

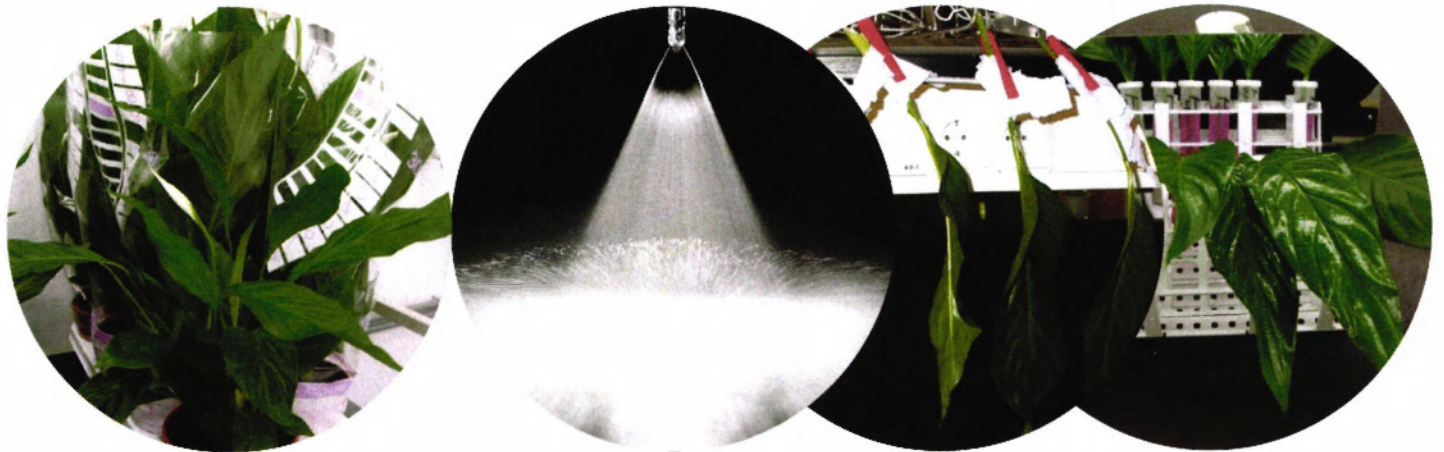
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# Coatings voor potplanten

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Final report

14-08-2012, Erika Róth and Ulphard Thoden van Velzen



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# Agenda

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1. Goal
2. Demands to comply with
3. Summary of First stage
4. Results of Second stage
5. Conclusions
6. Next step?

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## Goal

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Increase the no-care lifespan of potted plants by the application of a novel coating and therefore increasing selling time for both flowering and leafy ornamentals

# Requirements for the new product

Initial requirements	Fulfilment	Specified after First stage	Fulfilment
Transparent	✓	Water soluble	✓ (less effective than Leafshine)
Extending shelf life and marketability	Likely	No high shine	✓
Improved anti-transpirant effect	solvent based ✓ water based --	User friendly, REACH chemicals	solvent based -- water based ✓
Applicability for a large assortment	Not yet tested	Not or hardly smelling	solvent based -- - processing ✓ - final product water based ✓
Non-phytotoxic	✓	Raw material cost less than 4 €/l	(✓)
		Immediate dry surface after spray	solvent based ✓✓ water based ✓
		Homogenous concentrate and RTU	✓

## Plans and achievements

Phase	Plan	Realisation
Phase 1 – Project definition	<ul style="list-style-type: none"> <li>Detailed workplan</li> <li>Contract</li> </ul>	<ul style="list-style-type: none"> <li>Detailed workplan</li> <li>Contract</li> </ul>
Phase 2 - Invention	<ul style="list-style-type: none"> <li>Aim of coating</li> <li>Application mode</li> <li>Type of polymer</li> <li>Model system</li> </ul>	<ul style="list-style-type: none"> <li>Physical barrier against water loss</li> <li>Spraying</li> <li>Hydrophobic groups</li> <li>Spathiphyllum leaf – optimised test conditions</li> </ul>
Phase 3 - Screening	<ul style="list-style-type: none"> <li>4 basispolymers</li> <li>20-30 coatings</li> <li>On Spathiphyllum leaves</li> <li><b>Select 4 best coatings</b></li> </ul>	<ul style="list-style-type: none"> <li>Tested polysaccharides, oils, waxes</li> <li>33 coatings (Stage 1) + 27 coatings from concentrates (Stage 2)</li> <li>On Spathiphyllum leaves + 1 test with Hydrangea leaves (Stage 1)</li> <li><b>5 coatings with superior antitranspirant effect</b> compared to Leafshine and with <b>different levels of shine</b> (Stage 1) but only 1 water-based</li> <li>Water-based coatings (Stage 2) underperformed compared to Leafshine</li> </ul>
Phase 4 – Optimisation of best coatings	<ul style="list-style-type: none"> <li>Test best 4 coatings with 3-5 potted plants 1x in FBR</li> <li>Adjust formulation in case of phytotoxicity</li> <li>Select best coating + Why does it work?</li> </ul>	<ul style="list-style-type: none"> <li>Preliminary test has proven the functionality of solvent-based coatings</li> </ul>



