Isothermal crystallization kinetics of cocoa butter

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EXPERIMENTAL SET-UP

Samples: standard factory product cocoa butter and cocoa butter from Nigeria

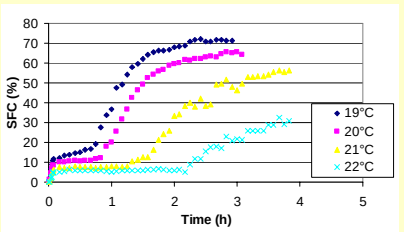
Analytical techniques: DSC: sample preparation, 65°C / 15 min (removal of memory effect), cooling at 8°C / min to crystallization temperature, hold until crystallization is finished

pNMR: sample preparation, 65°C / 1h (removal of memory effect), transfer to cryostat at crystallization temperature, take readings at appropriate intervals

Crystallization temperatures: between 19°C and 23°C (0.5°C interval for DSC experiments, 1°C interval for pNMR experiments)

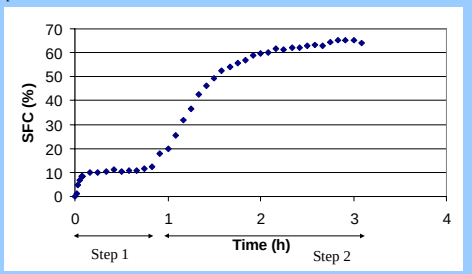
Analyses: step 2 of the DSC curves is integrated and modeled by the Avrami and Gompertz models

INFLUENCE OF TEMPERATURE AS MEASURED BY pNMR

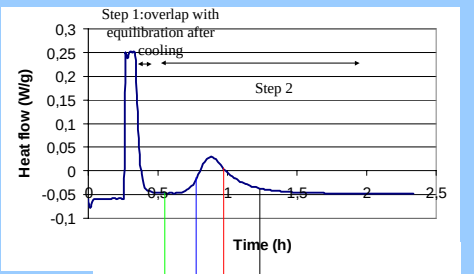


ISOTHERMAL CRYSTALLIZATION IS A TWO STEP PROCESS

pNMR



DSC



THUS

Step 1: melt → α polymorph

Step 2: melt → β' polymorph

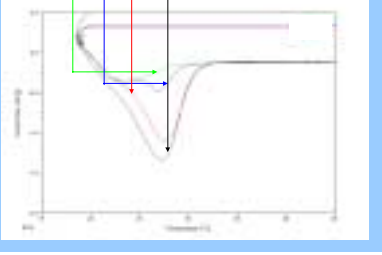
and

α polymorph → β' polymorph

To confirm and elucidate details (mechanism of step 2):

real – time XRD

For more details on two steps: STOP AND RETURN METHOD, i.e after certain isothermal crystallization times: melting profile (heating at 5°C/min) which gives information on the polymorphs formed



CONCLUSIONS

- Isothermal crystallization of cocoa butter is a two-step process, starting with some crystallization in the α polymorph, followed by (re)crystallization in the β' polymorph
- The amount of α crystals formed decreases as temperature increases
- The final SFC value decreases as temperature increases, while the *a* parameter of the second step as measured by DSC only starts to decrease at high temperatures
- The induction time increases as temperature increases, while the maximum increase rate and the Avrami rate constant decrease as temperature increases
- There is no clear dependence of the Avrami exponent on the temperature

Acknowledgements

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INFLUENCE OF TEMPERATURE ON STEP 2 OF THE CRYSTALLIZATION PROCESS AS MEASURED BY DSC

AVRAMI MODEL

$$f(t) = a * \left(1 - e^{-k*t^n}\right)$$

f(t): amount of crystallization at time *t* [J/g]

a: value for *f* as *t* approaches infinity [J/g]

k: crystallization rate constant [h^{-n}]

n: Avrami exponent [-]

GOMPERTZ MODEL

$$f(t) = a * e^{-e^{\frac{\mu * e}{a} * (\lambda - t) + 1}}$$

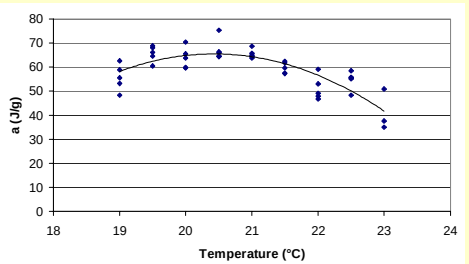
f(t): amount of crystallization at time *t* [J/g]

a: value for *f* as *t* approaches infinity [J/g]

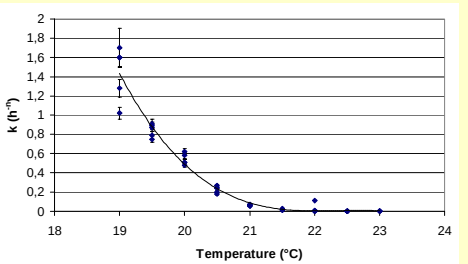
μ: maximum increase rate [J/g h^{-1}]

λ: induction time [h]

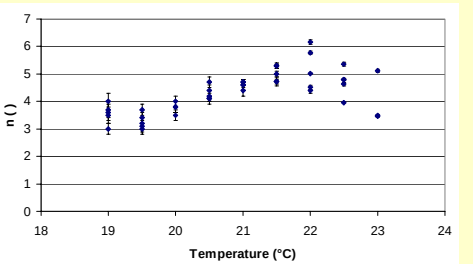
Influence on a



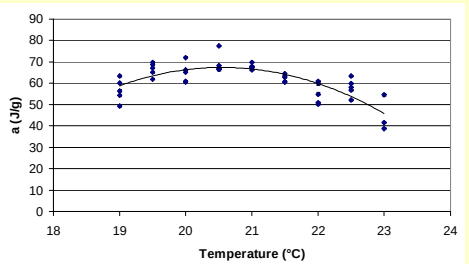
Influence on k



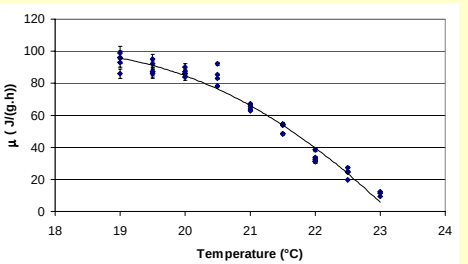
Influence on n



Influence on a



Influence on μ



Influence on λ