



BIO4MAP - GA 606144

Project presentation

Transparent and high barrier biodegradable film and sheet for customised Modified Atmosphere Food Packaging

Natural wax based coatings
Martina Lindner

www.bio4map.eu
bio4map@aimplas.es

This project has received funding from the European Union's Seventh Framework Programme for research, technological development and demonstration.



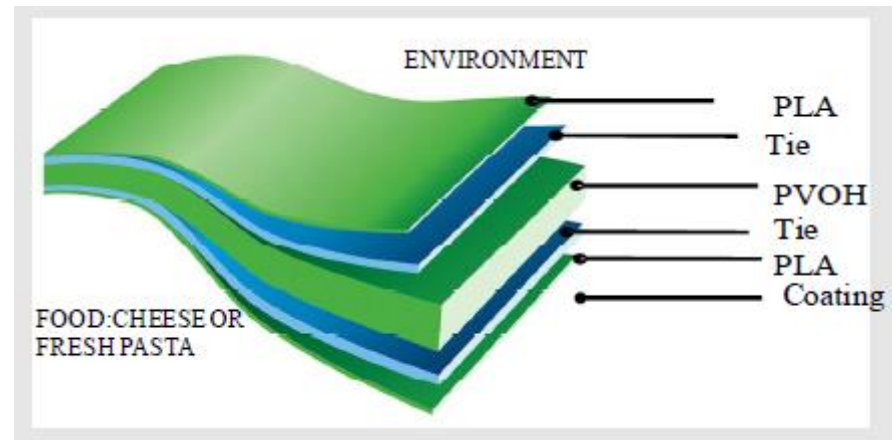
EMPACK fair (Madrid)
18-19 November 2015



Agenda

2

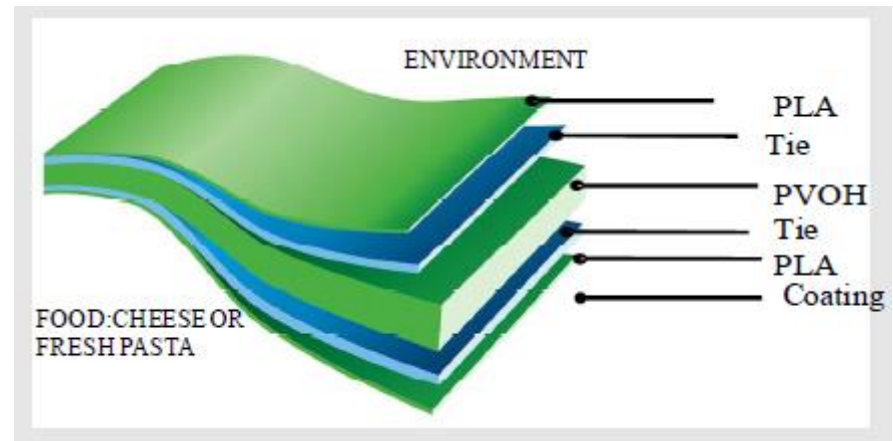
- Introduction to Fraunhofer IVV
- Wax extraction
- Permeation through pure waxes
- Formulation development
 - ▣ Effects on particle size
 - ▣ Effects of blending
- Pilot plant trials



Agenda

3

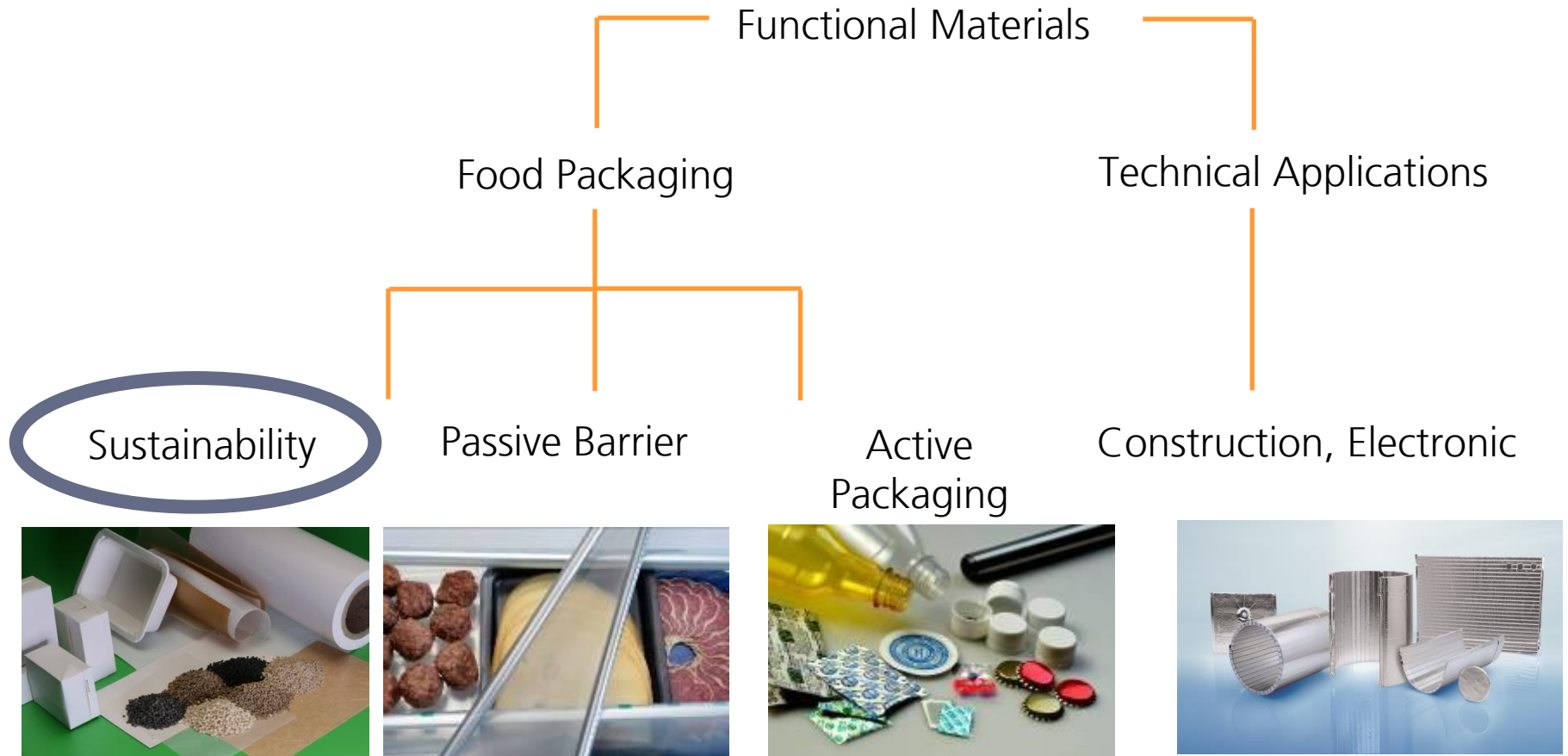
- Introduction to Fraunhofer IVV
- Wax extraction
- Permeation through pure waxes
- Formulation development
 - ▣ Effects on particle size
 - ▣ Effects of blending
- Pilot plant trials



