

Analysis of the expansion phenomenon during the extrusion process: Experiments and model



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Ludovic Club, March 26, 2014 - Lyon



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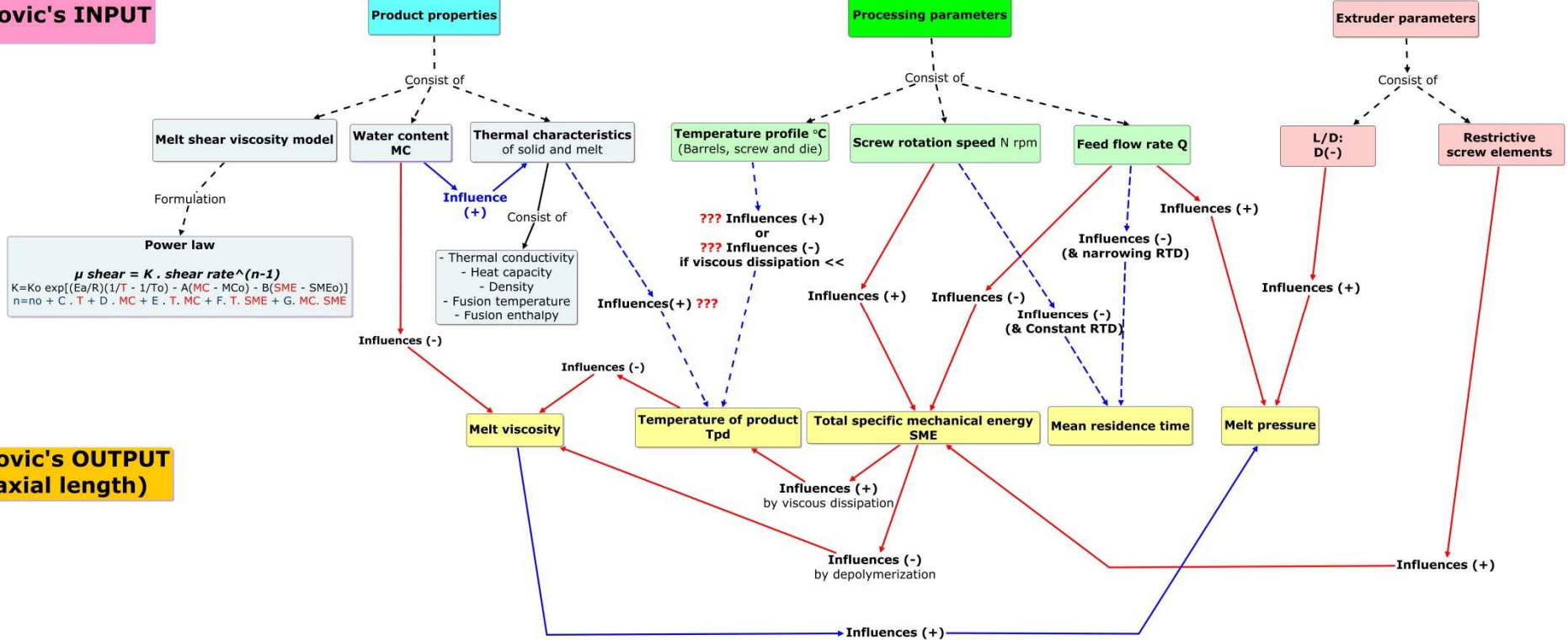
- Objective of this work

- ✓ Test the validity of Ludovic[®] simulation for starch extrusion
- ✓ Show how to integrate the **phenomenological model** of **expansion** with Ludovic[®]'s interface

Aims: Extend the Ludovic[®]'s capability **to predict macro & microstructure of starchy foam**

Concept map: Structure of Ludovic®

Ludovic's INPUT



Ludovic's OUTPUT f(axial length)

**Input variables of models =
Extrusion's output variables at die exit (calculated by Ludovic®)**



EXPERIMENTS



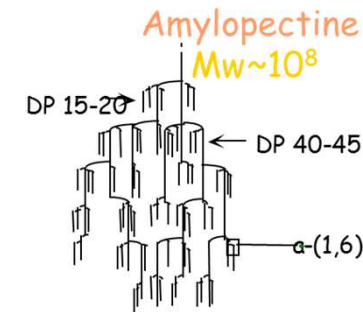
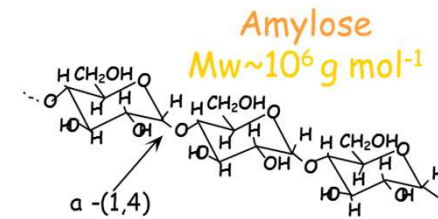
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1) Raw material - Maize starches

Starch	Product	Amylose / Amylopectine
A	Amylomaize	70 / 30
B	Blend of A : D = 2 : 1	47 / 53
C	Blend of A : D = 1 : 2	23.5 / 76.5
D	Waxy maize	0 / 99

Molecular structure



2) Extrusion – Plasticizer : water

Control variables	Water content MC	Product temperature Tp °C	SME kWh/t
Min	0.21	105	101
Max	0.36	186	580

Experimental sets:

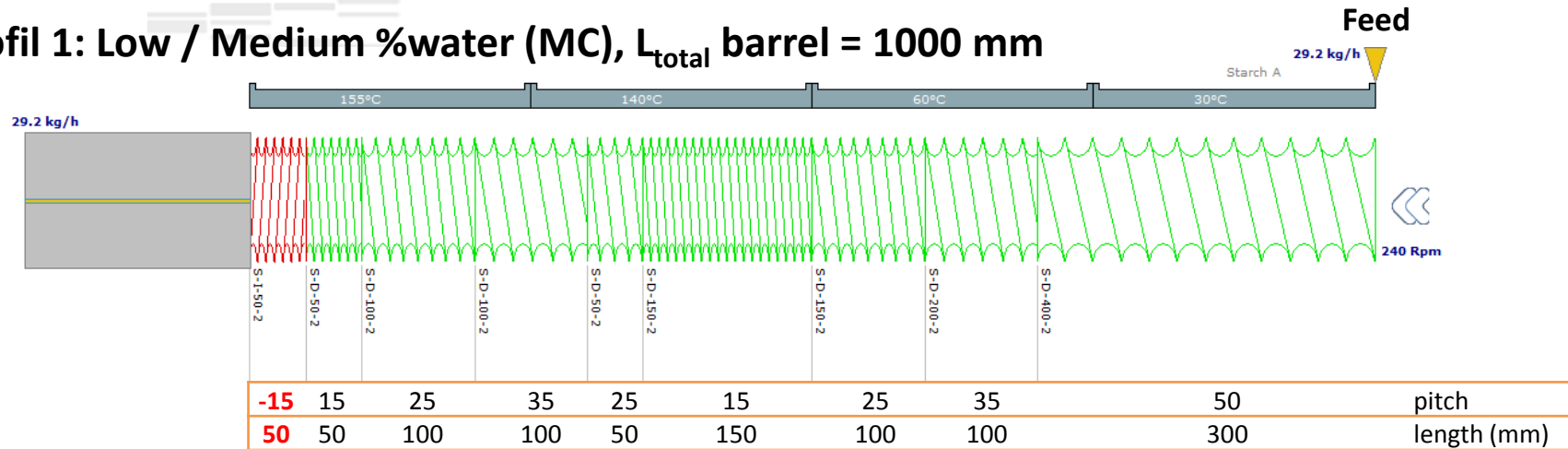
- Low MC with Med & High Tp
- High MC with Low & Med Tp

Q = 12 – 35 kg/h

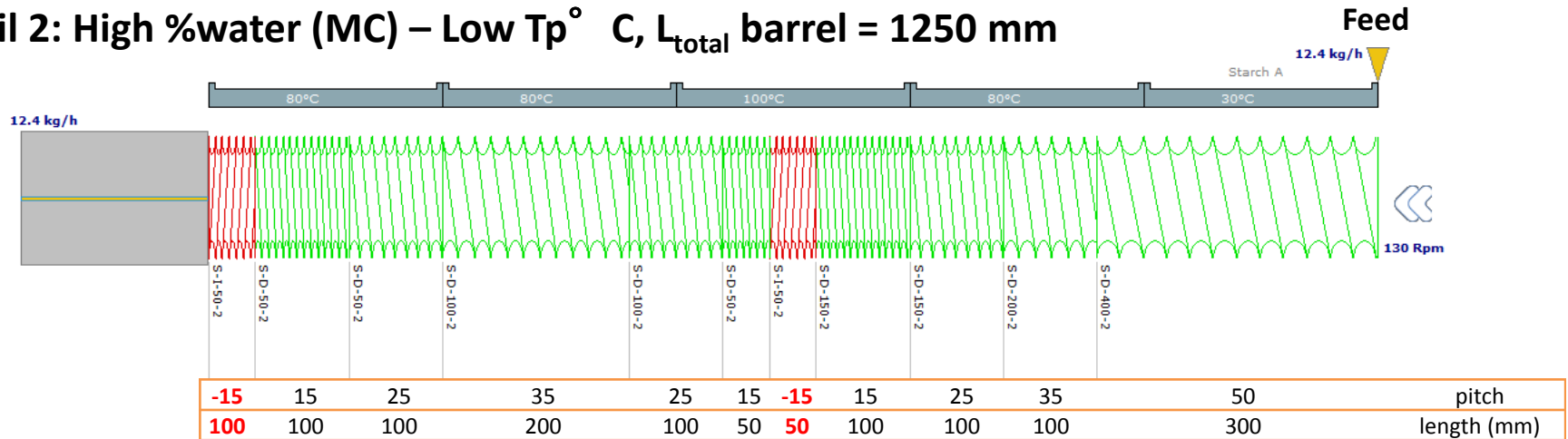
N = 80 – 240 rpm

Extruder configurations: Clextral BC45 & Slit die

Profil 1: Low / Medium %water (MC), L_{total} barrel = 1000 mm

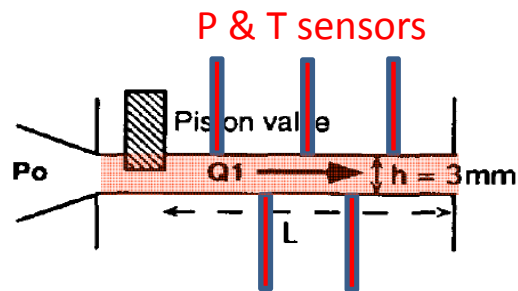
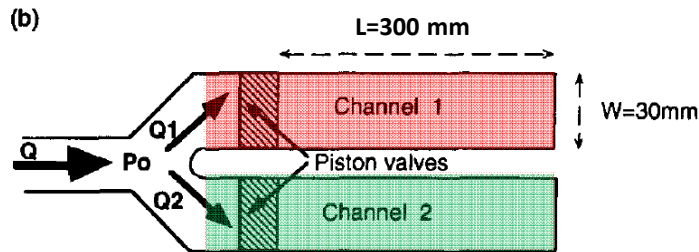


Profil 2: High %water (MC) – Low T_p° C, L_{total} barrel = 1250 mm



3) Determination of melt viscosity using Rheopac

Top view



- An in-line rheometer
- A slit die with **twin rectangular channels** attached to the die head of the extruder
 - Channel **1** for **the measurement**
 - Channel **2** for **bypass a part of the total flow rate Q** by adjusting the piston valves

Principle

- **Vary the local shear rate in the Channel 1**
- **Keep constant** the Q, SME and die head P **in extruder**
- Measurement of $\Delta P / \Delta L$ and **Q1** in **the Channel 1** to obtain shear viscosity