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# STAGE 1

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## Results of Phase 2: Invention

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- Model system



Spraying



Drying



Shelf life

# Phase 2: Experimental conditions – Model system

## Raw material:

- Spathiphyllum 'Sweet Silvio' (Plantion)
- Tubes: 35 cm<sup>3</sup> flower tubes with 30 cm<sup>3</sup> "Chrysal Professional 2"
- Repetition: 5 leaves, 10 cm stem



## Spraying:

- Flow cabinet
- Pressure: 3 bar
- Air-brush

## Shelf life

- Light conditions: 12 h light + 12 h dark ( $\pm 250 \mu\text{mol m}^{-2} \text{s}^{-1}$ )
- Temperature: 20 °C
- Humidity: 50% RH

## Realisation of Phase 3: Screening

### Coatings

	Experiment					
	0	1	2	3	4	5
T0	Water	Water	Water	Water	Water	Water
T1	Leafshine	Leafshine	Leafshine	Leafshine	Leafshine	Leafshine
T2	beeswax emulsion in water: 0.5%	PVOH + Carnaubawax	Chitosan	Exsiccator fat	Chitosan + castor oil	PVOH + Paraffin oil A
T3	shellac, 1% in ethanol	PVAC (10 g)	Chitosan + sunflower oil	PEVAC (5 g)	Exsiccator-fat (9 g) + L30648	PEVAC (15 g) + paraffin oil A (15 g)
T4	CMC-glycerol 1% in water	PVAC + SPAN-20	PVAC (10 g) + sunflower oil	PEVAC (5 g) + sunflower oil	PEVAC + paraffin oil A + L30648	PEVAC + paraffin oil B
T5		PVAC + SPAN-20 + Carnuba wax	sunflower oil + beeswax	PEVAC + sunflower oil + beeswax (0.5g)	PEVAC + castor oil	PEVAC + paraffin oil C
T6		sunflower oil (50ml) + carnauba wax	sunflower oil (10ml) + carnauba wax	PEVAC + sunflower oil + beeswax (0.2g)	PEVAC + castor oil + L30648	paraffin oil A + paraffin wax
T7		PEVACMA		low PEVAC + sunflower oil	PEVAC (5 g) + paraffin oil A (5 g)	paraffin oil A + Octadecanamide
T8		PEVACMA + sunflower oil		PVOH + beeswax	commercial wood glue	
T9		PEVACMA + carnauba wax		shellac + beeswax	Leafshine + sunflower oil	

# Summarised result of Experiment 1

		Weight loss day 6	Phytotoxicity	Gloss	texture of the coating	homogeneity of the coating	other comments
T0	Water	55.38% -	no	++ little bit	+ smooth	+ homogenous	+
T1	Leafshine	15.57% ++	no	++ lot	++ smooth	+ homogenous	+
T2	PVOH + carnaubawax	45.26% -	no	++ little bit	+ rough	- homogenous	+
T3	PVAC	48.35% -	no	++ no shine, 80% of the leaf is covered by white powder	++ smooth	+ heterogeneous (some white powder)	-
T4	PVAC+ SPAN-20	34.76% 0	no	++ matt	- smooth but hard leaf (does not fold)	- heterogeneous (dry oil droplets?)	- leaf really hard (rigid)
T5	PVAC+SPAN-20 + carnauba wax	31.82% 0	no	++ matt	- really rough (white punt)	- heterogeneous (white powder)	-
T6	Sunflower oil (50ml) + carnauba wax	14.11% ++	no	++ matt	- smooth	+ homogenous	+ coating not dry -
T7	PEVACMA	15.90% +	yes, 3/4 of the leaf is brown	--- little shine	+ smooth	+ homogenous	+ leaf completely flaccid
T8	PEVACMA + sunflower oil	22.06% +	yes, 1/4 to 1/2 of the leaf is brown	- little shine	+ smooth	+ homogenous	+
T9	PEVACMA + carnaubawax	16.30% +	yes, 5-10 % of the leaf is brown	- little shine	+ smooth	+ homogenous	+

The (sunflower oil+carnauba wax) coating has good anti-transpirant properties, but it is not shiny and formulation has to be improved.