Business Units IVV

4

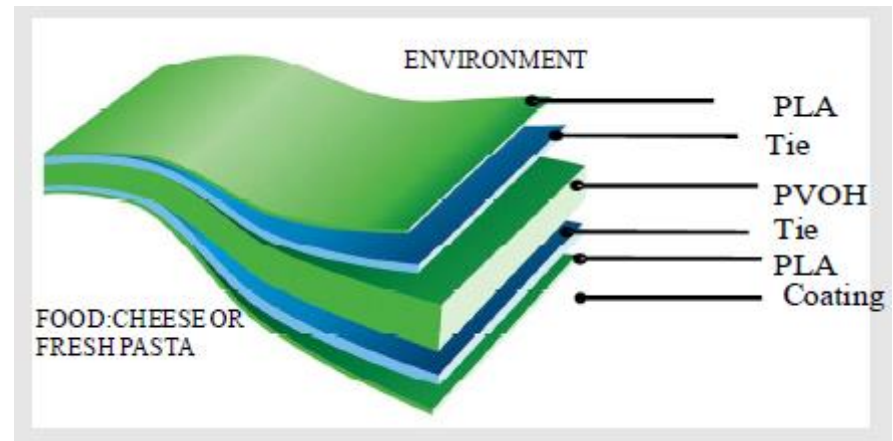




Agenda

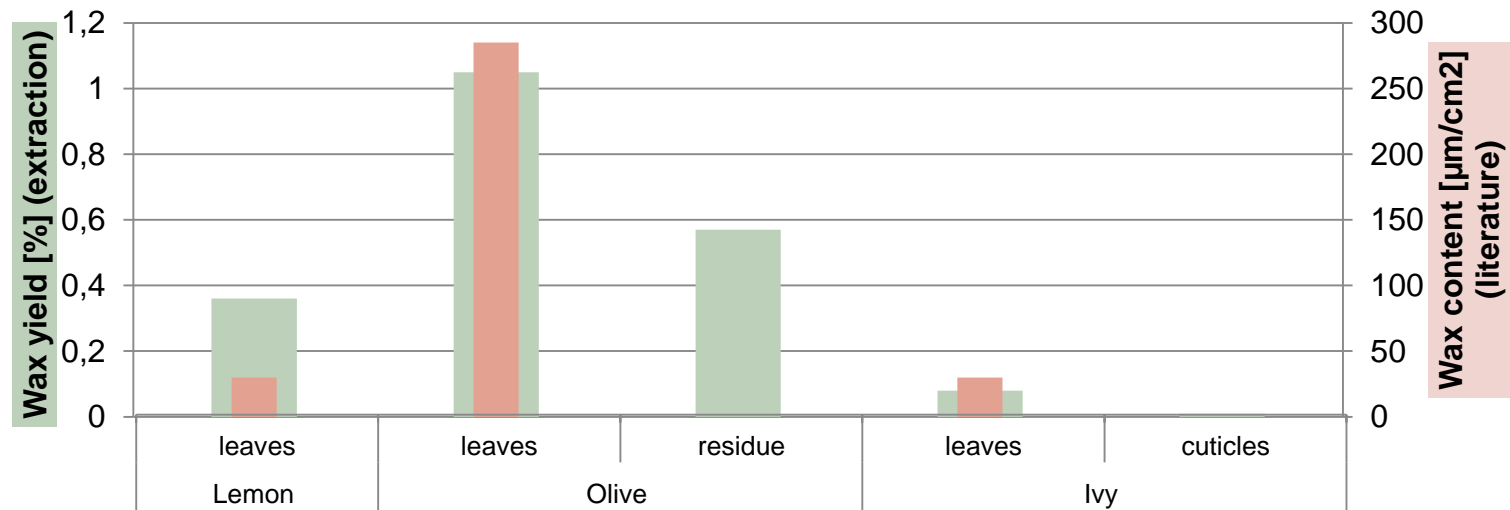
6

- Introduction to Fraunhofer IVV
- Wax extraction
- Permeation through pure waxes
- Formulation development
 - ▣ Effects on particle size
 - ▣ Effects of blending
- Pilot plant trials



Selection of sources for natural waxes

7

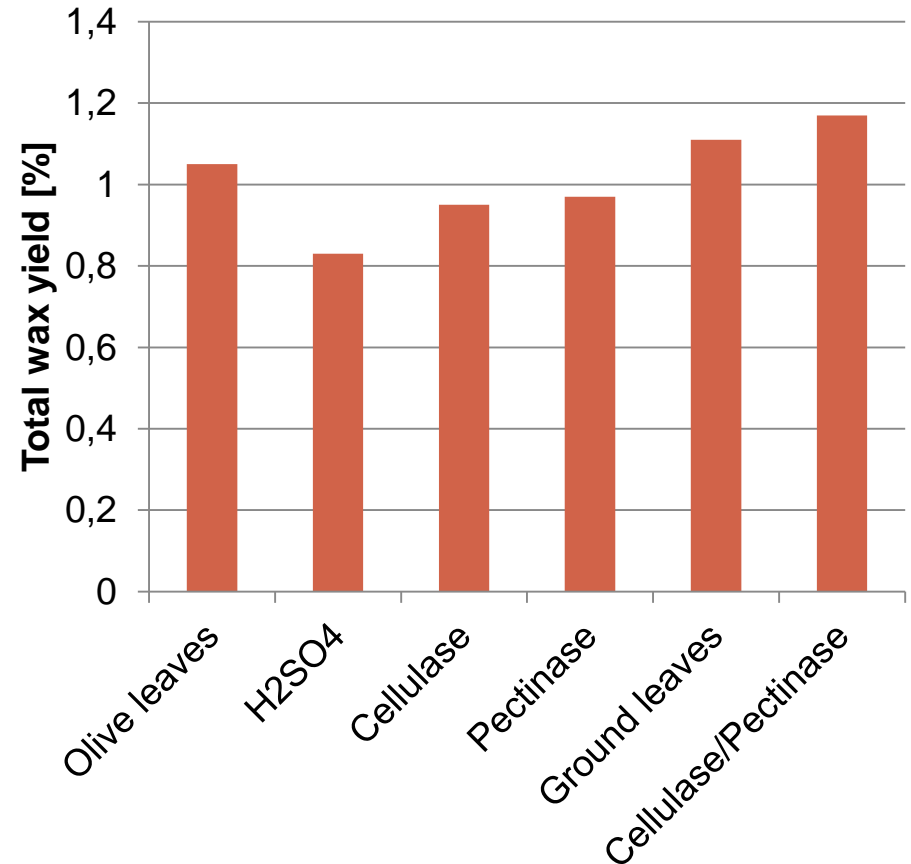
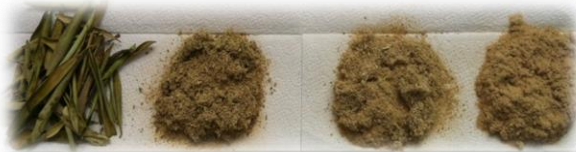


- Due to significant contents of natural waxes leaves from citrus, olive and ivy as well as needles from European spruce were planned to be utilized as raw material for wax extraction
- Referring to the extraction yield the olive leaves seem to be the most suitable extraction material