Using *Rabinowitsch correction*

General rheological model for the plasticized starch

- Power law fluid with **Non-newtonian** and **Shear thinning** behavior

$$\eta = K\dot{\gamma}^{n-1}$$

Case 1) Dependency of K and n only on **temperature (T)** and **moisture content (MC)**

$$K = K_o \left[\frac{E}{R} \left(\frac{1}{T} - \frac{1}{T_o} \right) - \alpha(MC - MC_o) \right]$$
$$n = a_1 T + a_2 MC + a_3 TMC$$

Case 2) Dependency of K and n on thermomechanical history through a **specific mechanical energy (SME)** term

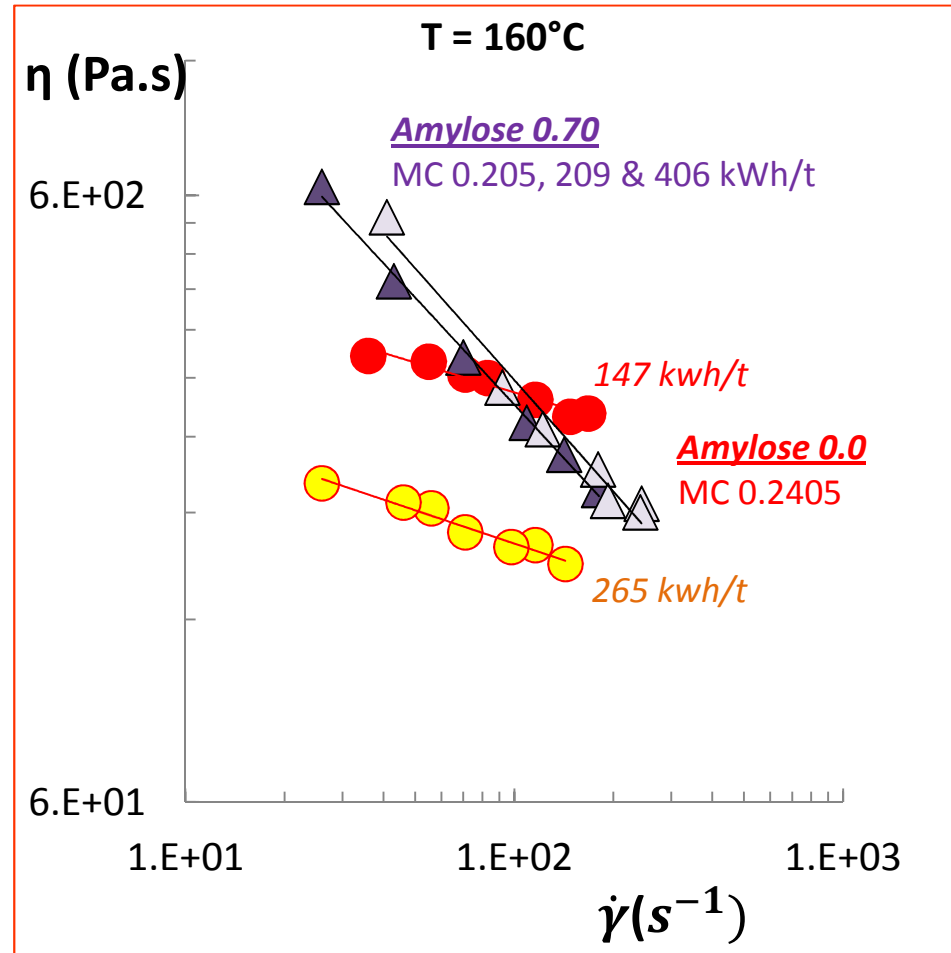
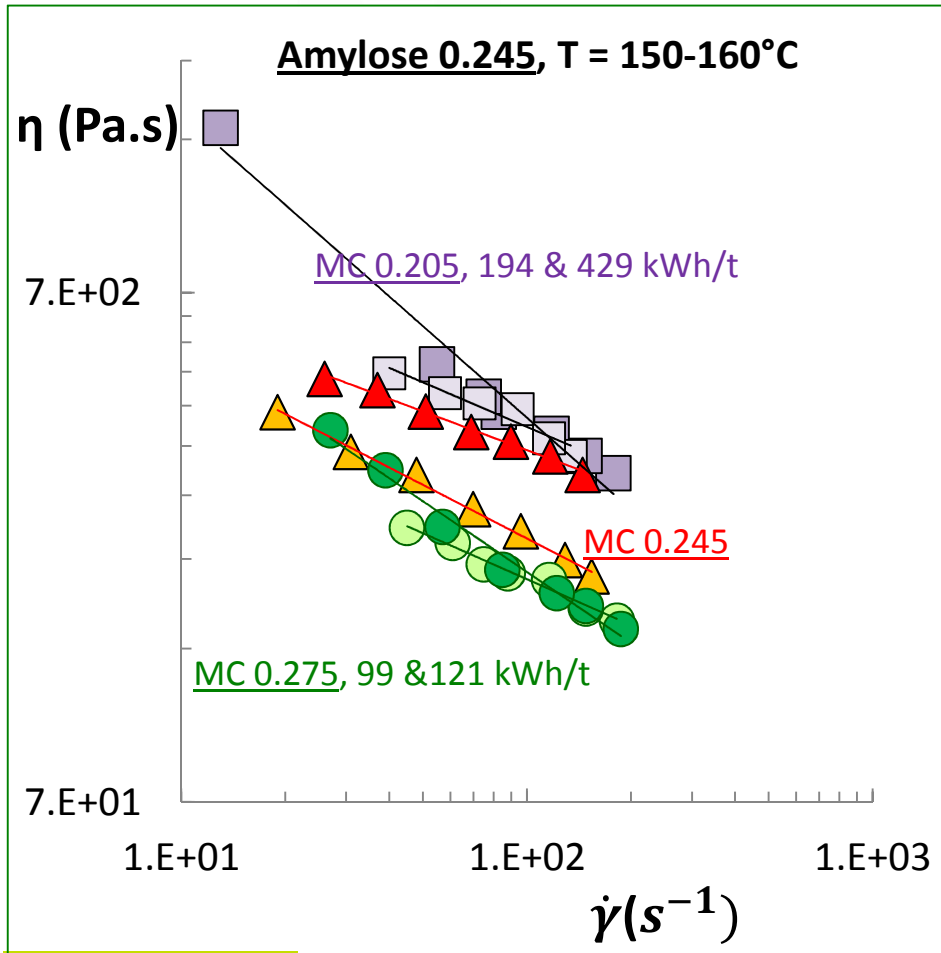
$$K = K'_o \left[\frac{E}{R} \left(\frac{1}{T} - \frac{1}{T_o} \right) - \alpha(MC - MC_o) - \beta(SME - SME_o) \right]$$
$$n = n_o + a_1 T + a_2 MC + a_3 SME + a_4 TMC + a_5 TSME + a_6 MCSME$$



