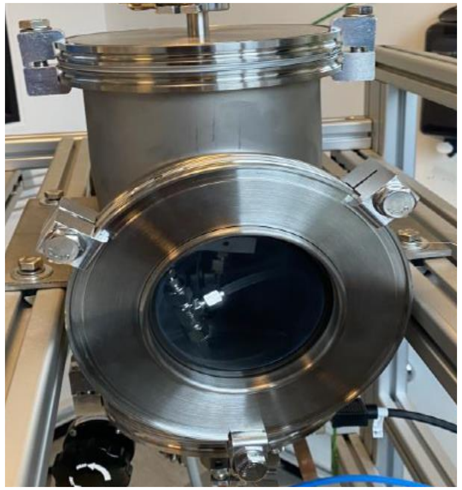
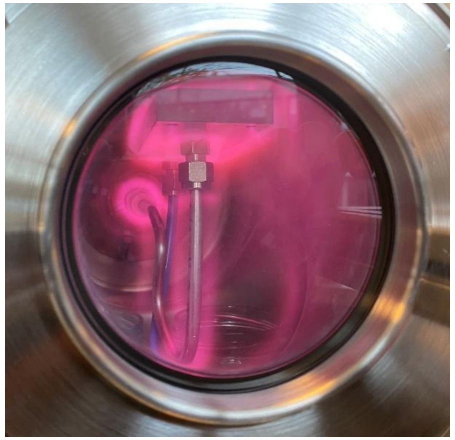
# Low pressure plasmas for Radio-Frequency Vacuum PECVD at DTI

- a-C:H, polymer-like or DLC coatings, precursor gas is $C_2H_2$

- $SiO_x/SiO_xC_yH_z$ coatings, precursor monomers are silicon alkoxides monomers - volatile liquids and even solids





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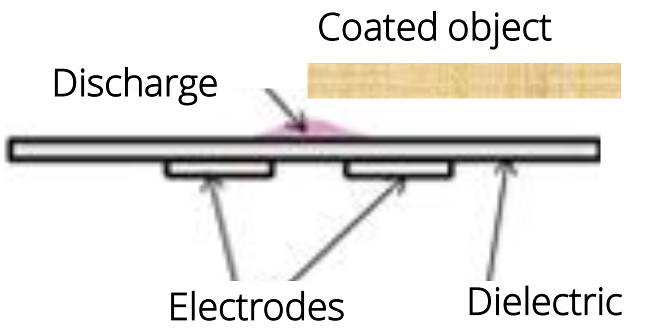
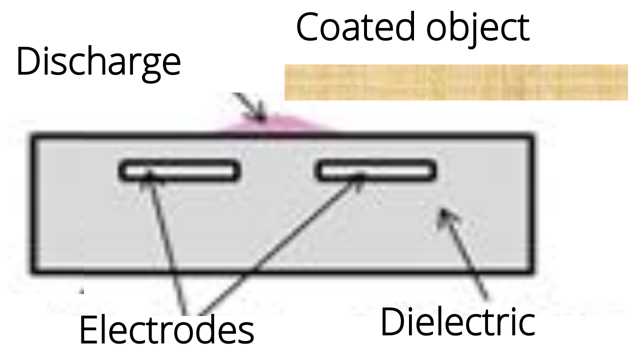
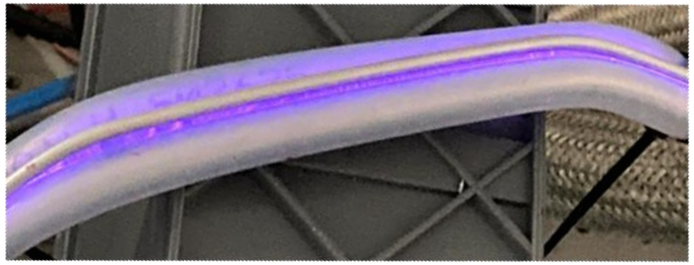
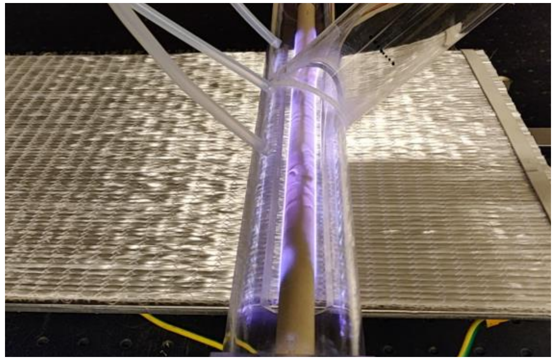
# Atmospheric pressure PECVD at DTI

- **<u>Dielectric barrier discharge (DBD)</u>** is used for coating synthesis on 2D and 3D substrates at atmospheric pressure
- $SiO_x/SiO_xC_yH_z$ coatings, precursor monomers are silicon alkoxides (alkoxysilanes) - volatile liquids and even solids
- DTI has a know-how to design surface discharge plasma so that any open 3D objects and even inside bottles can be easily coated at atmospheric pressure