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# Summarised result of Experiment 2

		Weight loss day 7	Phytotoxicity	Gloss	texture of the coating	homogeneity of the coating	other comments
T0	Water	52.65% -	no	++ little bit	+ smooth	+ homogenous	+
T1	Leafshine	20.24% ++	no	++ lot	++ smooth	+ homogenous	+
T2	Chitosan	50.33% -	no	++ mat	- scaly	- peeling spots	-
T3	Chitosan + sunflower oil	28.95% -	no	++ light shine	+ smooth	+ homogenous	+
T4	PVAC + sunflower oil	30.69% -	no	++ whitish veins	- smooth	+ concentration at veins?	-
T5	Sunflower oil (10ml)+ carnauba wax	38.85% -	no	++ mat	- some white crystals	- crystals	-
T6	Sunflower oil + beeswax	10.44% +++	no	++ mat	- somewhat coarse	- white powdery	-

The (sunflower oil+beeswax) coating has good anti-transpirant properties, but it is not shiny and formulation has to be improved. The (sunflower oil+carnauba wax) needs further optimisation. Including a wax into the coating is very challenging due to its crystallisation.



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## Summarised result of Experiment 3

		Weight loss day 7		Phytotoxicity		Gloss		texture of the coating		homogeneity of the coating	
T0	Water	54.58%	--	no	++	little bit	+	smooth	+	homogenous	+
T1	Leafshine	22.18%	++	no	++	lot	++	smooth	+	homogenous	+
T2	Exsiccator fat	17.67%	++	no	++	mat	-	waxy	-	white crystals	--
T3	PEVAC	33.32%	-	no	++	mat	-	smooth	+	white spots of drying	-
T4	PEVAC + Sunflower oil	19.25%	++	no	++	mat	-	smooth	+	few white crystals	-
T5	PEVAC+ Sunflower oil + beeswax (0.5g)	25.67%	-	no	++	mat	-	smooth	+	some white chrystals	-
T6	PEVAC + Sunflower oil + beeswax (0.2g)	34.51%	-	no	++	mat	-	smooth	+	few white crystals	-
T7	low PEVAC + sunflower oil	36.43%	-	no	++	extra	++	smooth	+	few white crystals	-
T8	PVOH + beeswax	31.43%	-	no	++	extra	++	smooth	+	thicker leaf	+
T9	Shellac + beeswax	33.01%	-	no	++	mat	-	smooth	+	white powdery	-

The coating based on exciccator fat and (PEVAC+sunflower oil) has comparable anti-transpirant properties to Leafshine, but it is not shiny and its formulation has to be improved.

## Summarised result of Experiment 4

		Weight loss day 7		Phytotoxicity		Gloss		texture of the coating		homogeneity of the coating		extra comments
T0	Water	46.62%	--	no	++	little bit	+	smooth	+	homogenous	+	
T1	Leafshine	23.76%	+	no	++	lot	++	smooth	+	homogenous	+	
T2	Chitosane	33.42%	-	no	++	little bit	+	smooth	-	homogenous	+	little sticky
T3	Exsiccatorfat	6.59%	+++	no	++	mat, waxy	-	smooth	+	homogenous	+	
T4	PEVAC + paraffin oil A + L30648	14.16%	++	no	++	extra	+++	smooth	+	homogenous	+	
T5	PEVAC + castor oil	12.94%	++	no	++	little bit	+	smooth	+	homogenous	+	
T6	PEVAC + castor oil + L30648	14.48%	++	no	++	little bit	+	smooth	+	homogenous	+	bit sticky
T7	PEVAC + paraffin oil A	7.17%	+++	no	++	extra	+++	smooth	+	homogenous	+	
T8	Commercial wood glue	45.13%	--	no	++	lot	++	smooth	+	homogenous	+	
T9	Leafshine + sunflower oil	21.02%	+	no	++	lot	++	smooth	+	homogenous	+	

The coating (excicatorfat+L30648) and (PEVAC+castor oil) has superior anti-transpirant properties compared to Leafshine, but it is not as shiny, while the (PEVAC+paraffin oil A) coating besides having a significantly better anti-transpirant property it is even shinier than Leafshine.