RESULTS & DISCUSSIONS

Shear viscosity of molten starch: SME (+) then viscosity (-)

**Effect of Specific Mechanical Energy:
Complex dependency with amylose, water content & temperature**



Determination of shear viscosity parameters for **Ludovic®** input

Rheopac data:

$$\eta = K\dot{\gamma}^{n-1};$$

$K, n = f(\text{MC}, T_p, \text{SME})$

Starch	A	B	C	D
Amylose content	0.7	0.47	0.245	0
K'_o (Pa.s)	1.13E+07	4.15E+05	7.54E+07	1.34E+06
E/R (K)	11,298	5,638	9,869	9,350
α	14.49	14.86	38.94	26.32
β (kJ/kg) ⁻¹	0	2.68E-04	1.25E-03	1.59E-03
n_o	0	0	0.34	0
a_1 (°C) ⁻¹	2.69E-03	1.87E-03	1.06E-06	8.92E-04
a_2	0	6.13E-01	9.69E-01	0
a_3 (kJ/kg) ⁻¹	0	0	2.08E-05	1.56E-04
a_4 (°C kJ/kg) ⁻¹	0	2.68E-03	1.09E-05	1.44E-02
a_5 (°C) ⁻¹	0	0	0	0
a_6 (kJ/kg) ⁻¹	0	8.36E-05	2.18E-04	0

$$n = a_1 T + a_2 MC + a_3 TMC$$

$$K = K_o \left[\frac{E}{R} \left(\frac{1}{T} - \frac{1}{T_o} \right) - \alpha (MC - MC_o) \right]$$

Compute **new parameters**
 $K, n = f(\text{MC}, T_p)$

Create **Set of data points**
 $(T_p, \dot{\gamma}, \text{SME}, \eta)$

How to activate **SME** coupling in viscosity law for Ludovic[®] material input ?

Product Name: Starch D

Thermal Characteristics | **Viscosity**

1) Create Set of Points

Set of Points
 Carreau Yasuda Law
 Power Law
 User's Law

Viscosity Law = $f(\text{Temp, Shear Rate, Total Dissipated Energy})$

Parameter 1 : Temperature
 Parameter 2 : Shear Rate
 Parameter 3 : Standard: Total Dissipated Energy Fixed Value (uncoupled)

Temp (°C)	Shear Rate (s-1)	Total Dissipated Energy	Viscosity (Pa.s)
90	10	20	6041.661948
90	10	30	5967.904831
90	10	45	5858.954026
90	10	67.5	5699.245857
90	10	101.25	5467.809122
90	10	151.875	5138.155868
90	10	227.8125	4680.563793
90	10	341.71875	4069.438693
90	10	512.578125	3299.050944

Points Count: 1,573

view SOP | Import Excel | Export to | Delete | Digitize | Test SOP

Modify | Cancel

power law testing:Patricia_D_S8 THESE SME

File Simulation ?

Main Extruder Products Process Options **Execute** Global Results RTD Results Results f(x)

Numerical parameters:
 Precision (°C): 2.0
 Max Iterations #: 50
 Reverse and Direct Precision: 1

Output Temperature (°C):

2) Active coupling

Compute ... Local Installation...

Viscosity Coupling Mode:
 Coupling Mode: **Active Coupling**

Coupled Viscosity Computation for:
 - Starch D on : Total Dissipated Energy (Standard Result)

Alarms Settings:

Alarm	Threshold
Torque/Shaft > Maximum	0 N.m
Max Temperature	0 °C
Min Temperature	0 °C
Max Pressure	0 Bar
Min Pressure	0 Bar