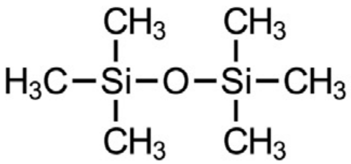



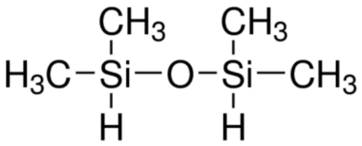



# PECVD and sol-gel SiO precursors used at DTI

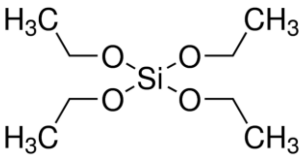
Hexamethyldisiloxane (HMDSO)



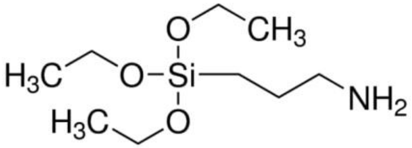
1,1,3,3-Tetramethyldisiloxane (TMDSO)



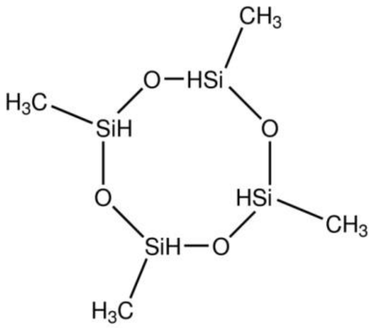
Tetraethyl orthosilicate (TEOS)



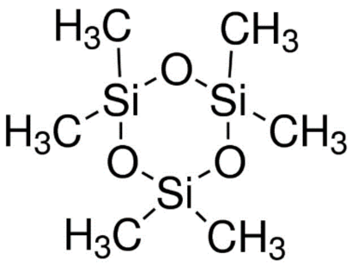
(3-Aminopropyl)triethoxysilane (APTES)



2,4,6,8-Tetramethylcyclotetrasiloxane (TMCTS)

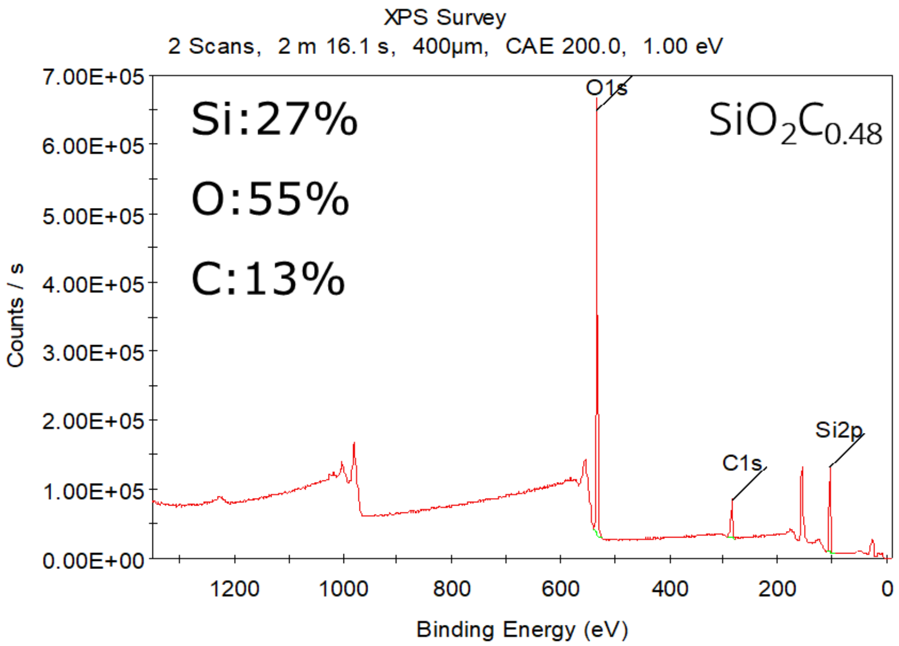


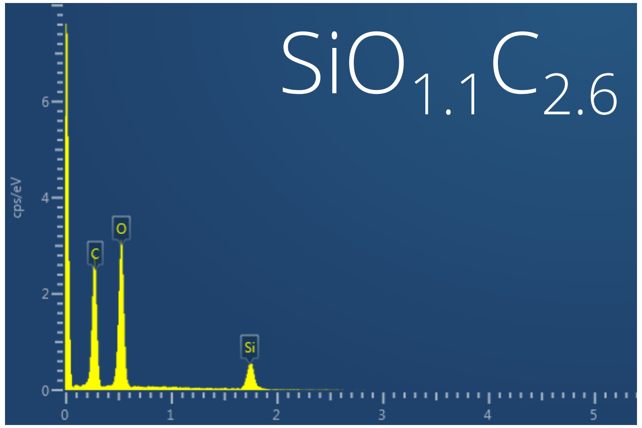
Hexamethylcyclotrisiloxane (HMCTS) or D3 (volatile solid)

