

# Crystallisation and Physical Properties of Fats

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Ghent University, Belgium



## Improving the Physical and Chemical Properties of Cocoa Butter by Selected Oil Processing Technologies

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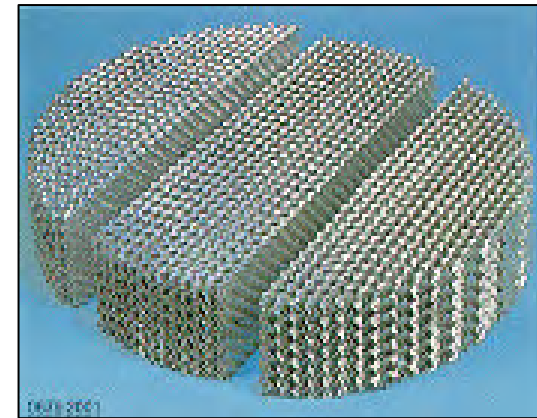
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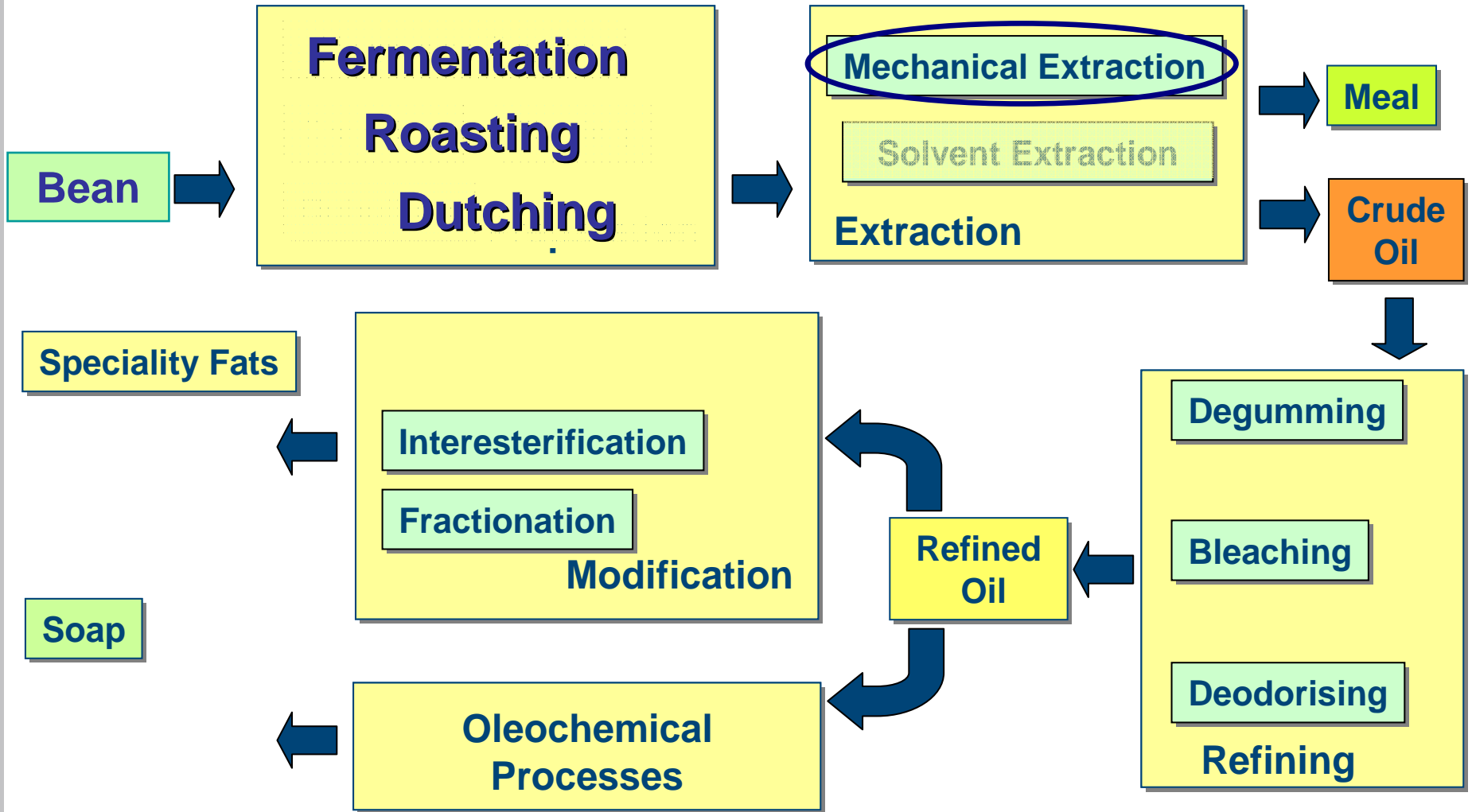
# Outline of the Presentation



- Introduction: Cocoa Butter Processing
- Steam Refining
  - Chemical impact
  - Crystallization “in process”
  - Crystallization “of product”
- (Static Fractionation)
  - Principle
  - Process
  - Product
- Conclusions



# Cocoa Butter Processing



# Oil Quality



## OIL PROCESSING

### Organoleptic/stability

- Desired taste, odour
- Light color (brilliant)
- High thermal stability
- High oxidative stability
- Long shelf life

### Refining

### Functional Properties

- Good melting profile
- Desired Plasticity
- Crystallisation kinetics

### Modification

OIL  
QUALITY

### Nutritional Quality

- Balanced FA composition
- No *trans* FA
- High tocopherol content,...



# Steam Refining: Packed Column Principle

# Steam Refining



## Purpose:

1. Removal of unwanted odour and taste
2. Removal of excessive FFA (>1.75% as oleic acid)

➔ while preserving TAG-distribution of oil, colour, oxidative stability,...