# Summarised result of Experiment 5

		Weight loss day 7	Phytotoxicity		Gloss	texture of the coating		homogeneity of the coating			
T0	Water	52.43%	--	no	++	little bit	+	smooth	+	homogenous	+
T1	Leafshine	29.35%	+	no	++	lot	++	smooth	+	homogenous	+
T2	PVOH + Paraffin oil A	21.22%	++	no	++	little bit	+	smooth	+	homogenous	+
T3	PEVAC (15 g) + Paraffin oil A (15 g)	13.81%	+++	no	++	extra	+++	smooth	+	homogenous	+
T4	PEVAC + Paraffin oil B	29.90%	+	no	++	extra	+++	smooth	+	homogenous	+
T5	PEVAC + Paraffin oil C	14.62%	+++	no	++	extra	+++	smooth	+	homogenous	+
T6	Paraffin oil A + Paraffine wax	25.71%	+	no	++	matt	-	some little "sandy" spots	-	very few little crystals	-
T7	Paraffin oil A + Octadecanamide	12.64%	+++	no	++	matt	-	bit "dusty" appearance at spots	-	very few little crystals	-

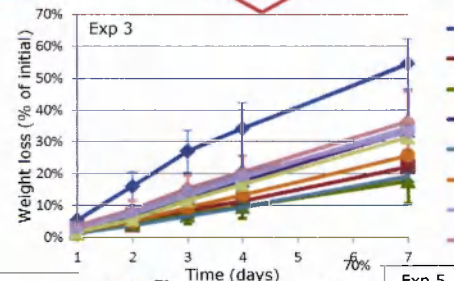
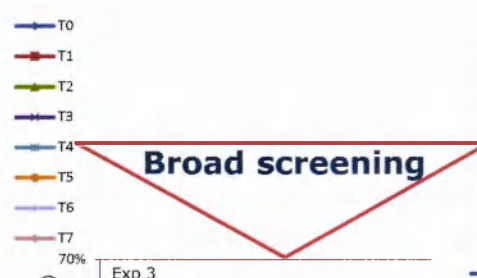
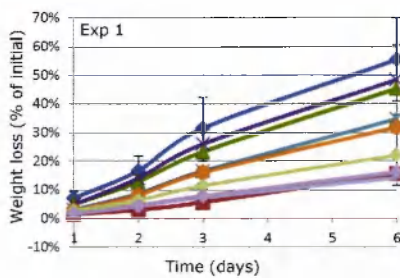
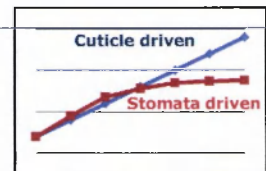
All tested coatings have comparable or superior anti-transpirant properties compared to Leafshine, while having different shines. The coating (paraffin oil A+paraffin wax) and (paraffin oil A+octadecanamide) are not shiny and need further optimisation of the formula.



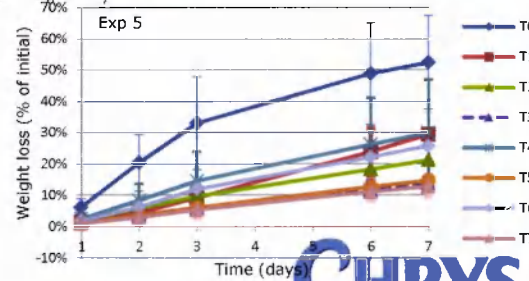
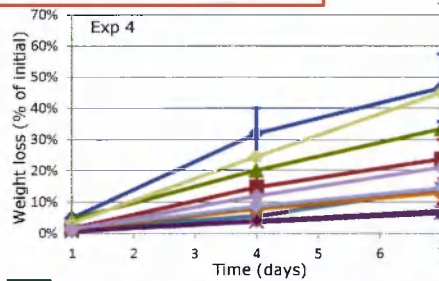
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## Summary of results of Phase 3



**Focused screening: oils**



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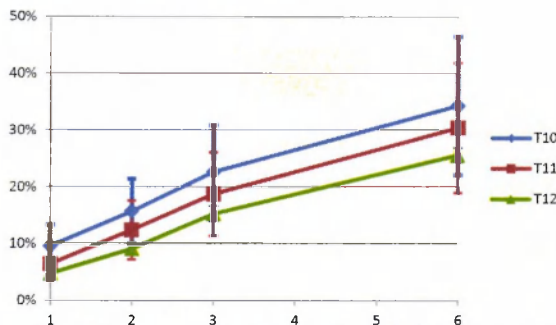


# Phase 2+3: Screening-Model system for Hydrangea

- Raw material:

- Hydrangea macrophylla (Plantion)
- Tubes: 35 cm<sup>3</sup> flower tubes with 30 cm<sup>3</sup> "Chrysal Professional 2"
- Repetition: 5 leaves, (stem length!)