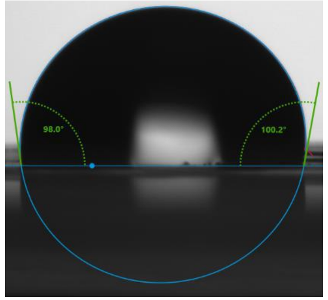


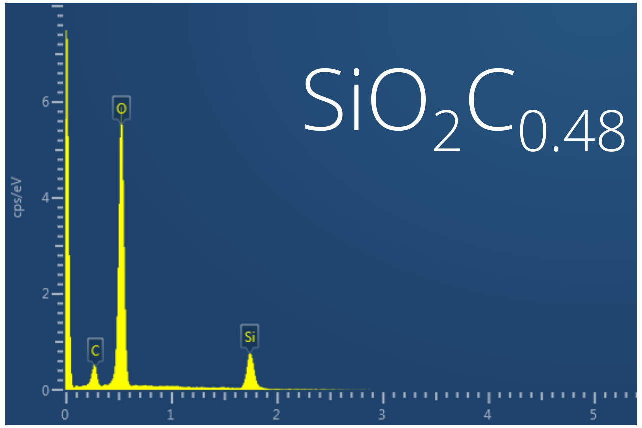
# What $SiO_x/SiO_xC_yH_z$ can be synthesized?

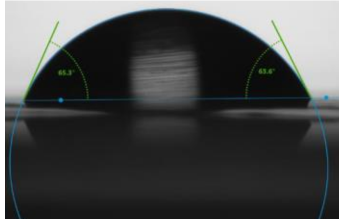
- Depending on a precursor choice and the oxygen concentration, both hydrophilic (silica-like, relatively hard) and hydrophobic (silicone-like, relatively soft) coatings with different barrier properties can be synthesized
- Typical thickness of 50-300 nm
- Compositional analyses and characterisations: FTIR, Energy Dispersive X-Ray (EDX), X-ray photoelectron spectroscopy (XPS), SEM, contact angle measurements etc.