## Principle:

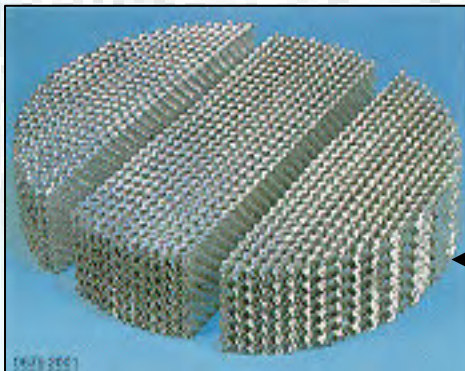
Stripping of volatiles with carrier gas (steam) at high temperature (laws of Raoult & Dalton)

# Steam Refining



## Packed Column:

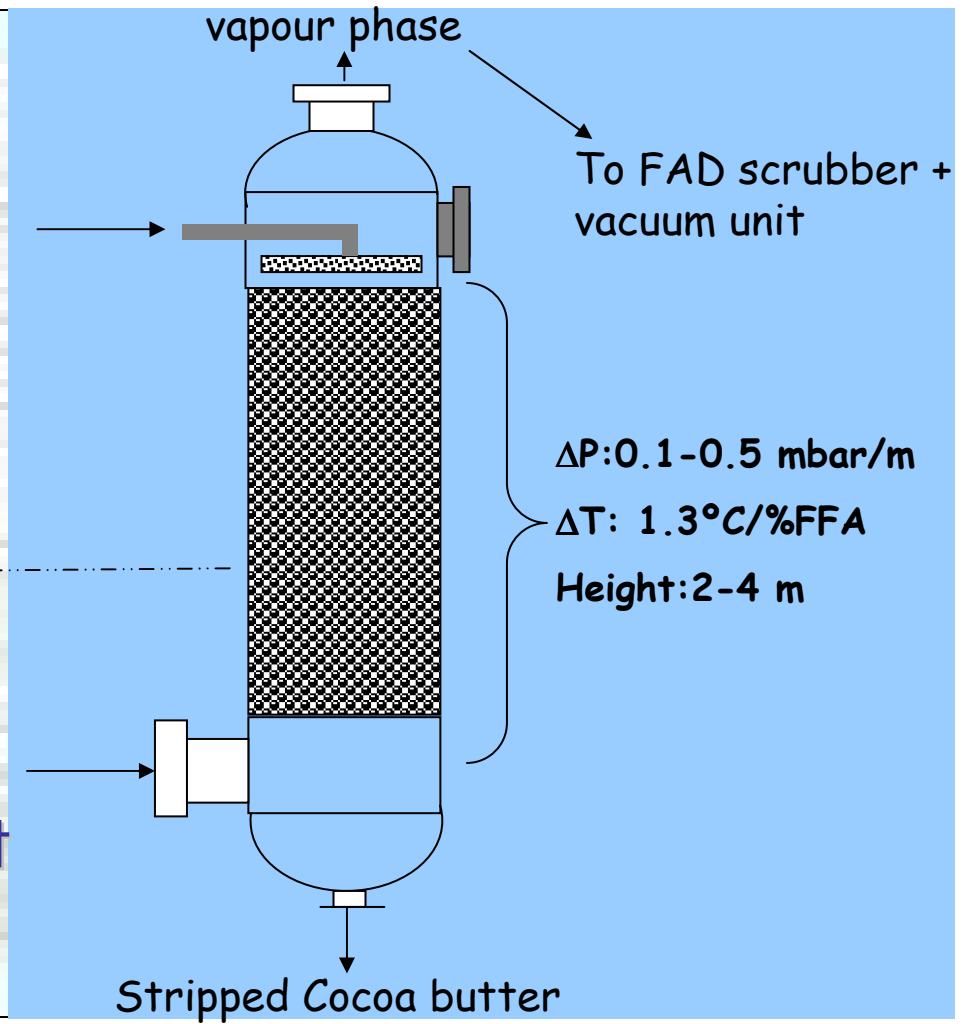
Filtered or pretreated cocoa butter



Structured packing

Stripping Steam

- Continuous countercurrent
- Short residence time





# Steam Refining: Chemical Impact

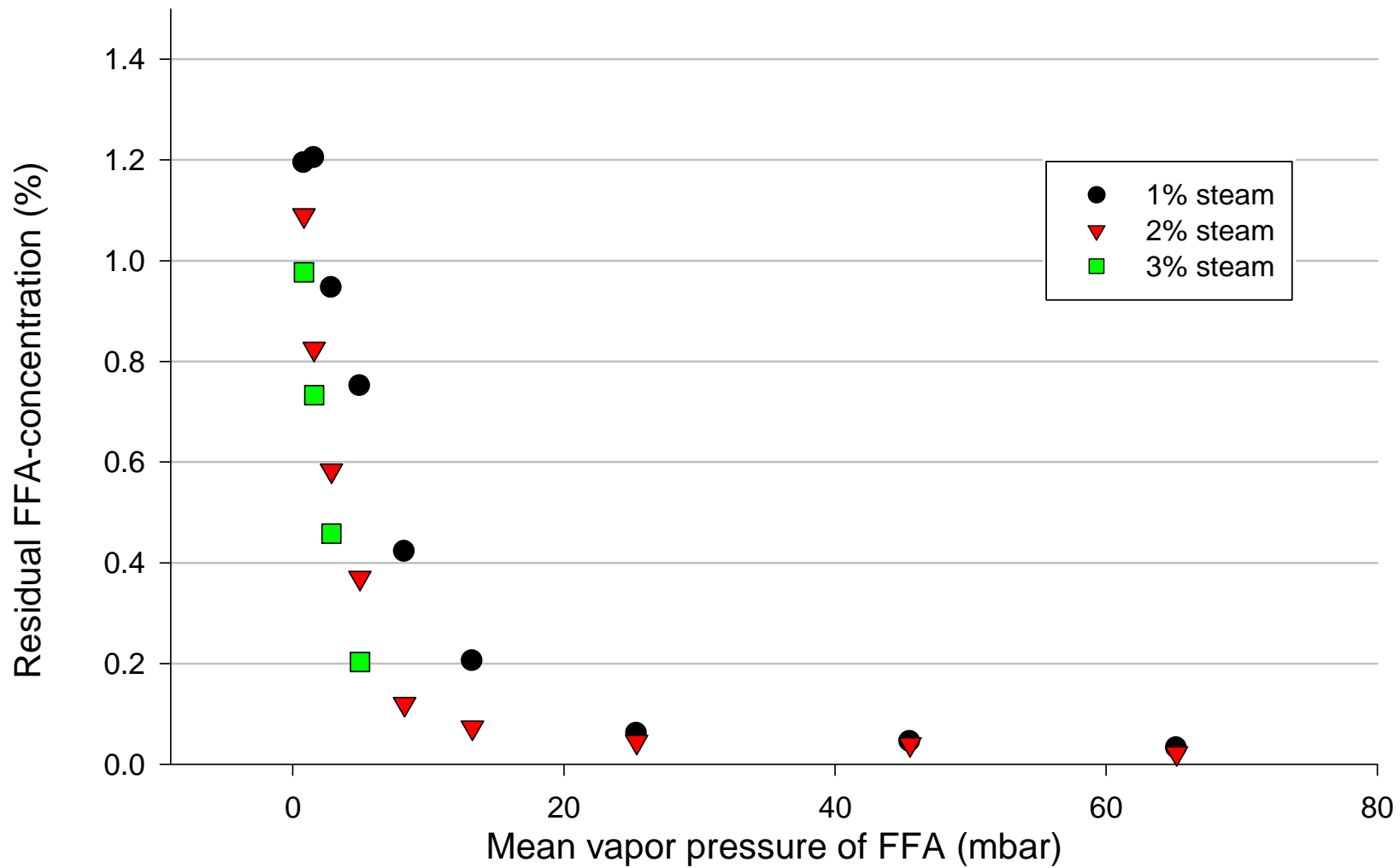


# Steam Refining: Odour & Taste, TAG



- Higher temperature leads to more bland taste
  - ✓ Europe: residual aroma desired (e.g. 180°C)
  - ✓ U.S.: favouring bland taste (e.g. 200°C)
- No observable effect on TAG-distribution
  - ✓ TAG-ratios remain unchanged
  - ✓ No TFA formation
  - ✓ No interesterification effects