- Handling: like Spathiphyllum



T10	no coating
T11	Leafshine
T12	PEVAC+paraffin oil A



- Conclusion: not suitable to be tested in this system (stem dies fast)



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## Preliminary test on potplants (scale+results)

- Best coating of Exp 5 (PVAC+paraffin oil A) tested
- Spathiphyllum
  - shinier leaves
  - longer shelf life
- Hydrangea
  - shinier leaves
- Gerbera
  - phytotoxic on the flower



Equal soil hydration level is crucial!



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# Conclusions of Stage 1

## ■ Best coatings of Stage 1:

	Coating	Shine	Anti-transpirant	Experiment
1	PEVAC +paraffin oil A	High	Significantly better than Leafshine	Exp 4+5
2	PEVAC +paraffin oil C	High		Exp 5
3	PEVAC+ castor oil	Normal		Exp 4
4	Exsiccator fat	Matt		Exp 3+4
5	Paraffin oil A + Octadecanamide	Matt		Exp 5
6	PVOH + Paraffin oil A	Normal	Better than Leafshine	Exp 5



## ■ Problem:

- Coating 1-5 is solvent-based – not acceptable in practice
- Coating 6 is water-based but not stable
- Additional demands formulated by Chrysal

## STAGE 2

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# New input from Chrysal

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1. Demands on composition: **water based**, environmentally and user friendly, **REACH approved** chemicals! **Not or hardly smelling!**
2. Use compounds provided by Chrysal for further tests!
3. Production process: **concentrate!**
4. Cheap: raw material cost **less than 4 €/l** and easy to produce
5. Inclusion of Reinold's expertise to define tests
6. Nice to have: fast drying, homogenous concentrate and RTU

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## Test set-up for the second stage

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1. Finding the best **emulsifying agent**
  - what is the best emulsifying agent for paraffin oil in water?
  - which emulsifying agent gives the most stable emulsion?
  - what is the effect of the emulsifying agent on the anti-transpiration effect? Which one has the least negative effect?
2. Variation of **oils** in the emulsion system
3. Plant tests (insufficient budget available)