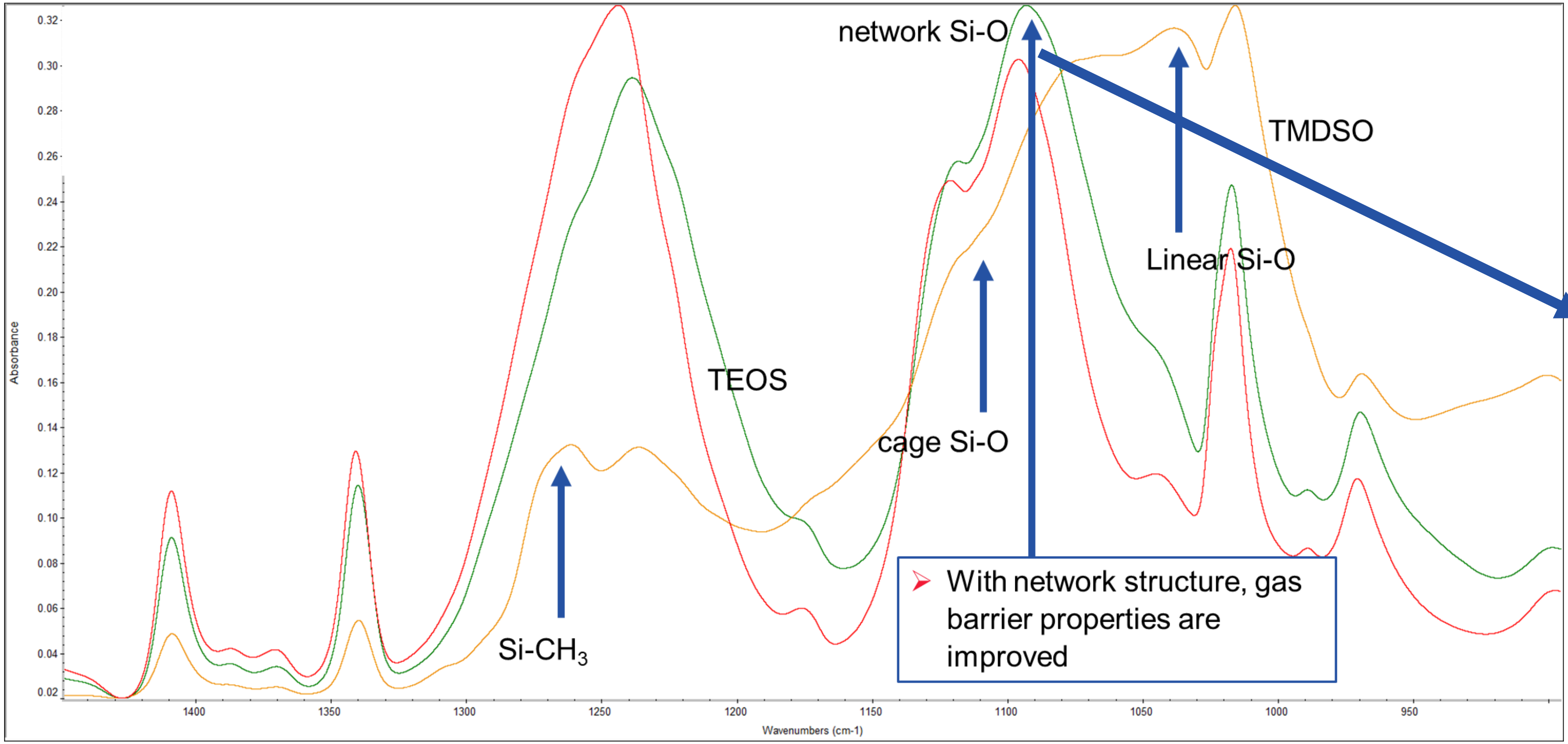



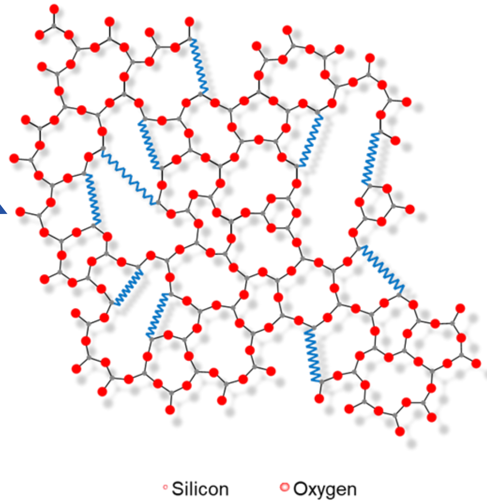





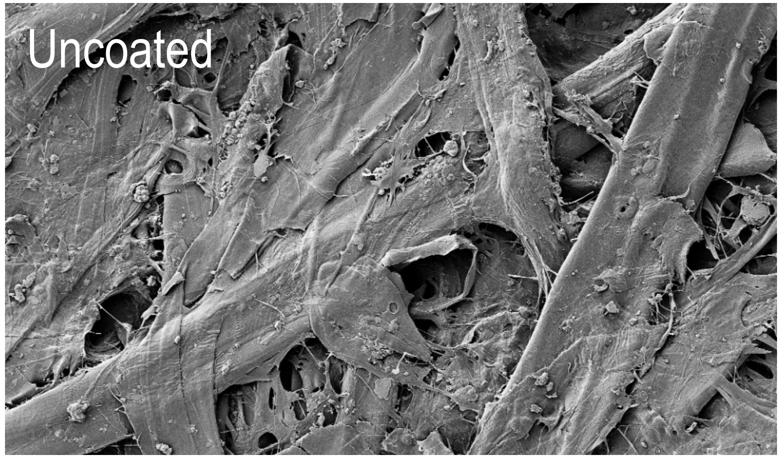



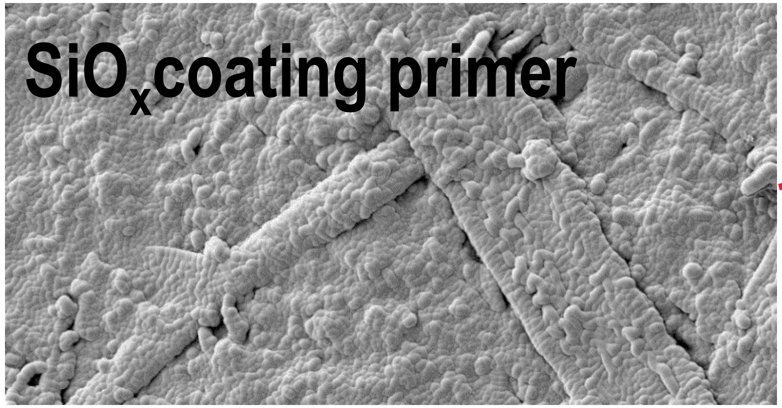
# FTIR spectra of $SiO_x/SiO_xC_yH_z$ PECVD coatings derived from different precursors