Raw material preparation

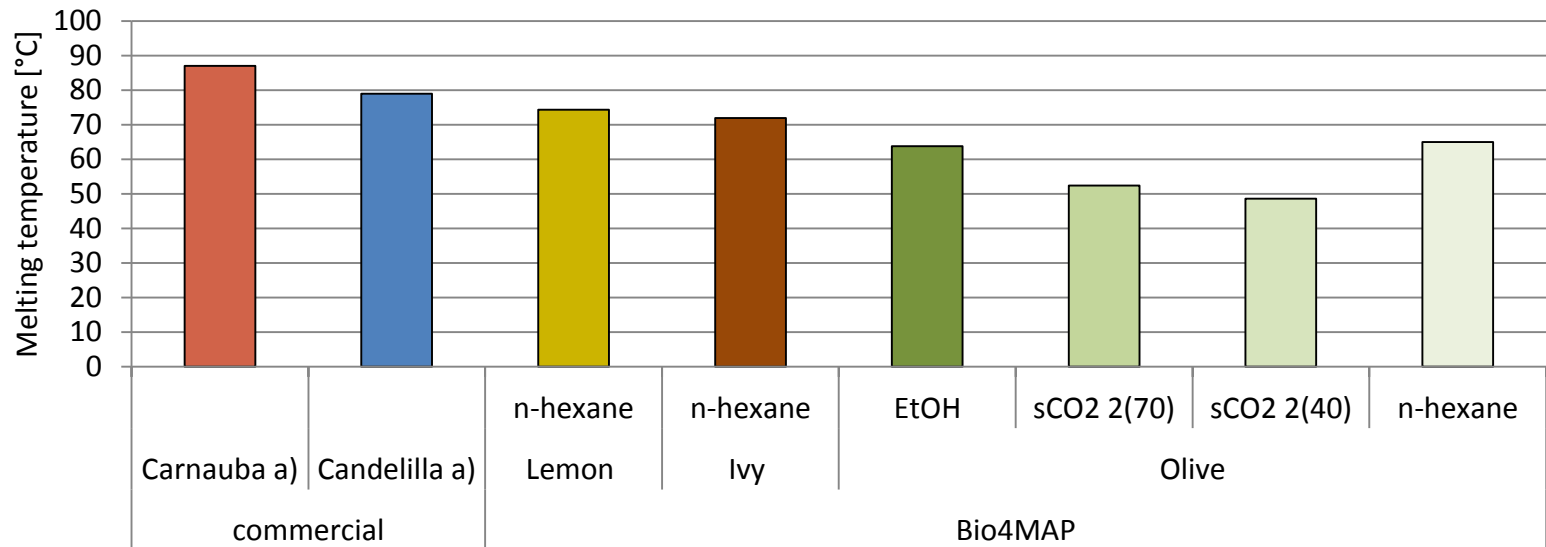
8

- Removal of impurities from the raw material
- Enhanced accessibility of the cutin polymer and waxy components by pre-treatment
- pre-treatment rather lead to the extraction of unwanted compounds than improvement of extraction yield



Thermal properties by differential scanning calorimetry (DSC)

9



^{a)}Pörschke, S., Herstellung pulverförmiger Biowachse und mögliche Anwendungen, in Fakultät für Maschinenbau 2014, Ruhr-Universität Bochum: Bochum

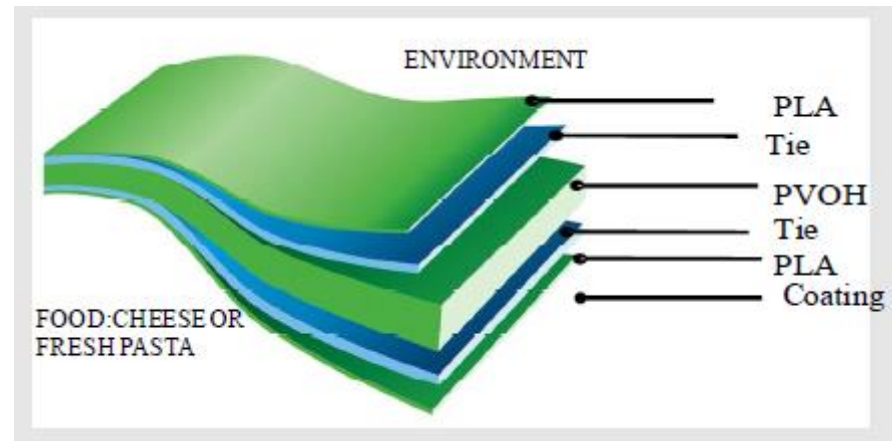
➤ Considerations

- Melting temperature shouldn't be higher than PLA substrate
- High melting temperature limits processability as dispersion in EtOH
- For sealing applications the melting temperature should ideally be in the range of common polymers like PE (>100°C)
- Considering extraction yield, EtOH extracted olive cuticula wax is most promising

Agenda

10

- Introduction to Fraunhofer IVV
- Wax extraction
- Permeation through pure waxes
- Formulation development
 - ▣ Effects on particle size
 - ▣ Effects of blending
- Pilot plant trials

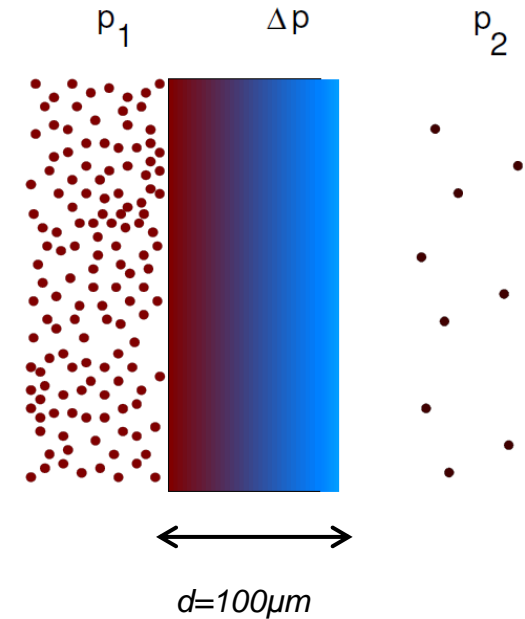
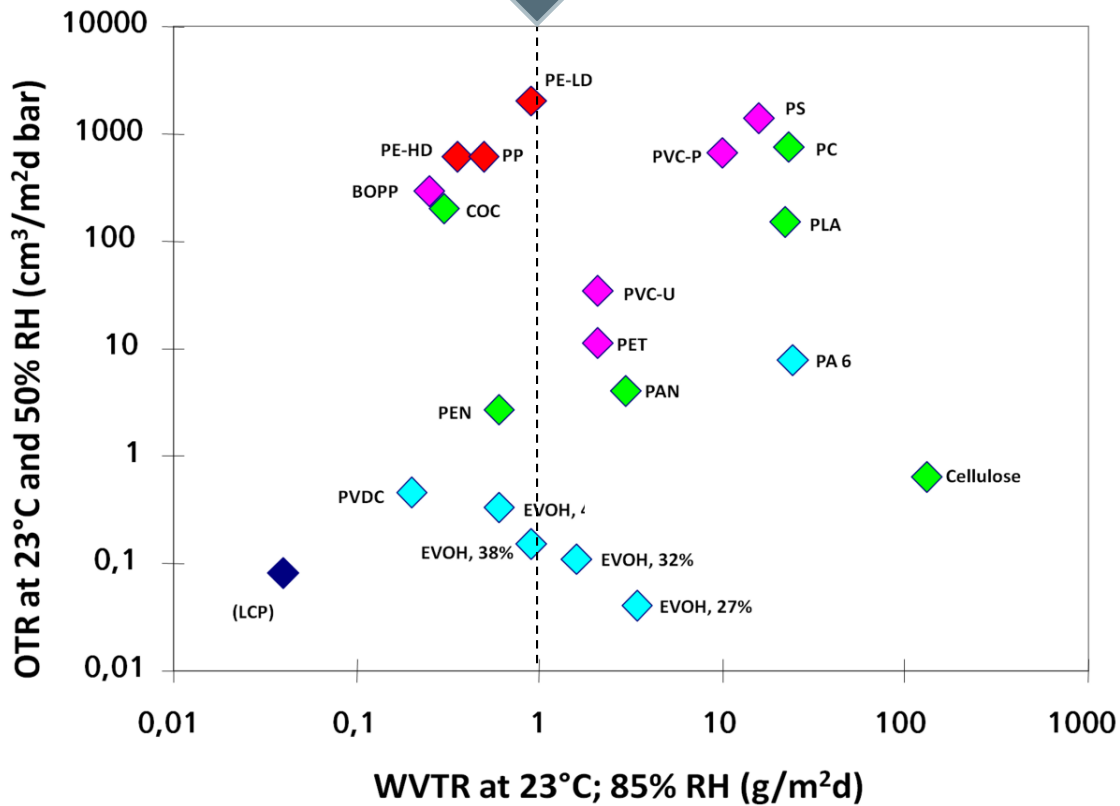