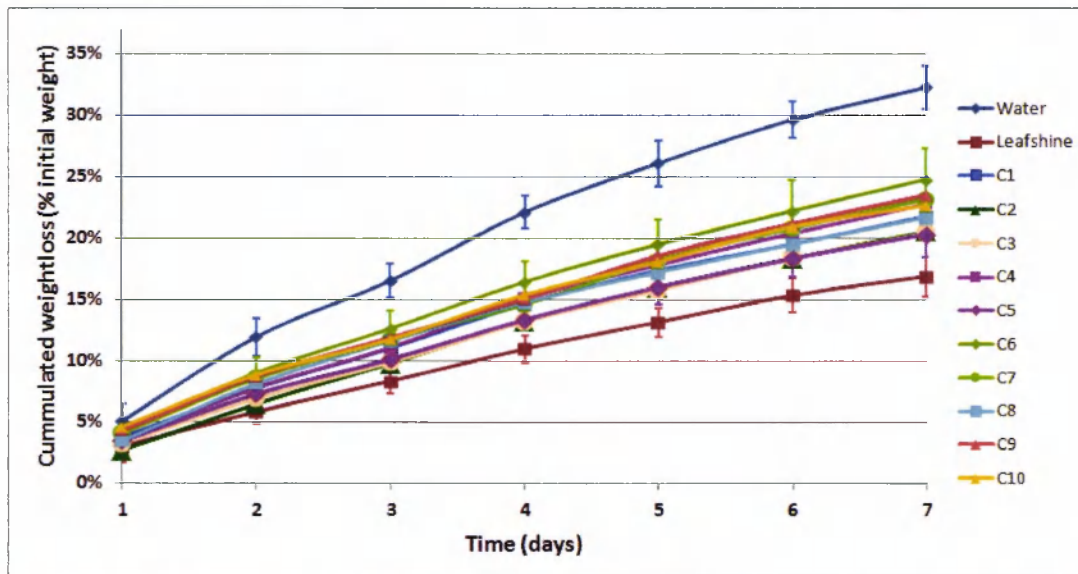


# Summary of results – selecting emulsifier

Coating	Paraffin oil (ml)	Water (g)	Paraffin wax (g)	Surfactant (g)							
				SDS	Span-85	Span-20	Tween-20	L30647	Brij35P	BrijS10	G0806
C1	25	5	0.33	3							
C2	25	5	0.33		3						
C3	25	5	0.33			3					
C4	25	5	0.33				2	1			
C5	25	5	0.33						3		
C6	25	5	0.33				1	1	1		
C7	25	5	0.33	0.5					2.5		
C8	25	5	0.33					3			
C9	25	5	0.33					0.5	2.5		
C10	25	5	0.33								3

Code	Stability concentrate	Stability dilution	Surface	Shine	Weight loss after 7 days
Water	n.a.	n.a.	Normal	Normal shine	32.23%
Leafshine	Yes	Yes	Homogenous spread=0	Extra shine=++	16.92%
C1	Yes	Fair	0	Little extra shine=+	21.78%
C2	No	No	0	+++	20.55%
C3	No	No	0	+++	20.48%
C4	No	No	Looks little sticky=0	+++	22.78%
C5	Yes	Yes	0	+++	20.30%
C6	Yes	No	0	+++ +++	24.76%
C7	Yes	Yes	Network pattern= -	++	23.16%
C8	No	No	0	+++	21.68%
C9	Yes	No	0	++	23.47%
C10	Yes	Fair	-	++	22.62%

# Summary of results – selecting emulsifier



Brij35P (emulsifier in C5) has formed the best emulsion with the paraffin oil and the water-retention almost equal to Leafshine.

Assumption for next test: the anti-transpirant property of new water-based coatings could be improved by increasing the oil:emulsifier ratio

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## Interpretation of result - emulsifiers

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- The **polarity of the surfactant** had limited effect on the water transmission
- **Tween-20** is the most polar surfactant and did not work
- The best surfactant **Brij35P** is fairly polar
- **G0806**, the surfactant of Leafshine (used in C10) did not give the best results here
- Does this mean that:
  - the **interaction between the surfactant and the oil** is very specific?
  - Or that the ratio **oil : surfactant** is wrong here?
  - Or that the oil is more important than the surfactant?

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## Interpretation of result - emulsifiers

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### Ratio Oil : Surfactant

- Composition of C1-10 were: 75% oil, 15% water, 5-9% surfactant and 1-5% thickener
- Leafshine composition (later informed): 70% silicone oil, 20% water and 10% surfactant (G0806)

### Oil

- Oils impact the water transmission: the fewer hydrophilic groups the better and hence the (paraffin oil + wax) system was chosen for further tests
- Vegetable oils performed less good in tests of Stage 1, silicone oil performs better

# Test setup – selecting the best oil

- General composition concentrate: 70% oil, 20% water, 9% surfactant and 1% thickener.
- Question 1: How much Brij35P do we really need?
- Question 2: Which oil is the best?
- Question 3: Do we need a thickener?



## Coatings to select best oil and oil:surfactant

	Oils						Wax	Water	Emulgator	
	Paraffin	Silicon	Sunflower	Rapeseed	Soya	Palmfat			Brij 35P	G0806
C1	25.00	0.00	0.00	0.00	0.00	0.00	0.33	5.00	3.00	0.00
C2	23.33	0.00	0.00	0.00	0.00	0.00	0.33	6.67	3.00	0.00
C3	23.33	0.00	0.00	0.00	0.00	0.00	0.00	7.00	3.00	0.00
C4	23.33	0.00	0.00	0.00	0.00	0.00	0.17	8.17	1.67	0.00
C5	23.33	0.00	0.00	0.00	0.00	0.00	0.07	9.27	0.67	0.00
C6	23.33	0.00	0.00	0.00	0.00	0.00	0.00	9.33	0.67	0.00
C7	23.33	0.00	0.00	0.00	0.00	0.00	0.00	6.67	0.00	3.33
C8	0.00	23.33	0.00	0.00	0.00	0.00	0.00	6.67	0.00	3.33
C9	0.00	23.33	0.00	0.00	0.00	0.00	0.00	7.00	3.00	0.00
C10	0.00	23.33	0.00	0.00	0.00	0.00	0.07	9.27	0.67	0.00
C11	0.00	23.33	0.00	0.00	0.00	0.00	0.00	9.33	0.67	0.00
C12	0.00	0.00	23.33	0.00	0.00	0.00	0.00	9.33	0.67	0.00
C13	0.00	0.00	23.33	0.00	0.00	0.00	0.07	9.27	0.67	0.00
C14	0.00	0.00	0.00	23.33	0.00	0.00	0.07	9.27	0.67	0.00
C15	0.00	0.00	0.00	0.00	23.33	0.00	0.07	9.27	0.67	0.00
C16	0.00	0.00	0.00	0.00	0.00	23.33	0.07	9.27	0.67	0.00
C17	0.00	0.00	0.00	0.00	0.00	23.33	0.07	6.60	0.00	3.33