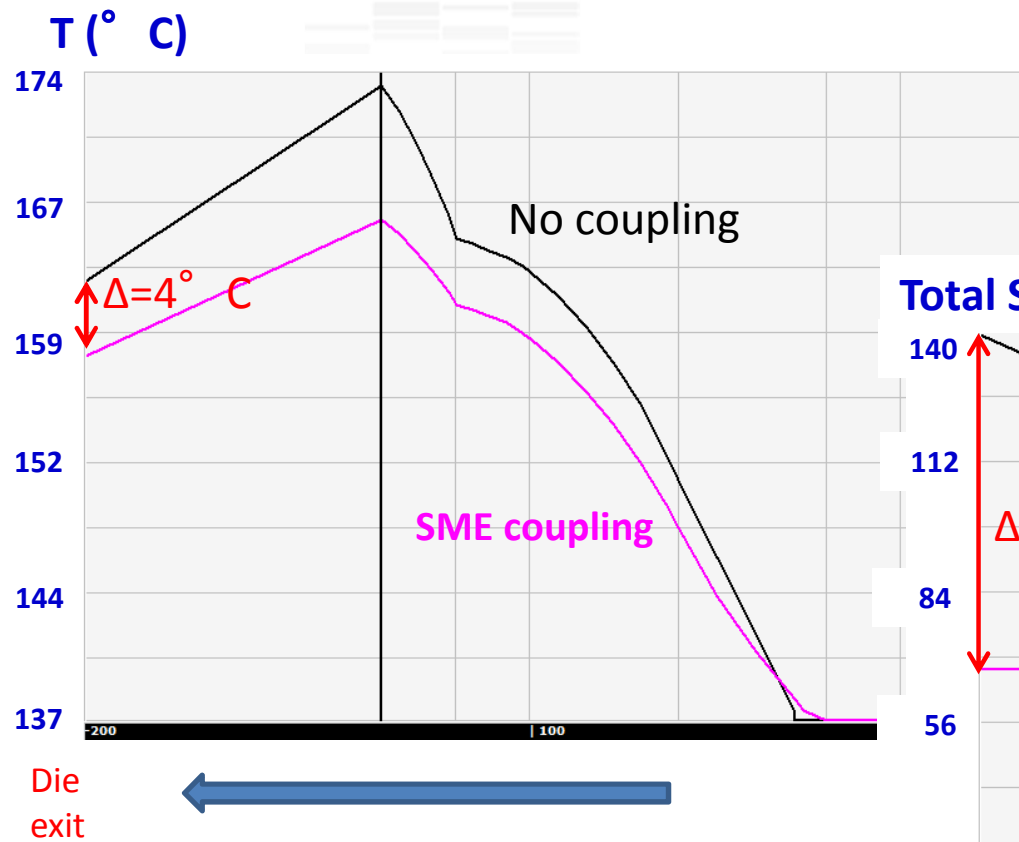
35.46 kg/h

120 Rpm

Modified solveur.exe (solveur_user0.d) new Local Permanent License... 3/25/2014 9:11:14 AM

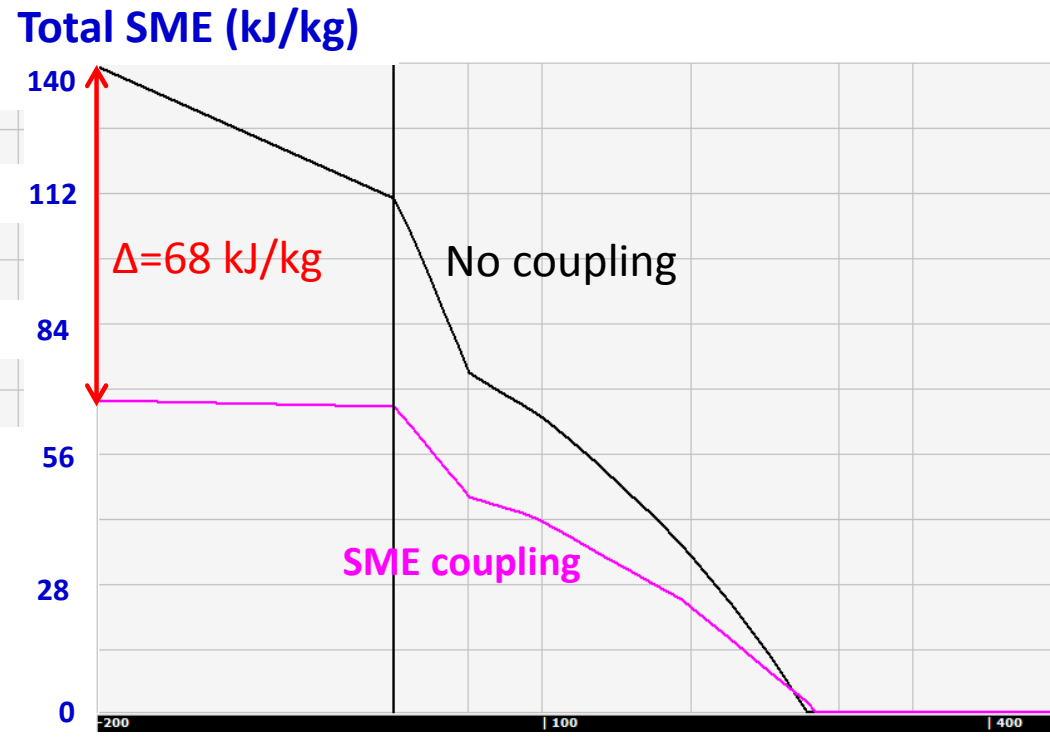
Ludovic simulation: Experimental set (*Amylose 0.0*, *Water 0.25*, *T_p 158 ° C*, *SME 131 kWh/t*)

Sensibility to thermomechanical history (**SME**, T_p, MC)