Permeation through pure waxes

11

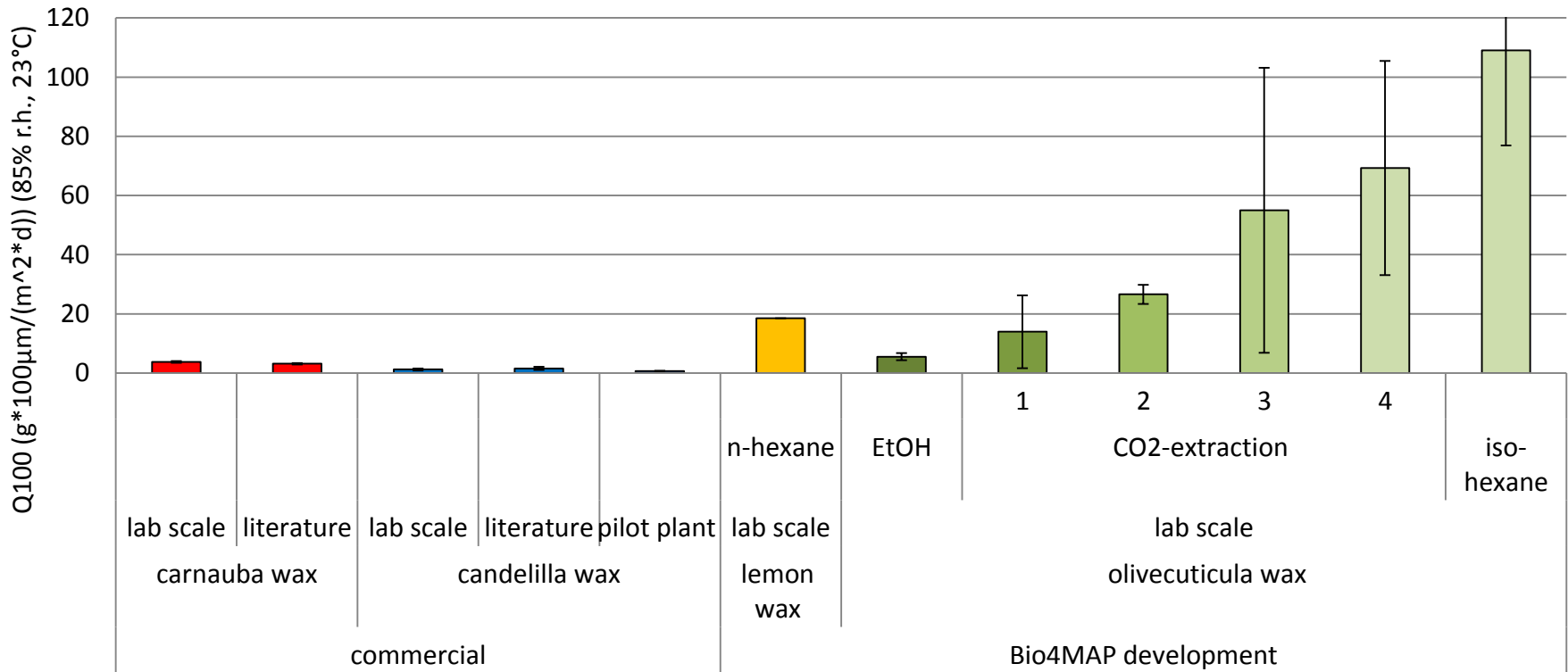
Goal: WVTR
(100 μ m) < 2g/(m²*d)
(85% r.h., 23°C)

Values standardized to
a thickness of 100 μ m



Permeation through pure waxes

12

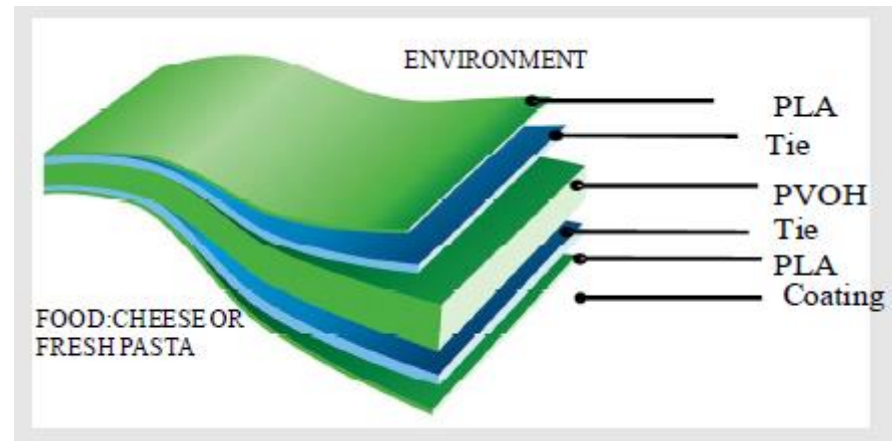


- EtOH extracted olive cuticula wax is comparable to commercial waxes
- Additional extraction cycles lead to extraction of other compounds which compromise the barrier

Agenda

13

- Introduction to Fraunhofer IVV
- Wax extraction
- Permeation through pure waxes
- Formulation development
 - ▣ Effects on particle size
 - ▣ Effects of blending
- Pilot plant trials