# Steam Refining: FFA-reduction



# Steam Refining: FFA-reduction



<i>Dijkstra equation</i>					
Steam (%)	<i>Transfer units u</i>			$R^2$	
1	1.70 ± 0.18			0.974	
2	2.04 ± 0.10			0.994	
3	1.93 ± 0.09			0.995	
<i>Bailey equation</i>					
Steam (%)	A	B	E (%)	$R^2$	
1	1.40 ± 0.06	0.136 ± 0.011	82	0.992	
2	1.33 ± 0.03	0.272 ± 0.014	91	0.992	
3	1.33 ± 0.02	0.393 ± 0.018	90	0.999	
<i>Extended Bailey equation</i>					
Steam (%)	A	B	$y_0$	E (%)	$R^2$
1	1.40 ± 0.05	0.138 ± 0.012	0.03 ± 0.02	83	0.993
2	1.32 ± 0.04	0.288 ± 0.021	0.04 ± 0.02	96	0.996
3	1.32 ± 0.02	0.390 ± 0.014	0.01 ± 0.03	94	0.999

lower model quality

$$V_2 = V_1 \frac{(PO)^u (P_v S - PO)}{(P_v S)^{u+1} - (PO)^{u+1}}$$

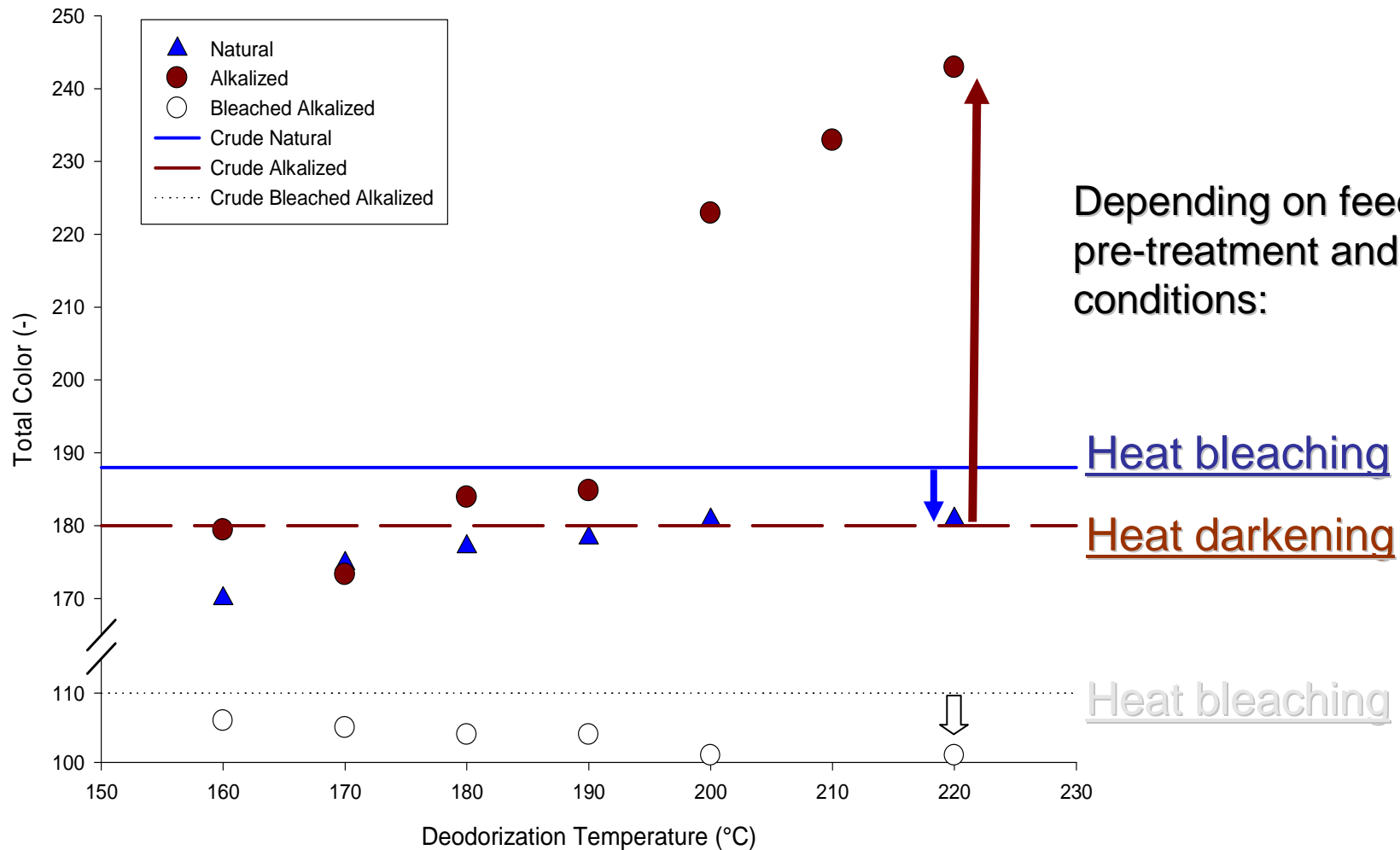
$$S = \frac{P \cdot O}{E \cdot P_v} \cdot \ln \frac{V_1}{V_2}$$