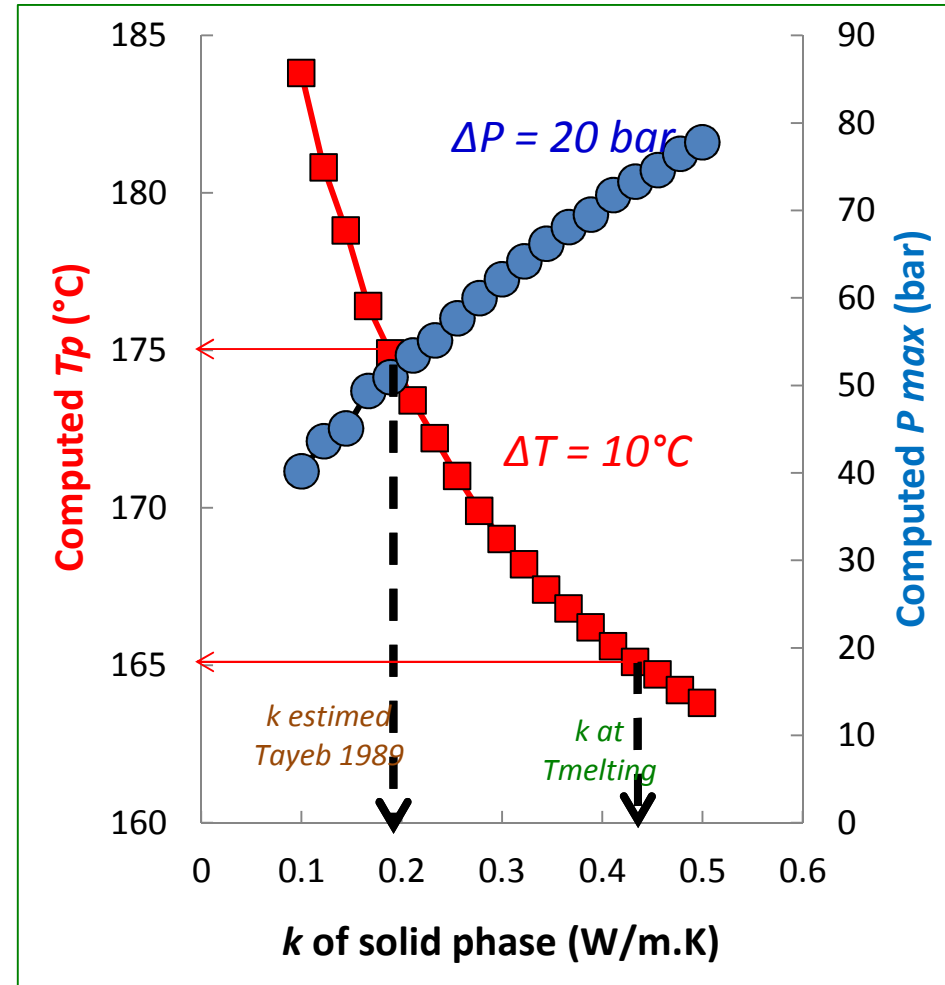
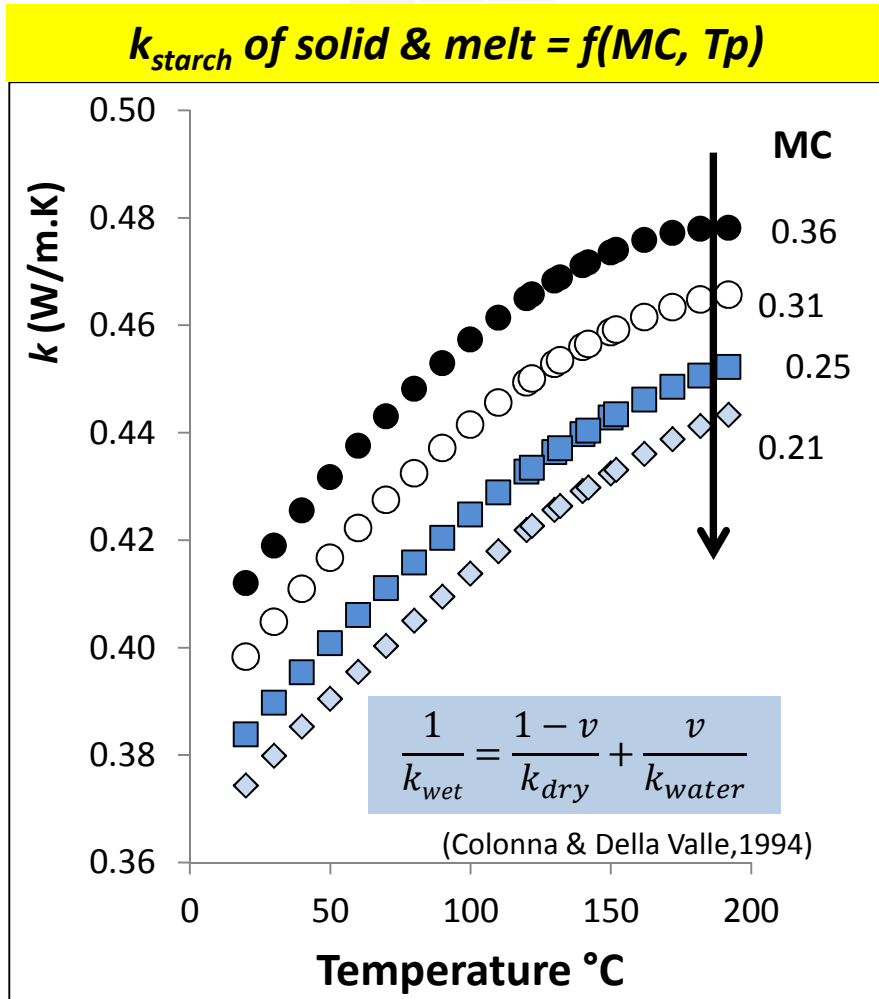
Melt viscosity $\eta = K\dot{\gamma}^{n-1}$

- $K, n = f(\text{Water}, T_p)$
- $K, n = f(\text{SME}, \text{Water}, T_p)$

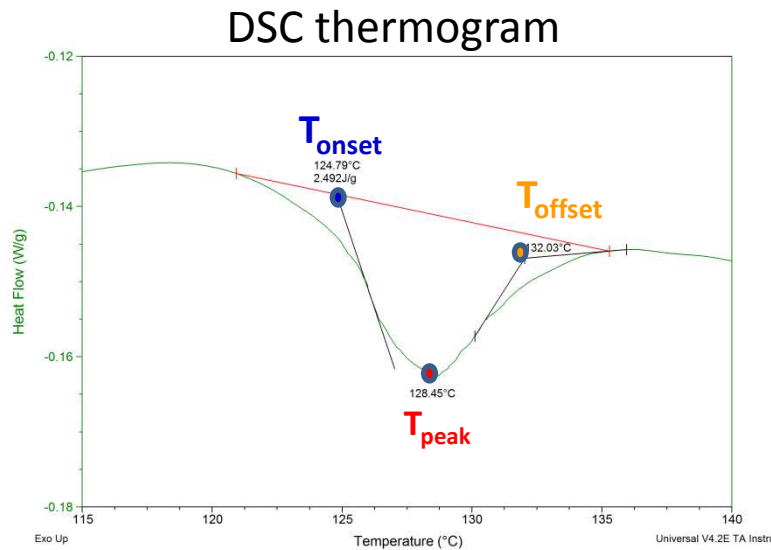


Ludovic simulation: Experimental set (Amylose 0.70, MC 0.21, T_p 166° C, SME 750 kJ/kg)