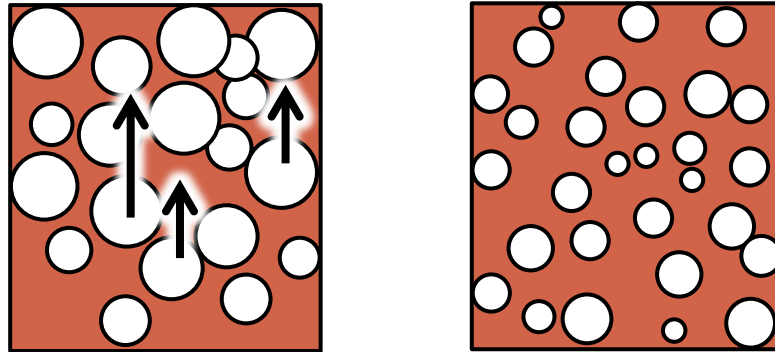


Particle size

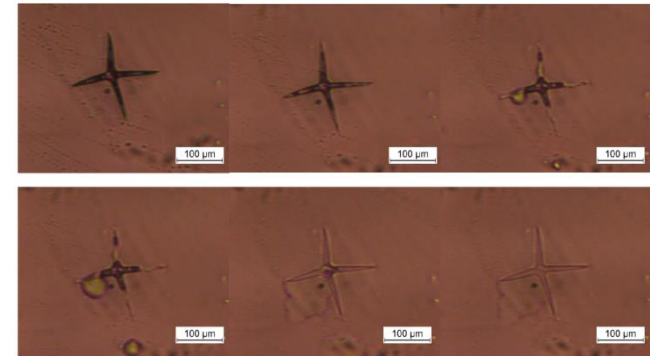
14

- particle size affects the creaming of the dispersion according to Stokes

$$v = \frac{d^2 \cdot (\rho_K - \rho_{Fl})}{18 \cdot \eta} \cdot g$$



- big dimensions lead to unmolten particles which induce cracks and low barrier

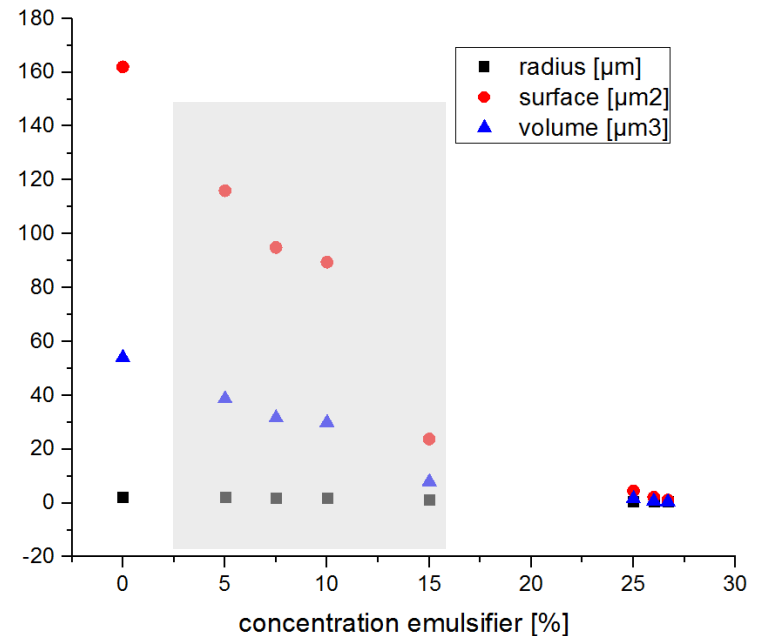
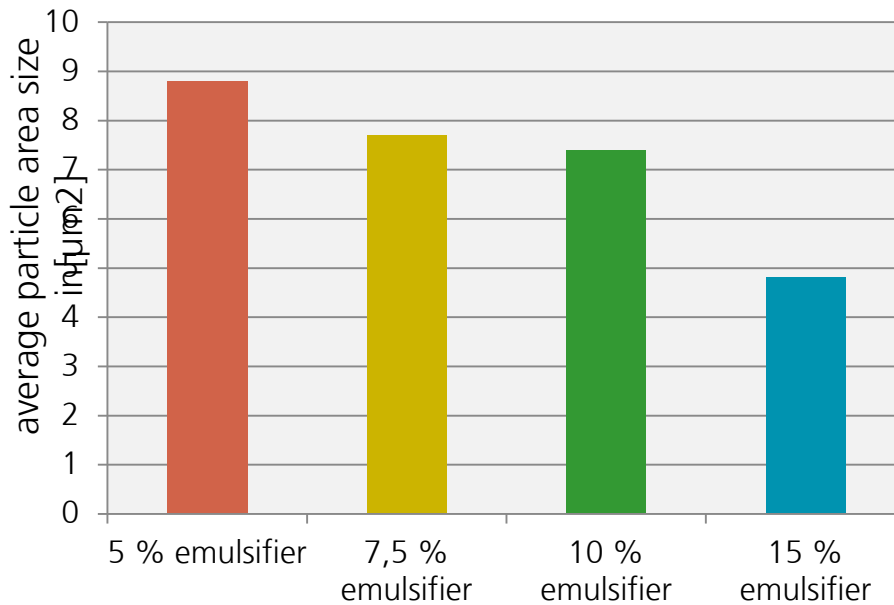
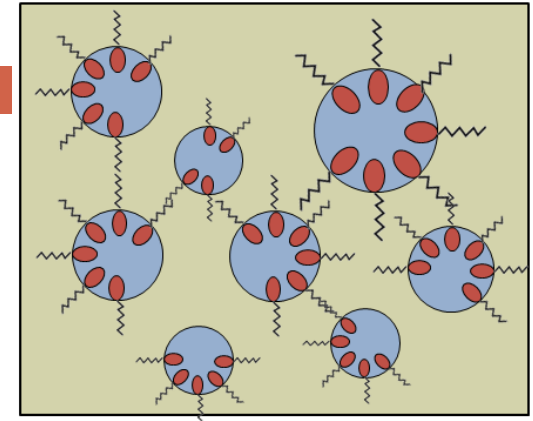


- finding ways to adjust particle size in order to receive small, evenly distributed particles
 - ▣ A) increasing the emulsifier concentration
 - ▣ B) reducing the wax concentration
 - ▣ C) Artibal additives

Emulsifier concentration

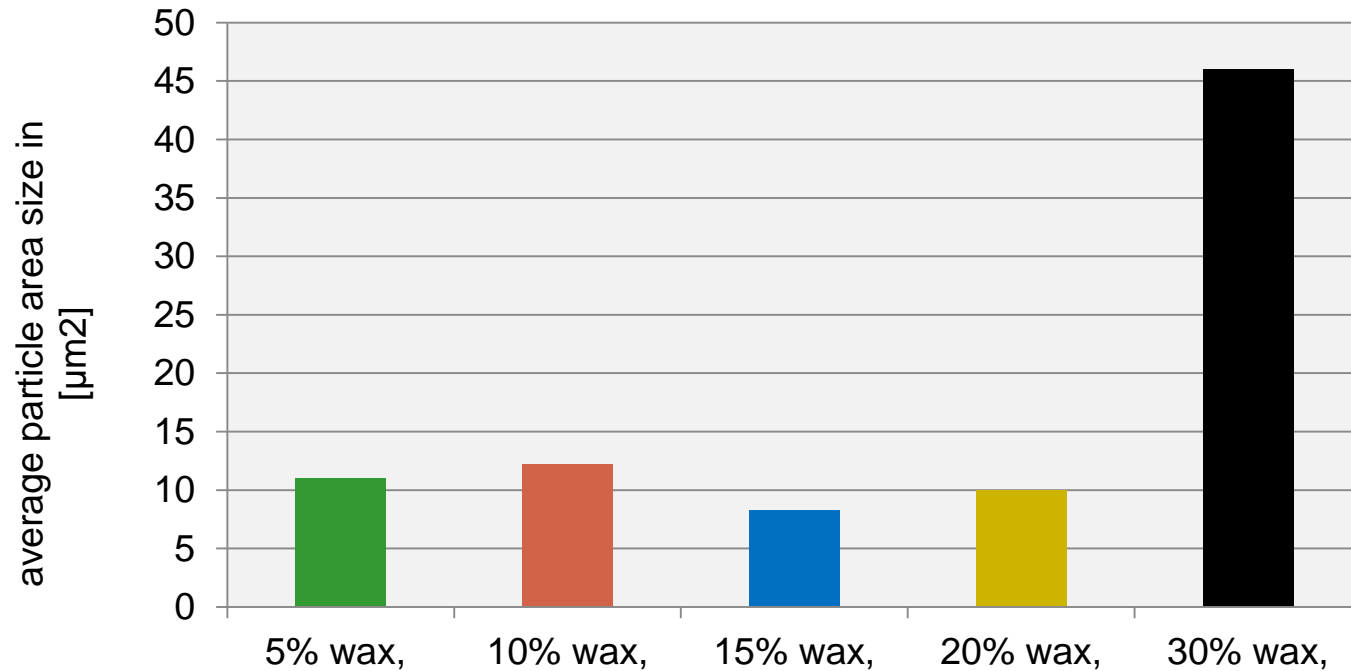
15

- Lower particle size induces higher specific surface and higher necessary amount of emulsifier
- Maximum emulsifier concentration approx. 25%
- However, according to literature, the emulsifier **compromises the water vapour barrier**