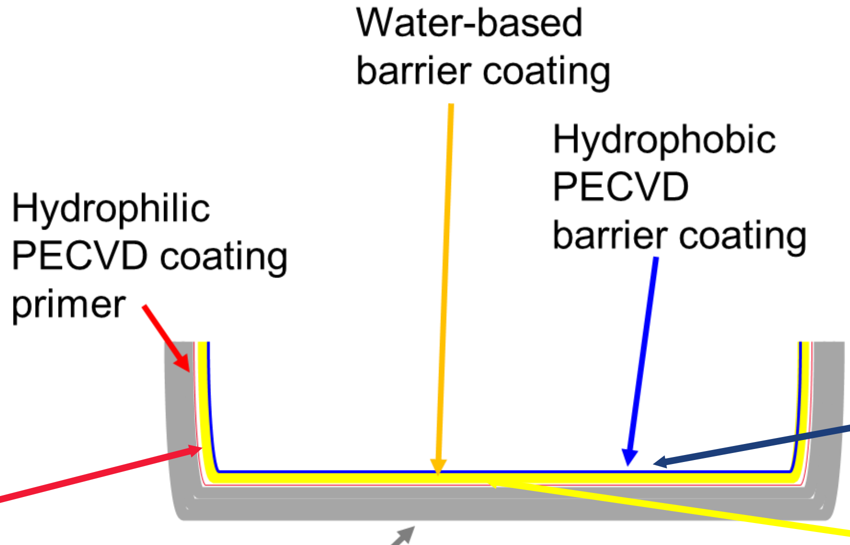


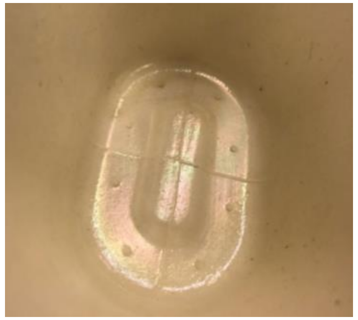



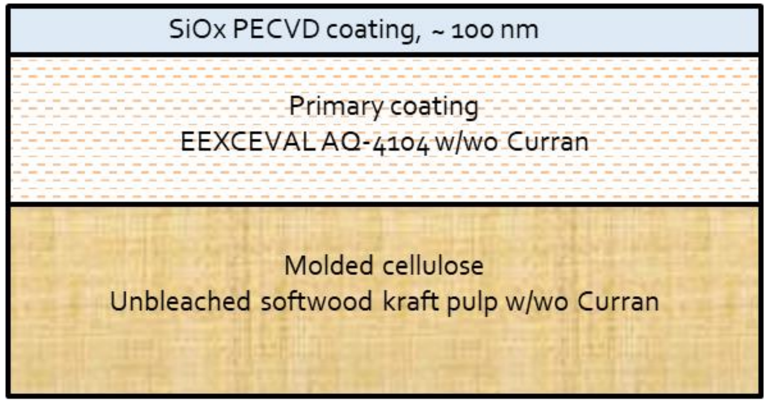
# PECVD coatings as primers and barriers