lower model quality

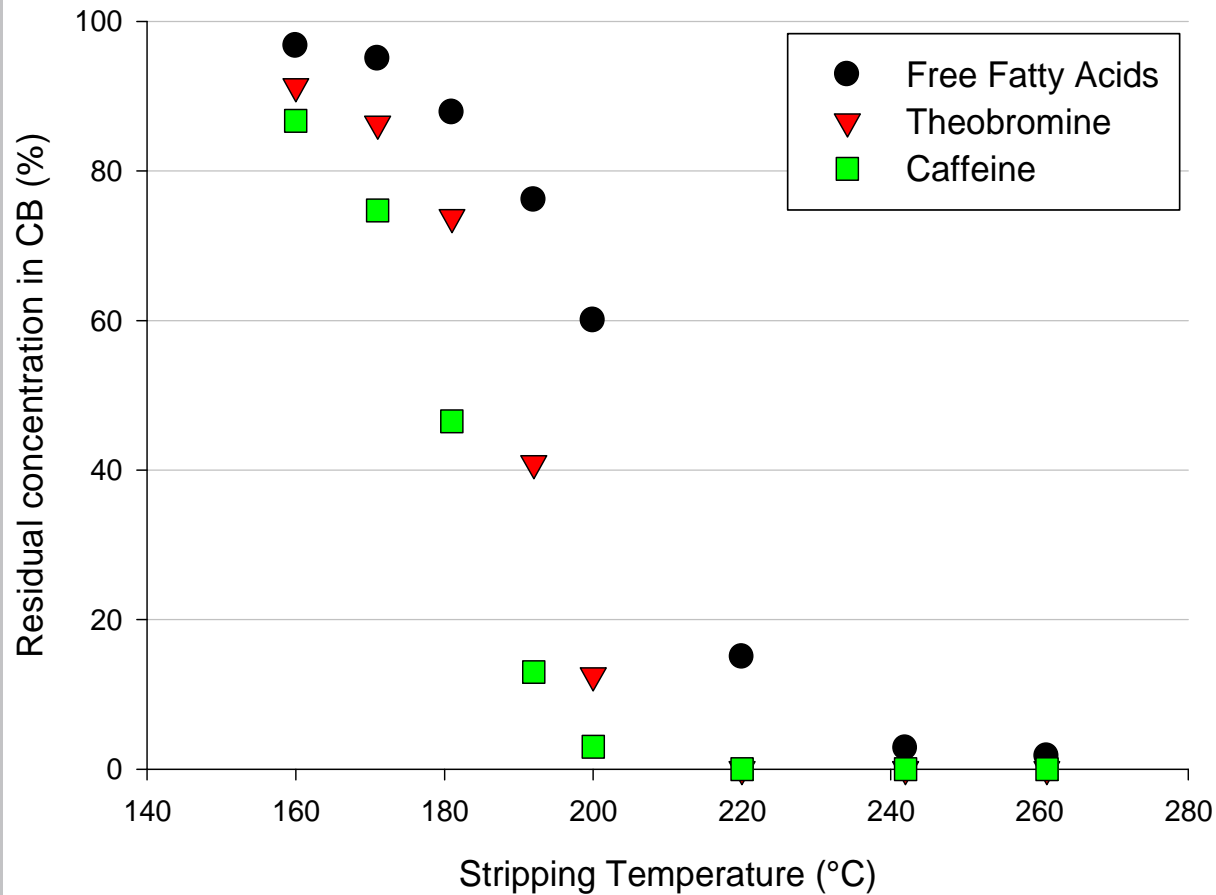
$$V_2 = y_0 + a \exp(-bP_v)$$

FFA-removal can be described by various physical/mathematical models

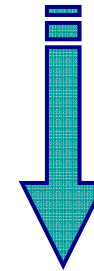
# Steam Refining: Colour of Cocoa Butter