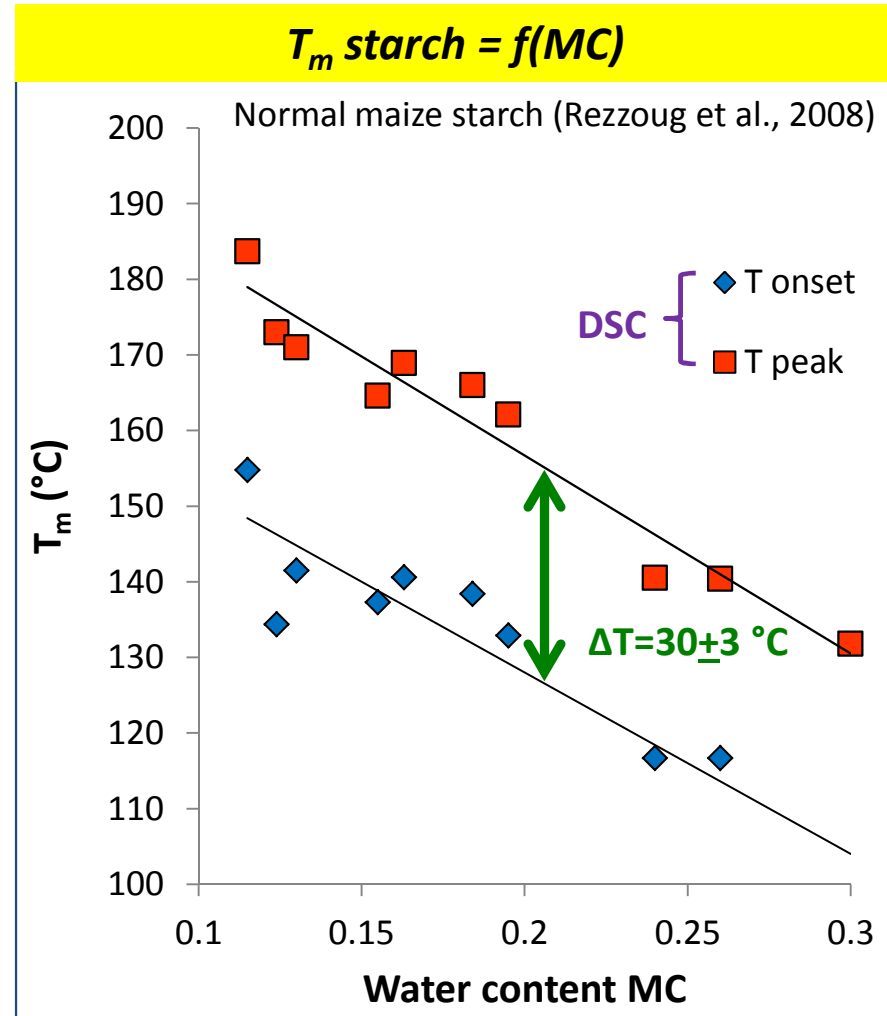
Sensibility to thermal conductivity k



Sensibility to melting temperature T_m



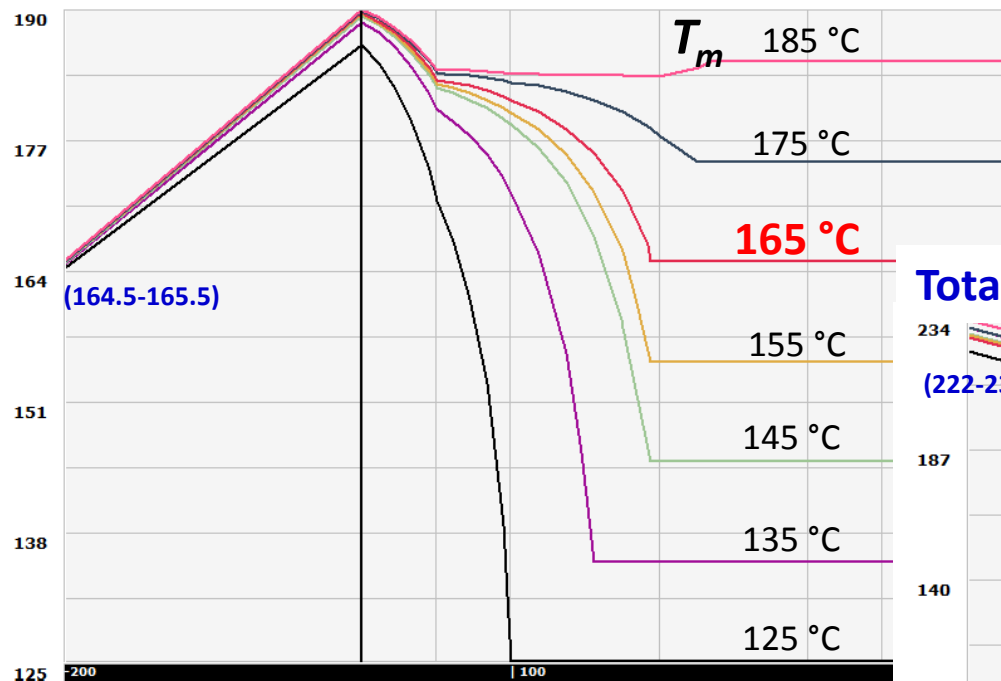
The higher amylose content,
the broader melting endotherme
(Mateev 2001)