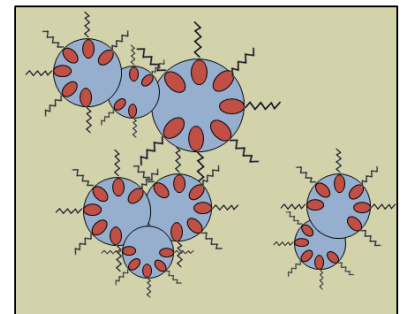


Wax concentration

16

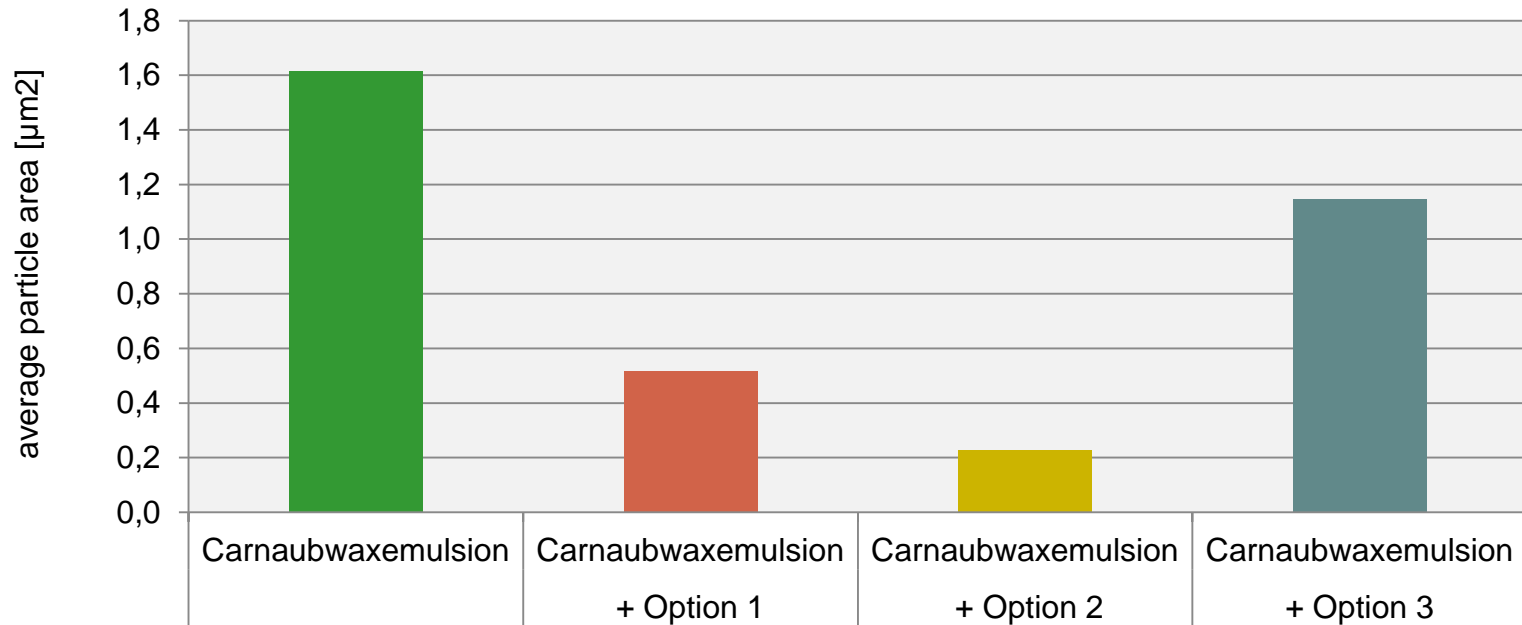


- at 30% first particles agglomerate again
- therefore the average particle size is extremely high
- → max. wax concentration is about 20 %



Artibal additives

17



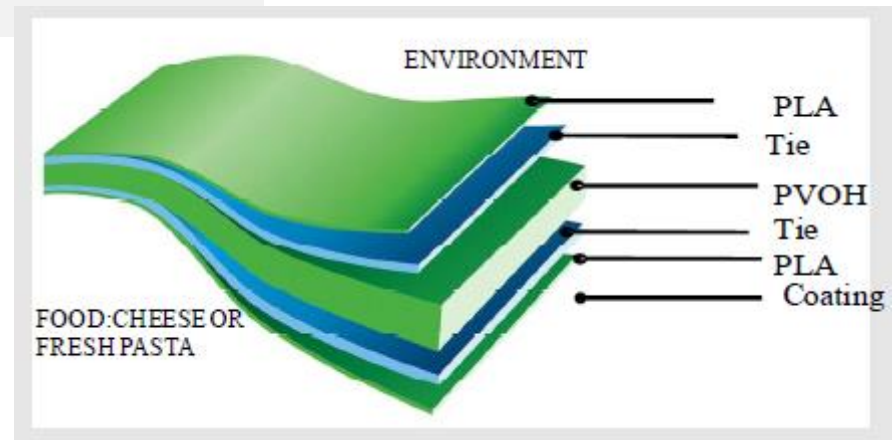
- Nitrocellulose based Artibal additives 1-3 have emulsifying character

artibal 

Agenda

18

- Introduction to Fraunhofer IVV
- Wax extraction
- Permeation through pure waxes
- Formulation development
 - ▣ Effects on particle size
 - ▣ Effects of blending
- Pilot plant trials