# Steam Refining: Theobromine & Caffeine



At mild temperatures:  
Removal of T & C > FFA

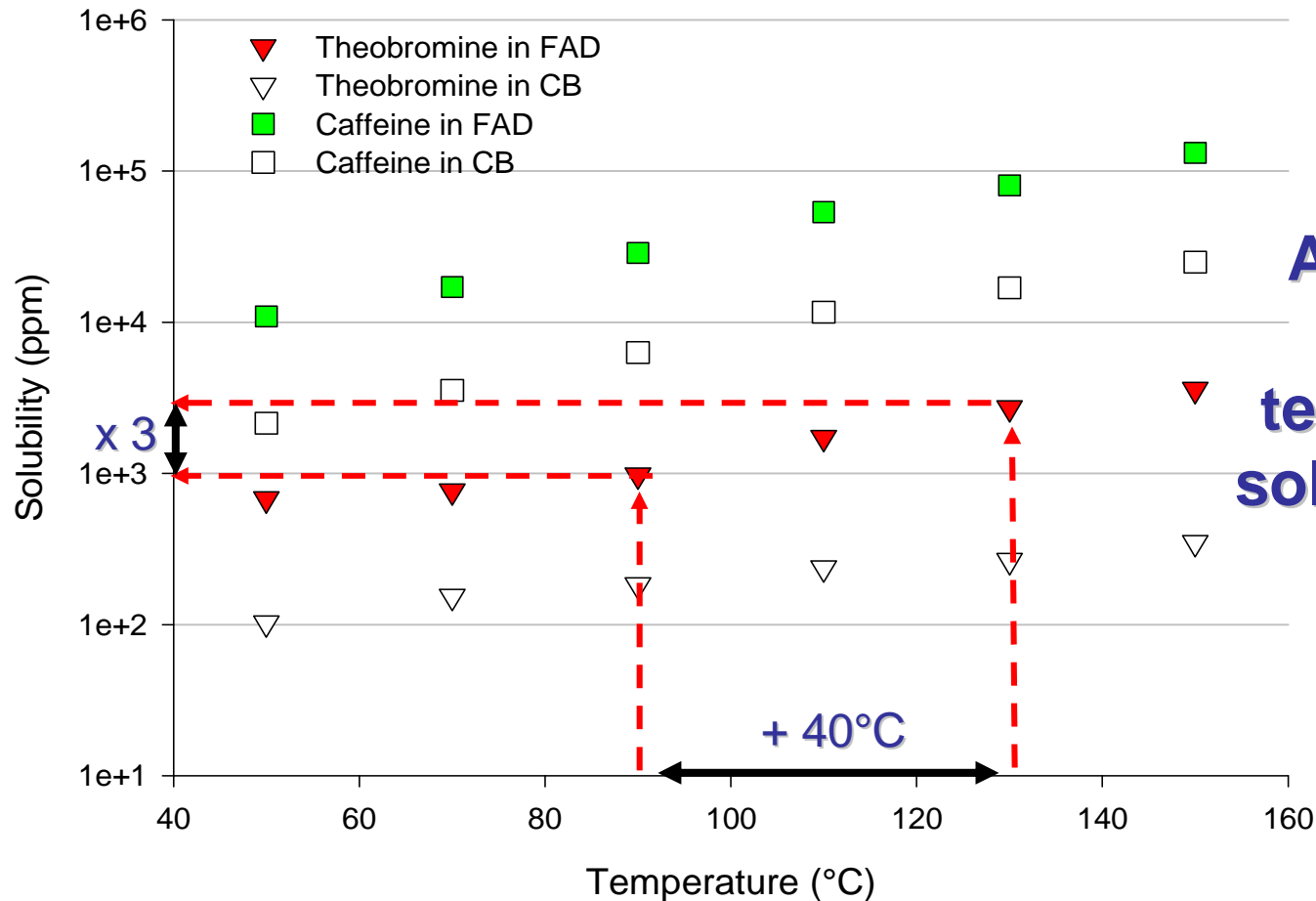


Deposit of T & C in vapour  
condensation system



# Steam Refining: Crystallization 'in process'

# Steam Refining: Theobromine & Caffeine



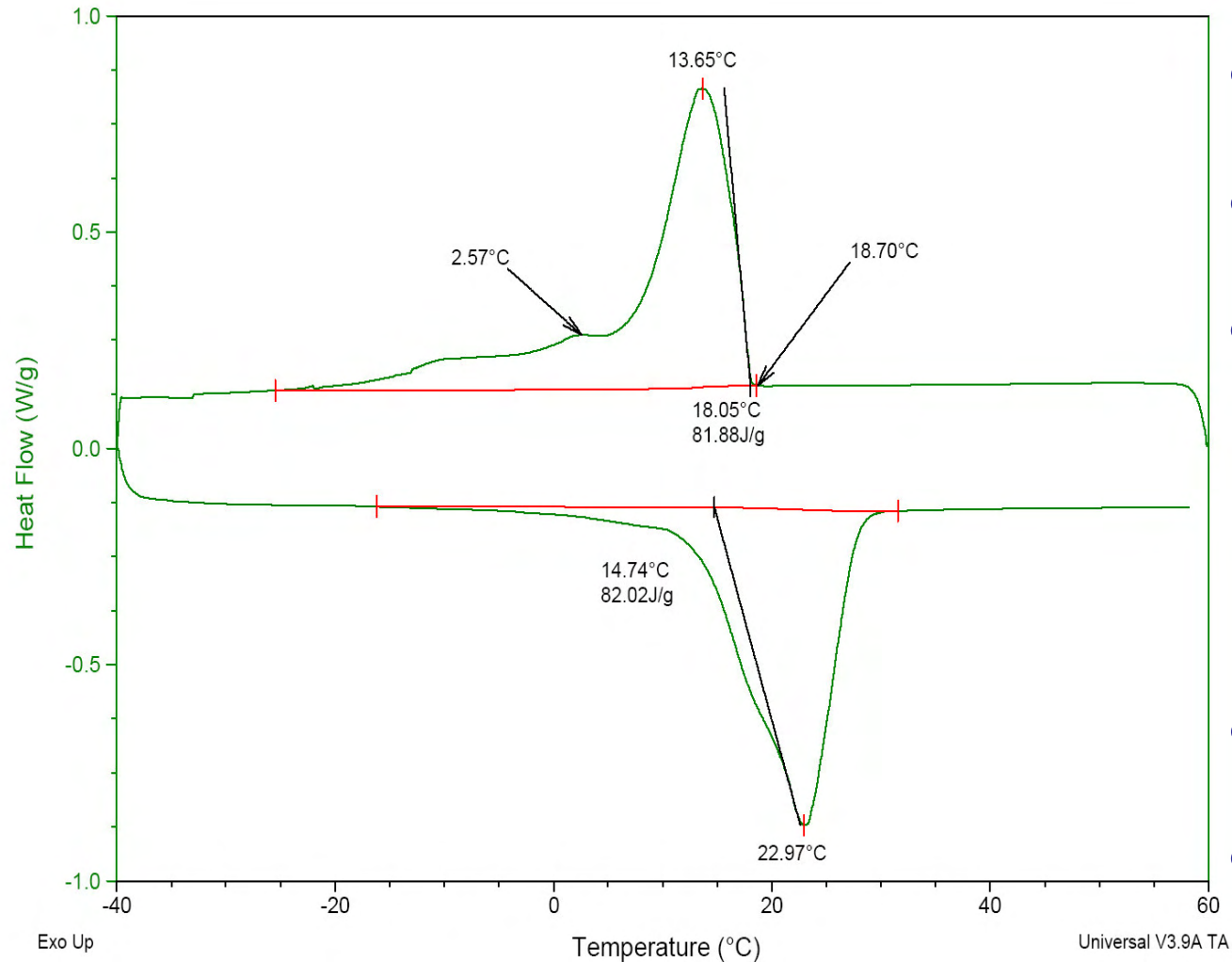
**Adjustment of  
scrubber  
temperatures to  
solve T&C in FAD**



# Steam Refining: Crystallization of Product



# Steam Refined Cocoa Butter: Non-isothermal crystallization



- DSC
- 3 repetitions
- 3 factors
  - Temperature
  - Steam
  - FFA
- 8 parameters
- FMLR