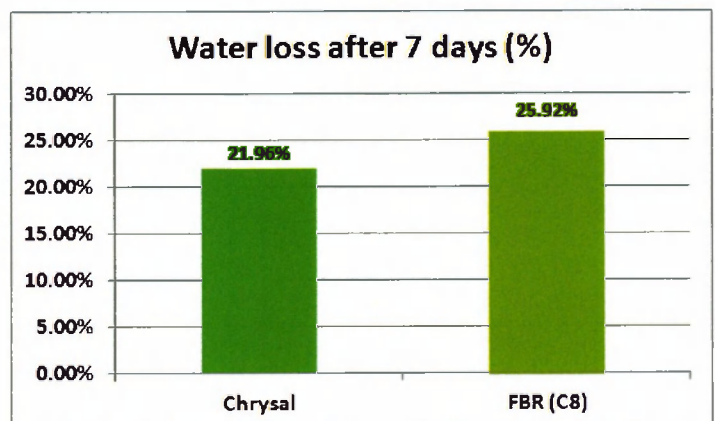


# Results – oil and optimisation oil:surfactant

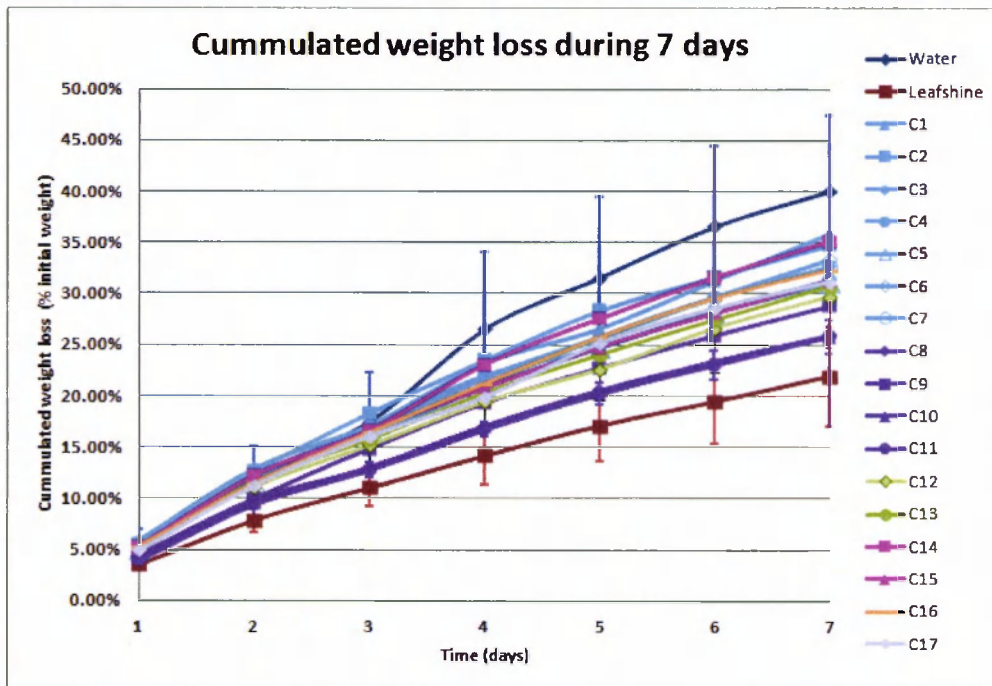
Code	Consistency		Surface	Shine	Weight loss after 7 days	Remarks
	Flows when upside down	Drops of from a stick				
Water	n.a.	n.a.	Normal	Normal	40.06%	
Leafshine	Yes	Yes	Homogenous spread=0	Extra shine=+++	21.96%	
C1	No	No	0 little spotted	Some extra shine=++	35.94%	Looks like thick wood-glue
C2	No	No	0-	++	34.79%	Looks like thick wood-glue
C3	No	No	0-	++	32.65%	Looks like wood-glue
C4	Yes	No	Looks sticky= -	+	31.35%	
C5	Yes	Somewhat	-	+	31.08%	
C6	Yes		-Oily, little drops	+	33.41%	
C7	Yes		-Oily, little drops	+	32.65%	
C8	Yes	Yes	0	+++	25.92%	
C9	No	No	0	+++	28.81%	
C10	Yes	Yes	0	+++	25.80%	
C11	Yes	Yes	0	+++	25.85%	
C12	Yes	Yes	0-Little oily	+	29.74%	Bit yellowish
C13	Yes	Yes	-oily	0	30.75%	Bit yellowish
C14	Yes	Yes	0-very little oily	++	35.17%	
C15	Yes	Yes	-	+	31.33%	
C16	Yes	Yes	0-	++	32.29%	yellow
C17	Yes	Yes	0- bit waxy	0	31.20%	yellow