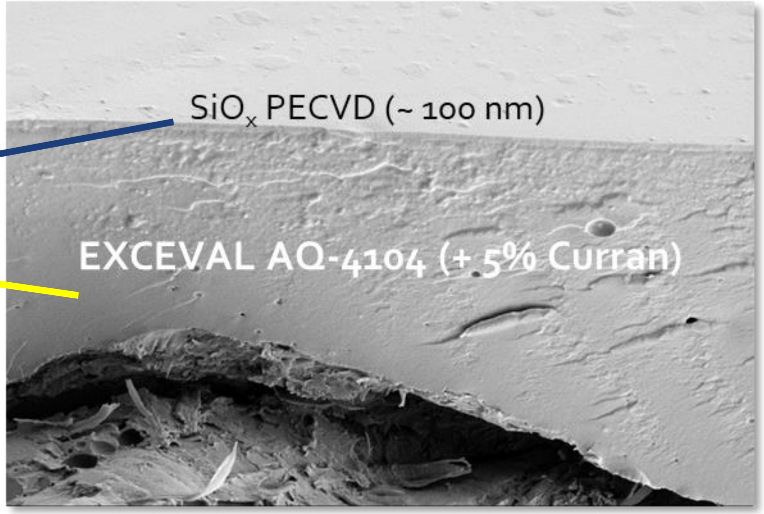




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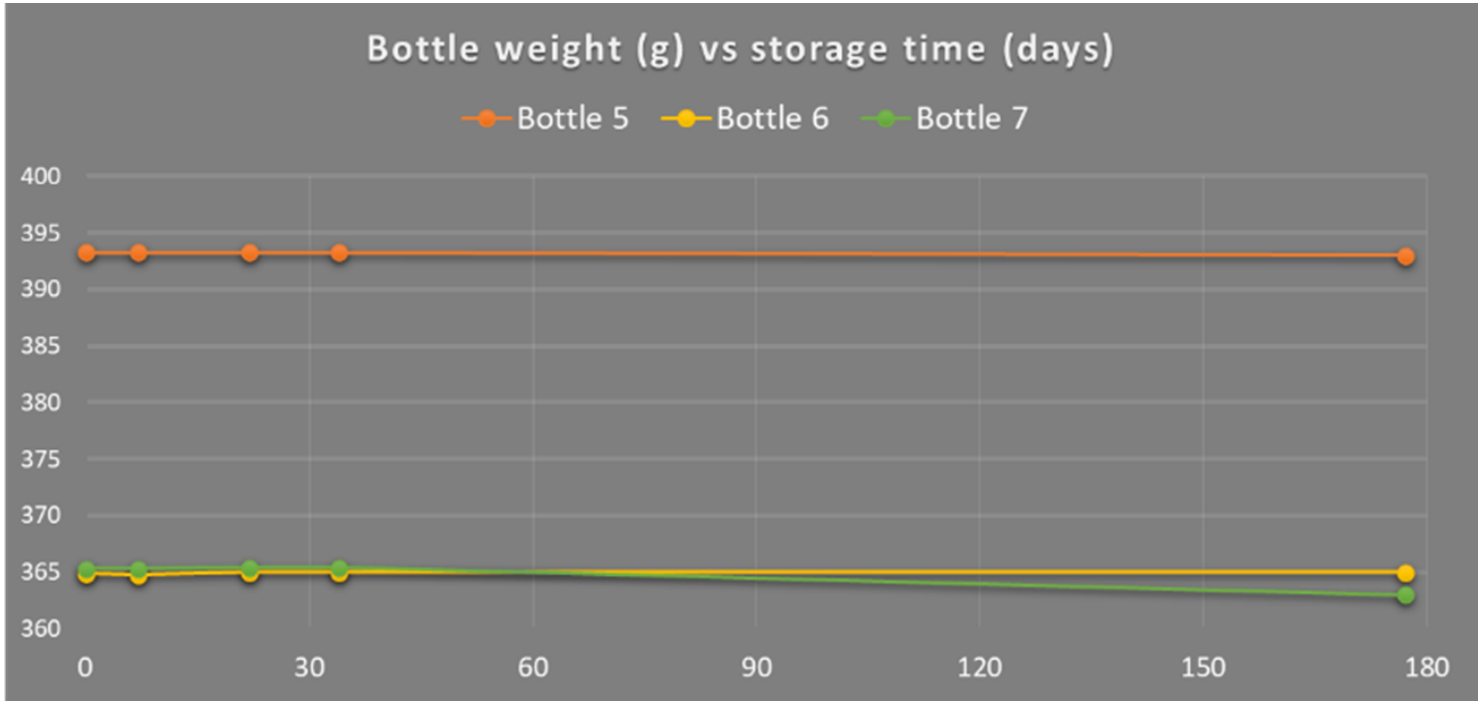
# Example of combining a PECVD SiOx primer and a water-based barrier coating