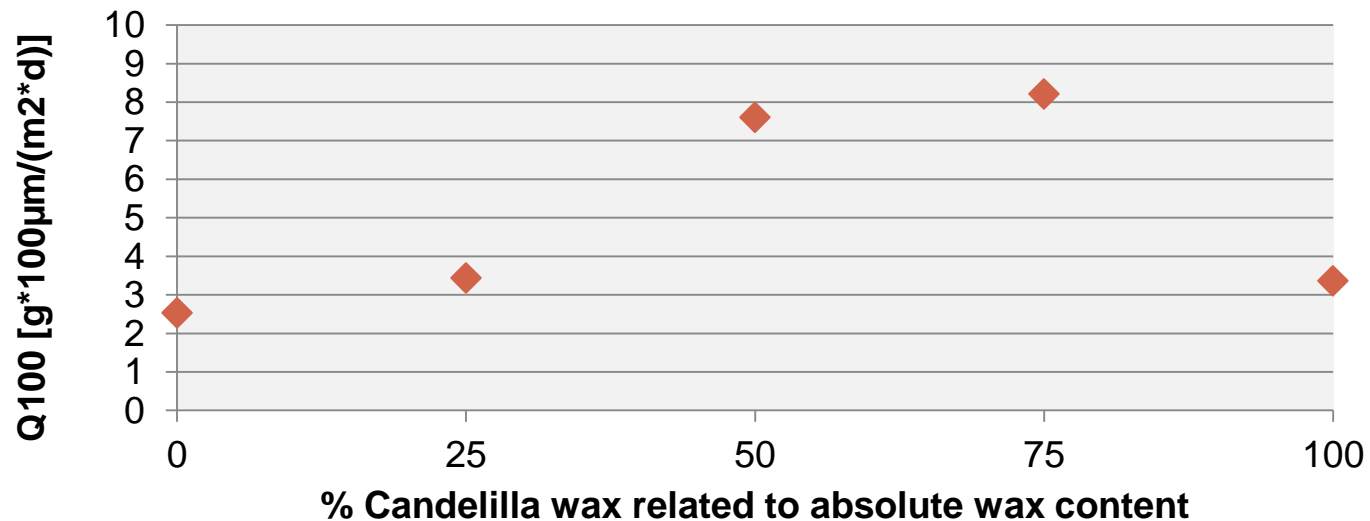
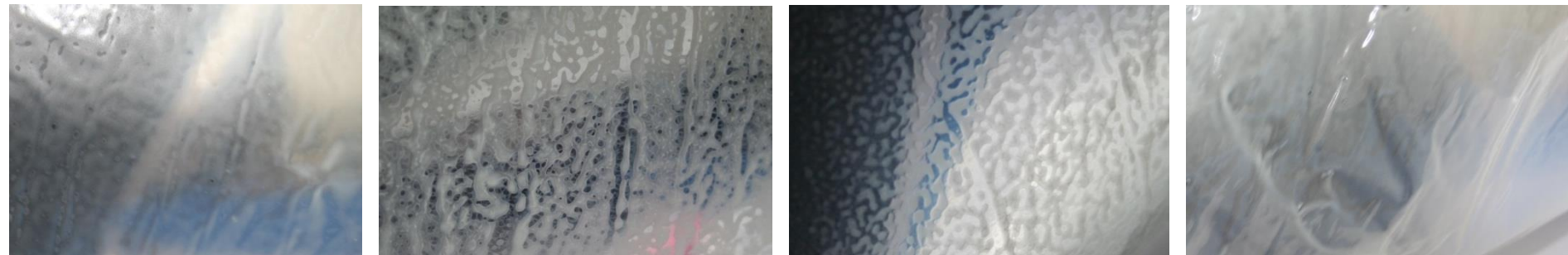


Blending Carnauba and Candelilla wax

19

Carnauba wax

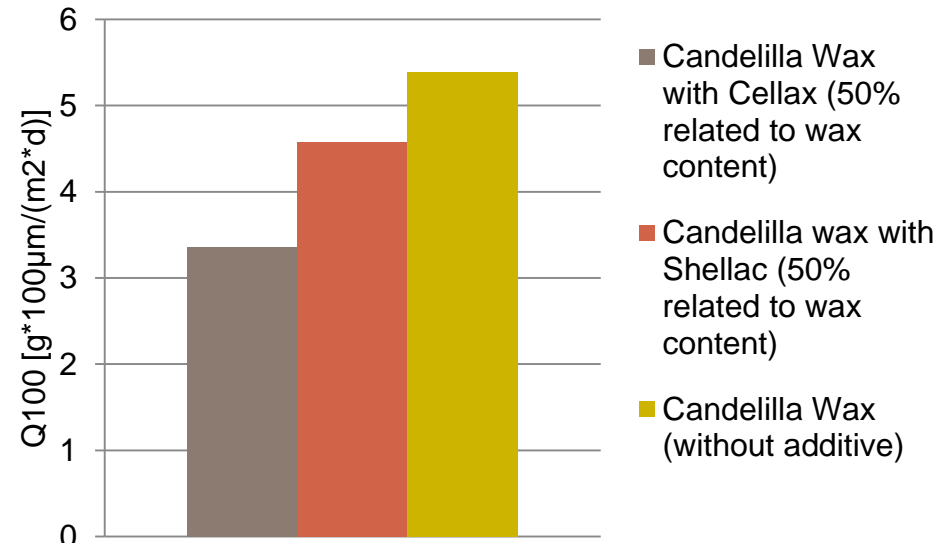
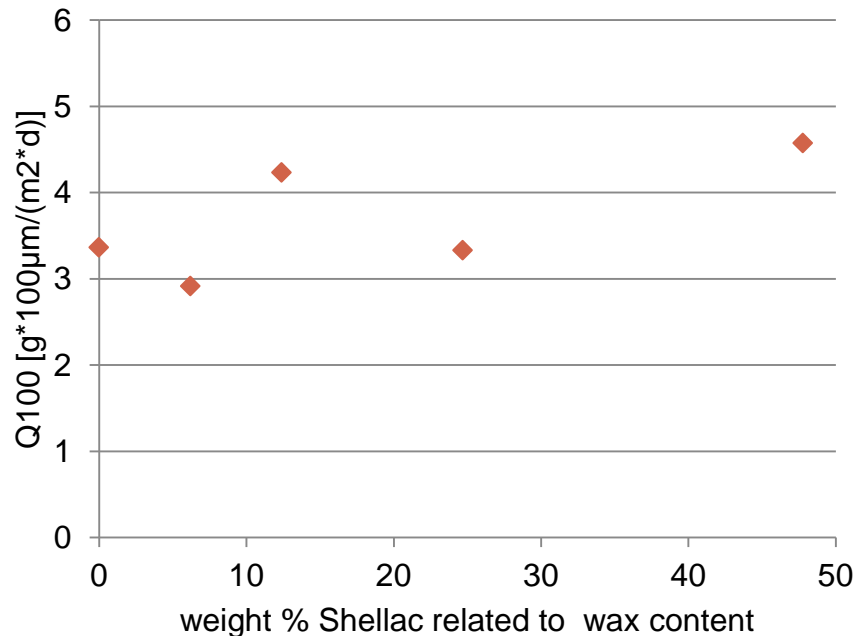
Candelilla wax