# Steam Refined Cocoa Butter: Non-isothermal crystallization



## • Models show...

		<i>I.C.</i>	<i>Temperature (°C)</i>	<i>Steam (%)</i>	<i>FFA (%)</i>	<i>R<sup>2</sup></i>
<b>Crystallization</b>	Heat (J/g)	74.53	0.0320*	n.s.	n.s.	0.22
	Start temperature (°C)	15.05	0.0235*	n.s.	1.163	0.19
	Onset temperature (°C)	18.07	n.s.	n.s.	-0.043*	0.43
	Peak max 1 (°C)	13.39	n.s.	0.185	0.376*	0.26
	Peak max 2 (°C)	2.09	n.s.	n.s.	n.s.	-
<b>Melting</b>	Heat (J/g)	86.27	n.s.	n.s.	-3.01*	0.31
	Onset temperature (°C)	11.01	0.0097	n.s.	0.982*	0.23
	Peak max (°C)	26.23	-0.0129	-0.022	-1.422*	0.53

• *I.C.:* intercept

• *n.s.:* not significant

• \*most significant variable as indicated by ANOVA

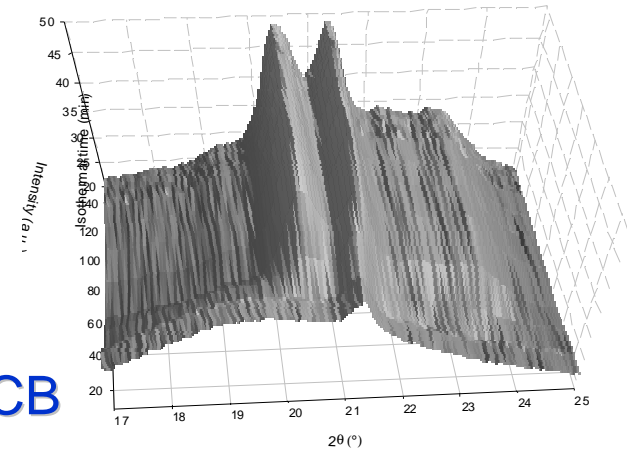
...overall low influence, **FFA** most significant

# Steam Refined Cocoa Butter: Isothermal crystallization at 20°C



- XRD-measurements show:

1. Two-step crystallization:  $\alpha$  to  $\beta'$
2. Earlier formation of  $\alpha$  in strongly refined CB
3. Slightly higher angles in SAXS for  $\alpha$  in strongly refined CB
4. Slightly sooner occurrence of  $\beta'$



- Modeling the  $\beta'$  isothermal crystallization with Foubert-model:

$$f(t) = a_F \left( 1 - \left( 1 + \left( 0.99^{(1-n)} - 1 \right) e^{((n-1)K(t-t_{ind}))} \right) \left( \frac{1}{1-n} \right) \right)$$

# Steam Refined Cocoa Butter: Isothermal crystallization at 20°C



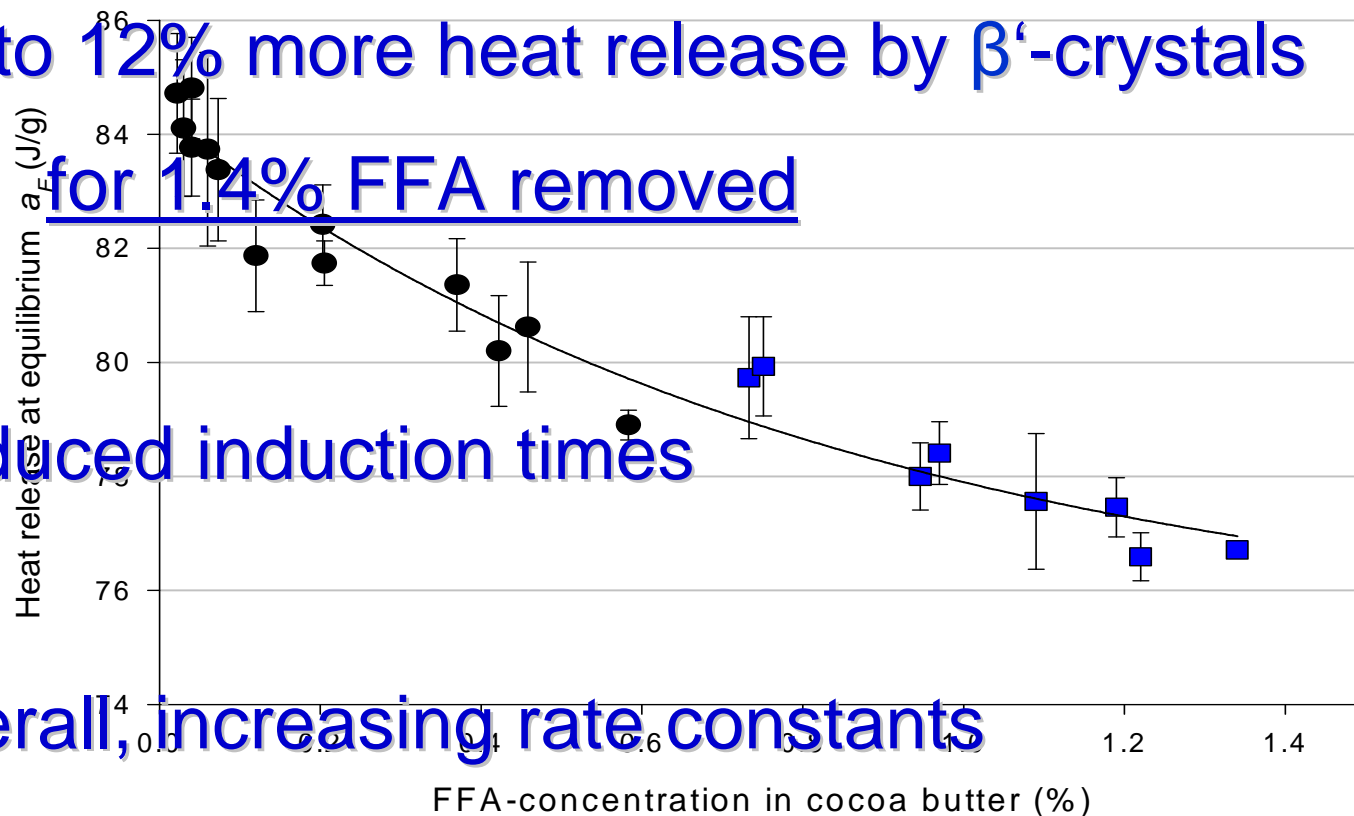
- When plotted as function of FFA, models show...