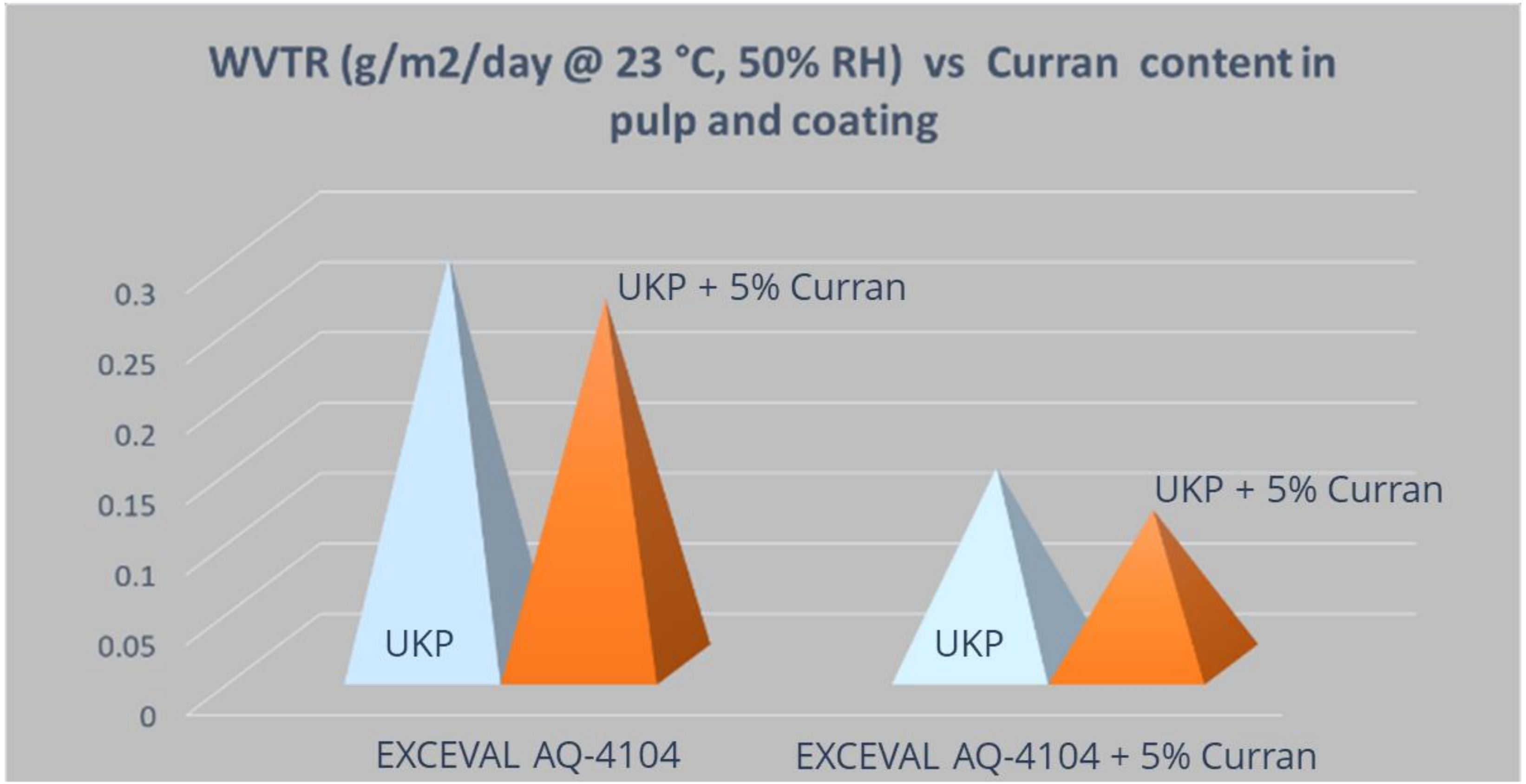
- Molded pulp bottles with PECVD SiO2C0.48 primer and REEF-2 coating
- 177 days in a fridge with 0.5%-fat natural yogurt, no mass loss, no bottle softening or staining happened

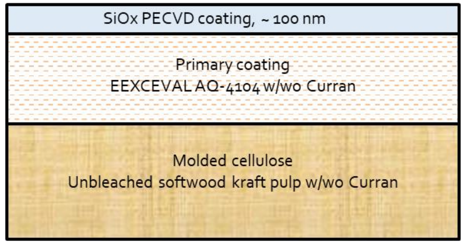


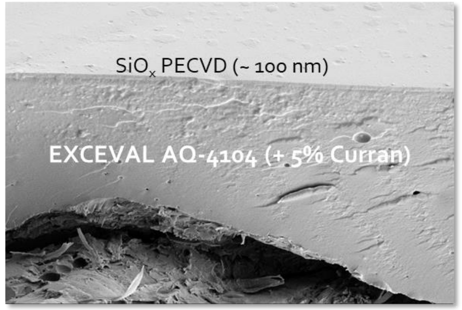


# Example of combining a water-based coating primer and PECVD SiOx barrier coating

- Curran® is in both pulp and water-based coating

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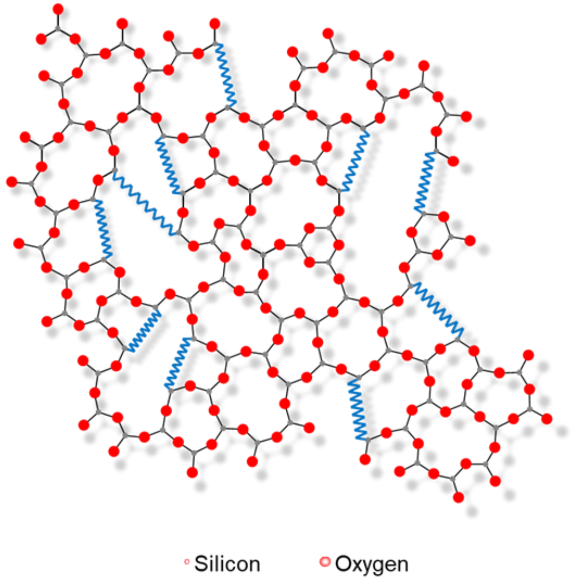
# $SiO_x$ / $SiO_xC_yH_z$ sol-gel and PECVD coatings

- Enhanced barriers for moisture and oxygen, overperforming any plastic and even metal of similar thickness
- Enhanced flexibility as compared to pure silica ($SiO_2$) coatings
- Chemical stability
- Water and oil repellency
- Chemical neutrality - no migration, no plastic pollution
- Well known to food packaging industry (e.g. anti-dew coatings)
- Deposition flexibility:
  - **plasma coating**, PECVD (10-1000 nm) or/and
  - **liquid coating**, sol-gel (1-10 µm) – **spraying** or even **printing**!