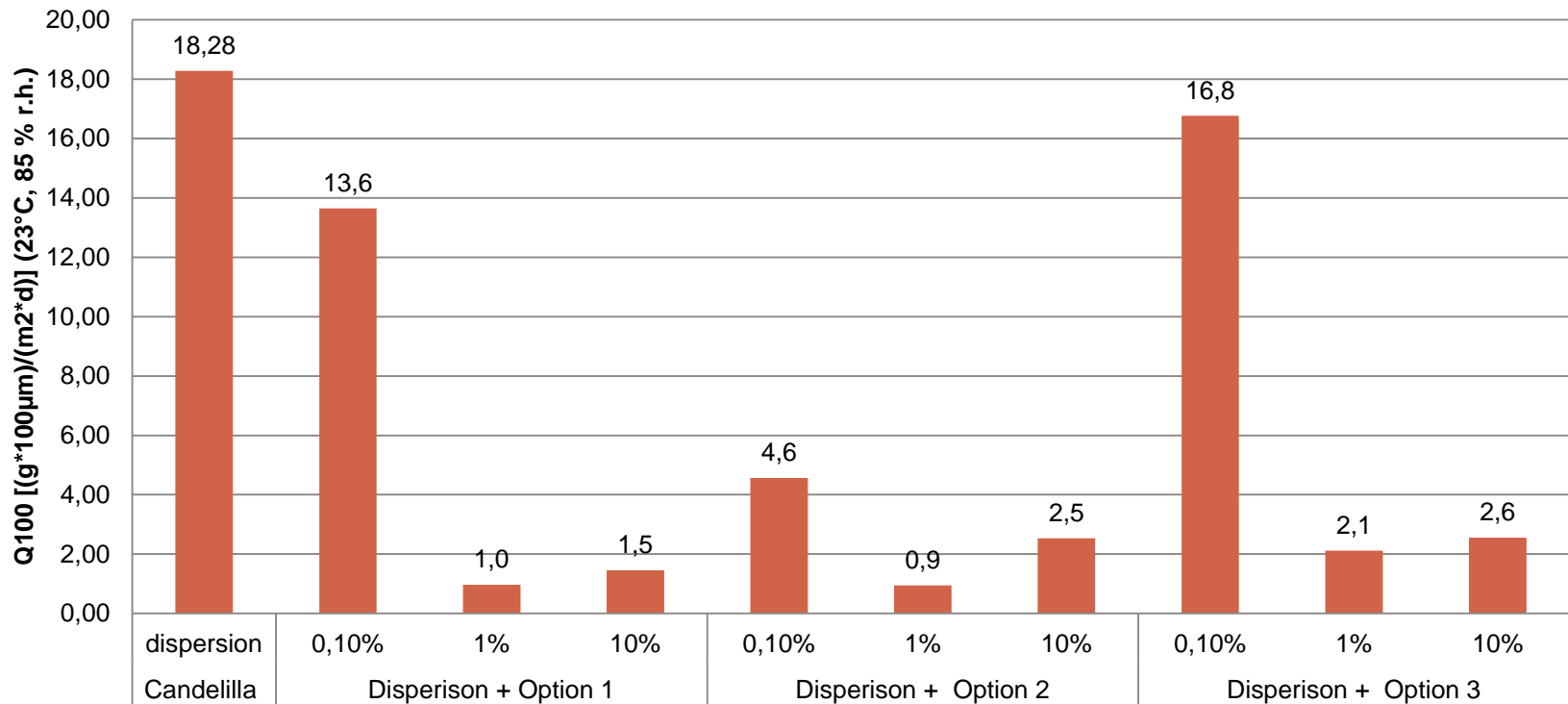


Adding Shellac and Nitrocellulose

20

- Shellac: resin secreted by the female lac bug
- Nitrocellulose: biobased binder for inks and lacquers
- Although surface finish was improved, the barrier decreased



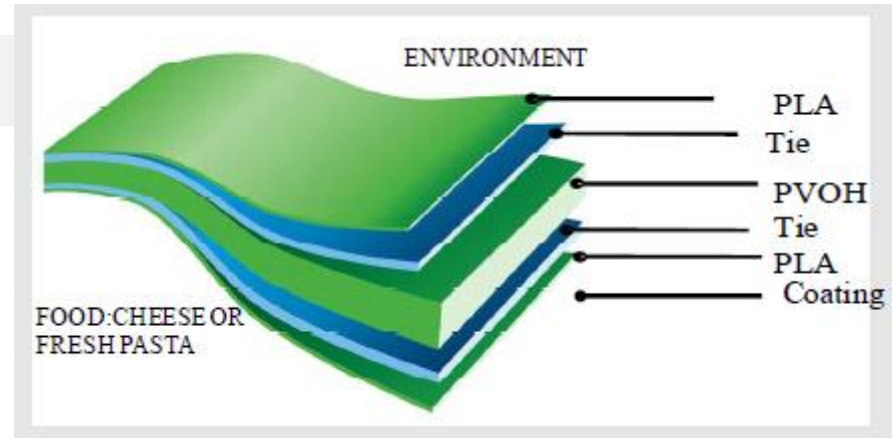


- ▣ Nitrocellulose based option 1-3 show barrier improvements and emulsifying character
- ▣ Option 1 is chosen due to FDA approval

Agenda

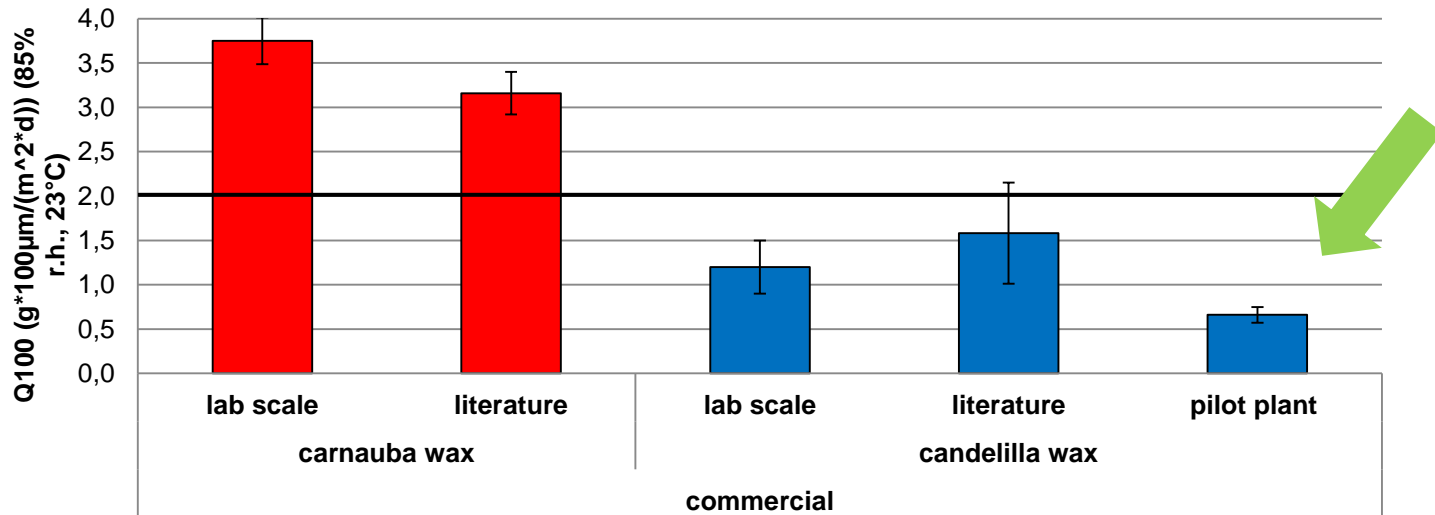
22

- Introduction to Fraunhofer IVV
- Wax extraction
- Permeation through pure waxes
- Formulation development
 - ▣ Effects on particle size
 - ▣ Effects of blending
- Pilot plant trials



4. Pilot plant trials

23



- ❑ Candelilla wax showed improved barrier in pilot plant trial in comparison with lab scale
- ❑ **Goal WVTR (100μm) < 2g/(m²*d) was reached!**

Outlook

24

- Extraction conditions have extensively been investigated
- However, extraction yield is low due to limited wax content of raw material
- Melting points are comparably low
- Particle size can be reduced by means of suitable additives
- Barrier is improved by adding nitrocellulose based additives from Artibal
- Scratch resistance needs to be improved
- **Goal WVTR (100 μ m) < 2g/(m²*d) was reached!**
- **All components are FDA approved**
- Water vapour barrier comparable to PE, PVOH, PP

Thank you for your attention



Martina Lindner

Martina.lindner@ivv.fraunhofer.de

Daniela von der Haar

daniela.von.der.haar@ivv.fraunhofer.de

**Fraunhofer Institute
Process Engineering
and Packaging IVV
Giggenhauser Strasse 35
85354 Freising
Germany
Phone: +49 8161 491-536**