## Leafshine

- Leafshine and C8 should be identical, but are not!
- Possible differences:
  - Composition?
  - Production process



# Summary of results – selecting oil



Silicon oil based coatings functioned the best



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## Interpretation of result - oils

- **Silicon oil based coatings** functioned the best
- Coatings based on the other oils failed to form a very **homogenous layer** on the leaf surface and most likely therefore performed worse as anti-transpirant
- Sunflower oil was the most effective plant oil as far as water retention is concerned
- Brij35P is a good emulsifier for silicon oil, but smaller amounts are needed than of G0806



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# Answers to posed questions

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- Question 1: How much Brij do we really need?
  - The smallest amount of Brij35P tested gave the best results with silicon oil.
- Question 2: which oil is best?
  - Silicon oil performed the best as anti-transpirant
  - Oils are more important than emulsifiers
- Question 3: do we need a thickener?
  - Not fully proven
  - Only a small positive effect (C8 versus C10)

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## Conclusions of Stage 1 and 2

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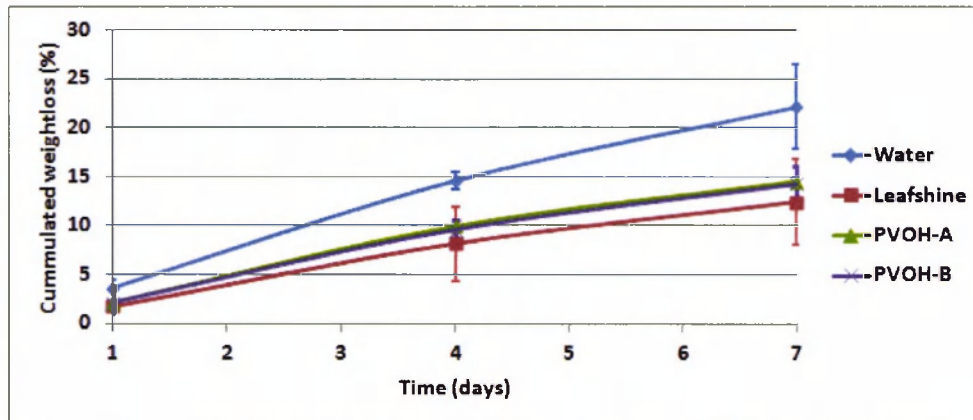
- Stage 1:
  - 5 good solvent-based coatings – not acceptable in practice
  - 1 water-based coating - not stable
- Stage 2:
  - Silicon oil-based coatings had the best anti-transpirant effect
  - Brij35P is well suited as emulsifier for silicon oil
  - The nature of the oil is much more critical than the nature of the emulsifier



## Last test with a water-based coating

- Coating based on PVOH were tested based on initial positive results of Stage 1

Coating	Silicon oil	Water	Brij35P	PVOH
PVOH-A	23.33	9.27	0.67	0.07
PVOH-B	23.33	8.67	0.67	0.67



PVOH-based coatings performed worse when starting from a concentrate, compared to results of Stage 1. As their water retention is comparable to that of Leafshine, by optimising the formula it might perform even better than.

## Final conclusions

- All the work done did not lead to a better water-based coating compare to Leafshine
- 5 better coatings were found but they were solvent-based
- Shine might be used to predict coating efficacy (the higher the bigger anti-transpirant effect)

# Importance of surface



## Future?

- Are there reasons to continue?
- Student at the university coached by Ernst Woltering and Erika is working on a quick test to judge anti-transpirant effect based on conductivity measurements
- Finalising current project: this presentation as a report + recipe of tested coatings already sent