Up to 12% more heat release by  $\beta'$ -crystals

for 1.4% FFA removed

Reduced induction times

Overall, increasing rate constants



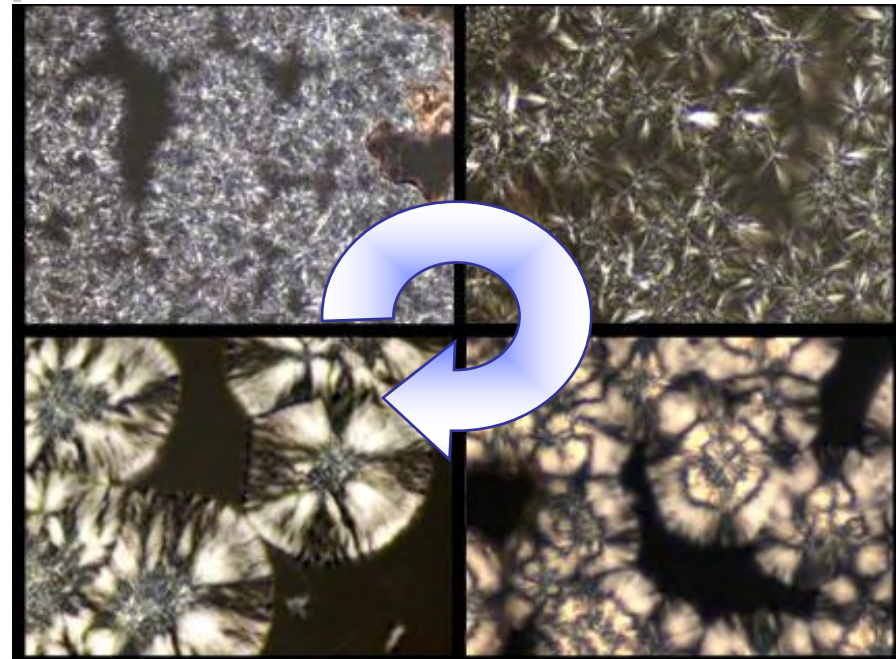
# Steam Refined Cocoa Butter: At Equilibrium



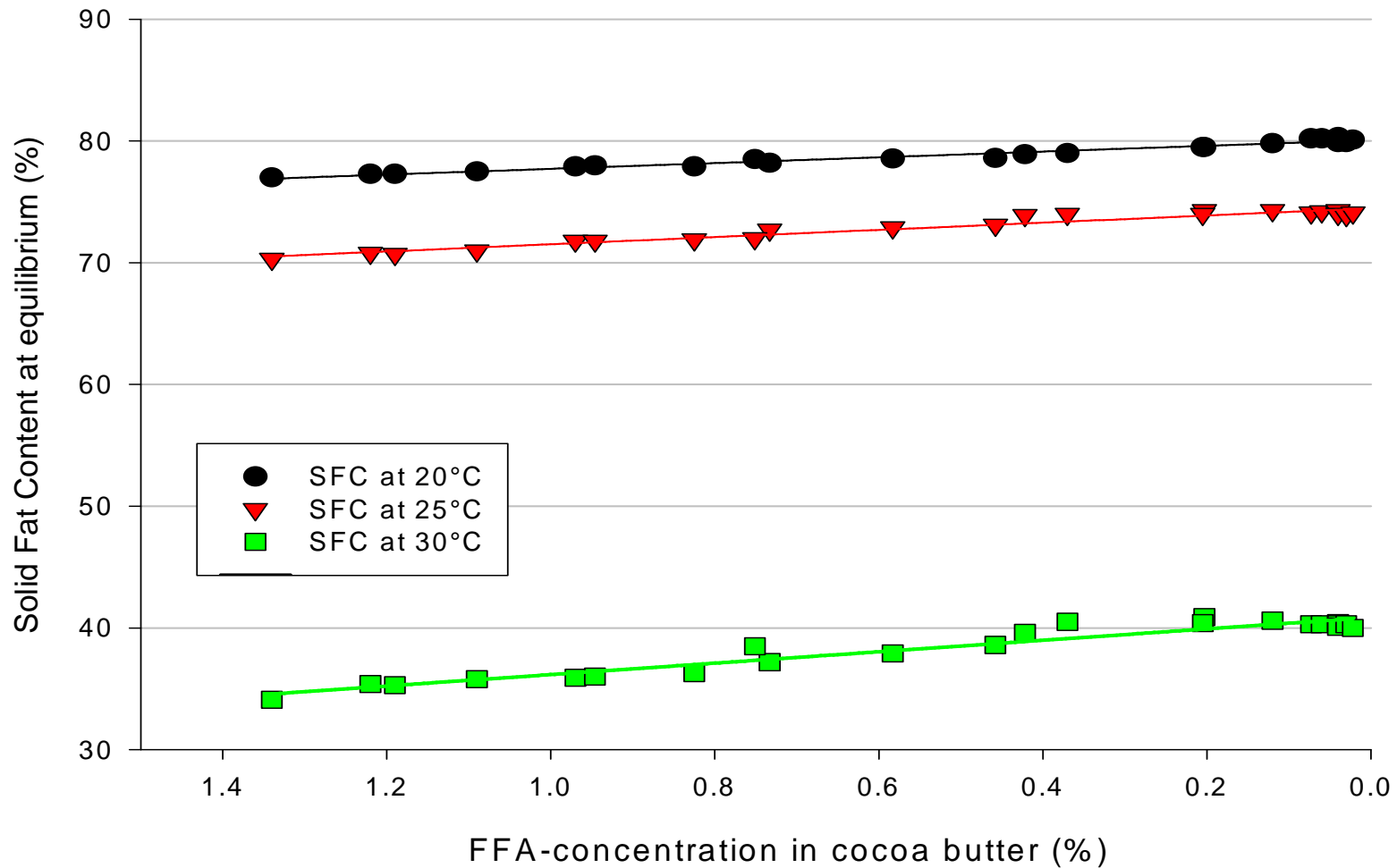
- 'Equilibrium' after tempering (40h at 24-26°C)
- XRD-measurements show overall presence  $\beta^V$
- However, PLM shows:

**high FFA**

**low FFA**




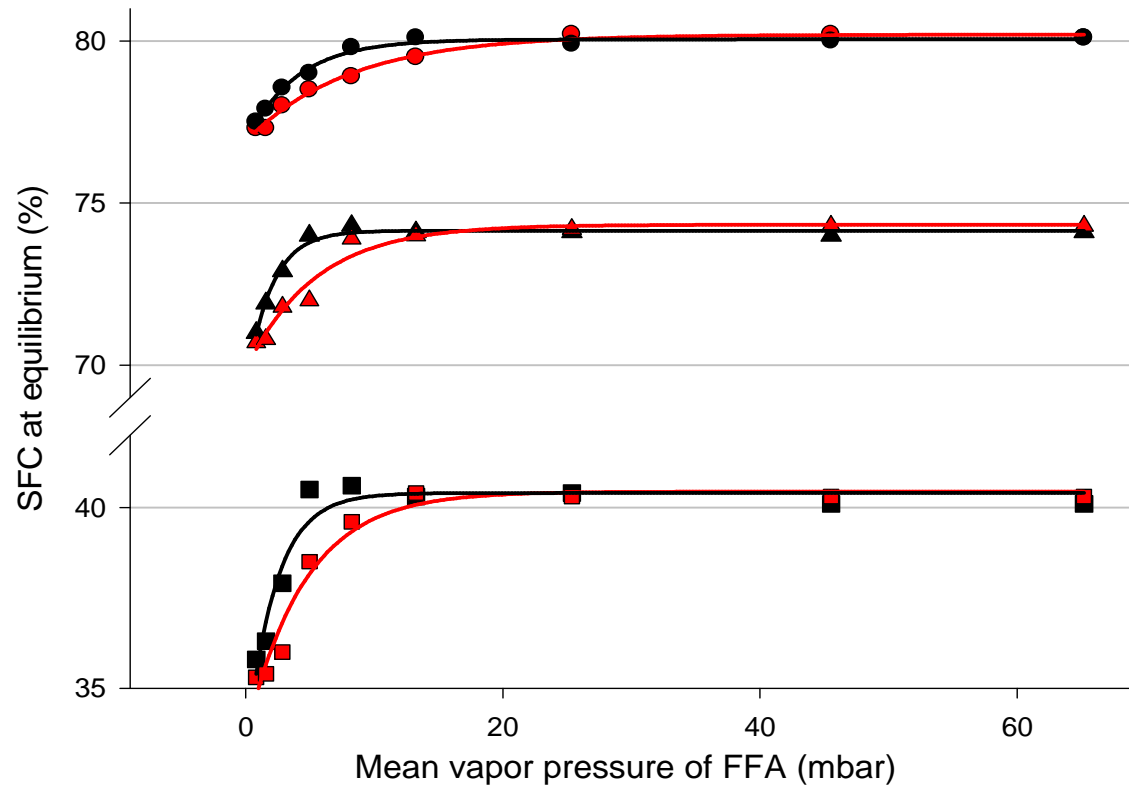
# Steam Refined Cocoa Butter: SFC-values at 20, 25 and 30°C



# Steam Refined Cocoa Butter: Predicting SFC?



Process conditions  SFC





## Conclusions

or

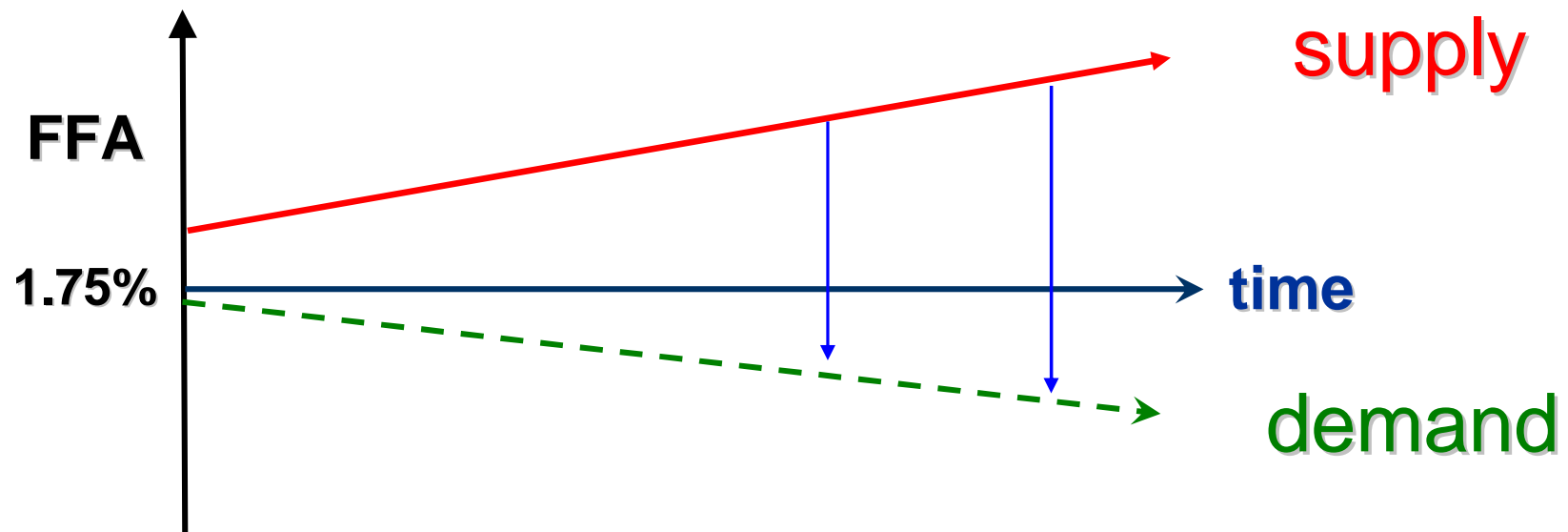
- *Final Remarks*
  - *The Wrap-up*
    - *The Endtro*
- *Take Home Messages*
  - *Food for Thought*



# The Endtro



## Current situation for CB:



- Two-way expanding playing field for steam refining
- Need for efficient FFA-removal in short residence time



## Acknowledgements

**Prof. Dr. Johan Wouters & ing. Bernadette Norberg  
(CBS, FUNDP, Namur) for the use of their XRD-facilities**

## Further reading

•Calliauw, G., Vila Ayala, J., Gibon, V., Foubert, I., Dewettinck, K., Wouters, J. & De Greyt, W. (2008) Models for FFA-removal and phase behaviour changes of cocoa butter by packed column steam refining. *Journal of Food Engineering* (available online)

•Vila Ayala, J., Calliauw, G., Foubert, I., Dewettinck, K., Dyer, B. & De Greyt, W. (2007). Impacts of bleaching and packed column steam refining on cocoa butter properties. *Journal of American Oil Chemists' Society*, 84, 1069-1077.

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