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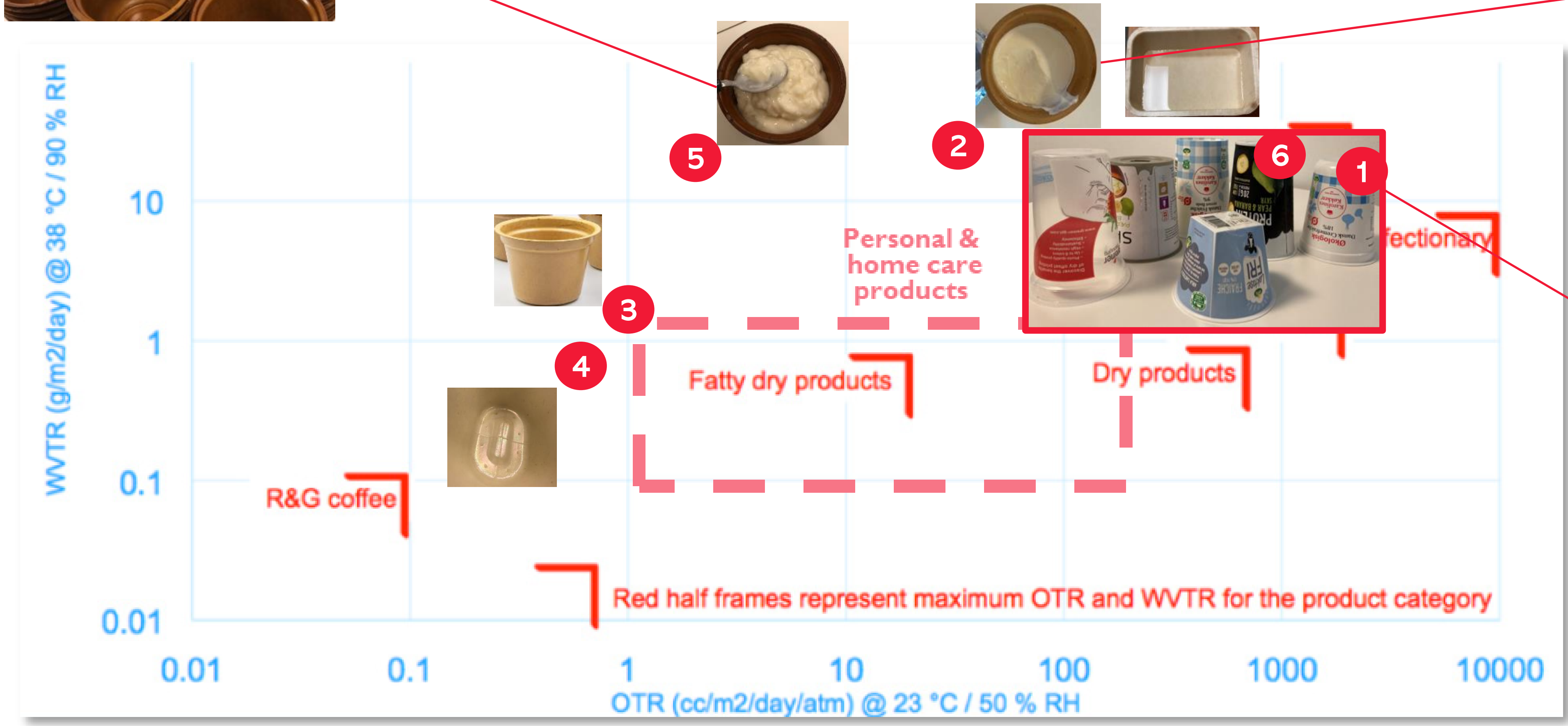
# Barrier performance overview

| | Pulp | Molded object | Interior coating(s) | Exterior coating(s) |
|---|---|---|---|---|
| 1 | 98% UKP + 2% Curran | Bottle | REEF-2 | REEF-2 |
| 2 | 95% UKP + 5% Curran | Cup, tray | REEF-2 | REEF-1 |
| 3 | 95% UKP + 5% Curran | Cup | EXCEVAL + 5% Curran / REEF-2 | EXCEVAL + 5% Curran / REEF-2 |
| 4 | 95% UKP + 5% Curran | Cup | EXCEVAL + 5% Curran / PECVD $SiO_2C_{0.48}$ | EXCEVAL + 5% Curran /PECVD $SiO_2C_{0.48}$ |
| 5 | 95% UKP + 5% Curran | Cup | EXCEVAL / ICO 020 | EXCEVAL / ICO 020 |
| 6 | Air-laid, dry-molded | Tray | ICO 060 / REEF-2 | ICO 060 / REEF-2 |

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**Thank you for your attention!**


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