Ludovic simulation: Experimental set (Amylose 0.70, MC 0.21, T_p 166 ° C, SME 750 kJ/kg)

Sensibility to melting temperature T_m

T_p (° C)



Die exit

Without SME - viscosity coupling

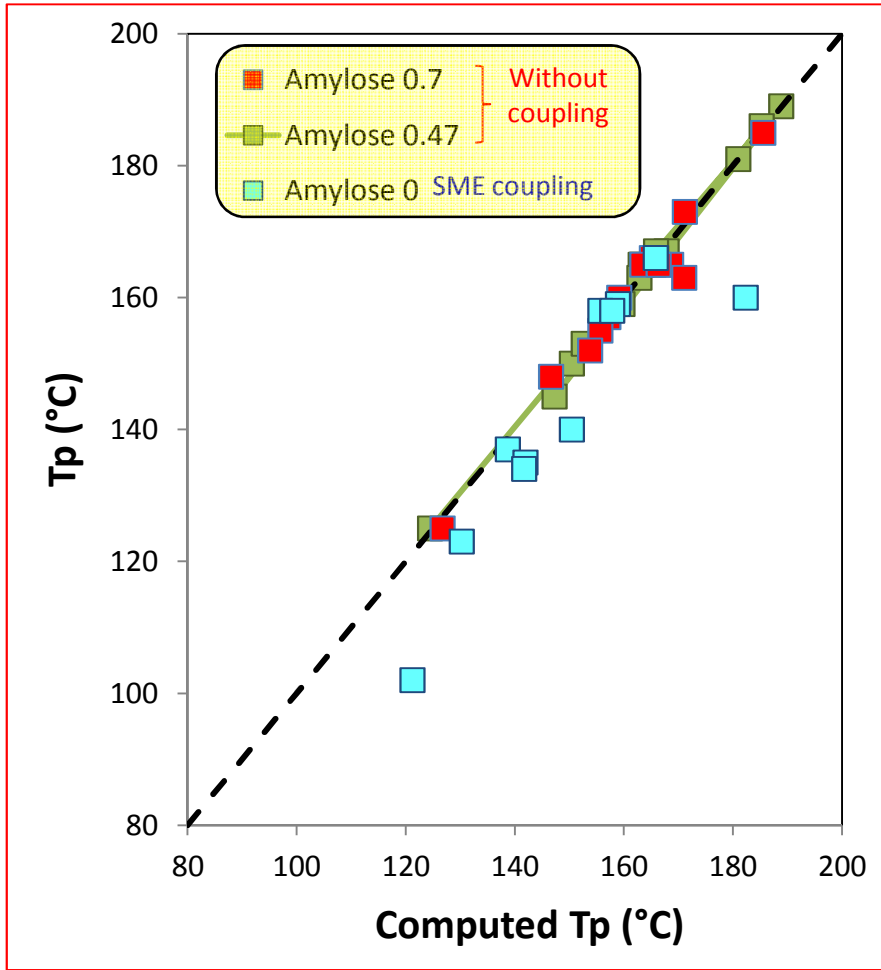
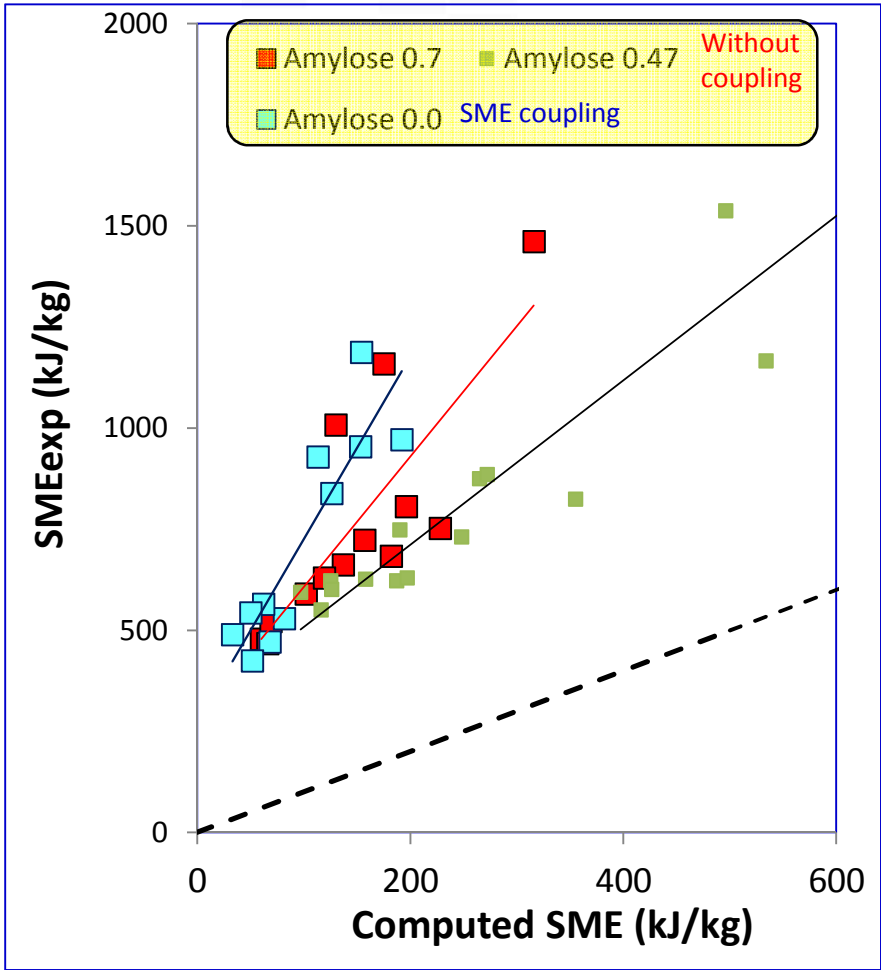
Total SME (kJ/kg)



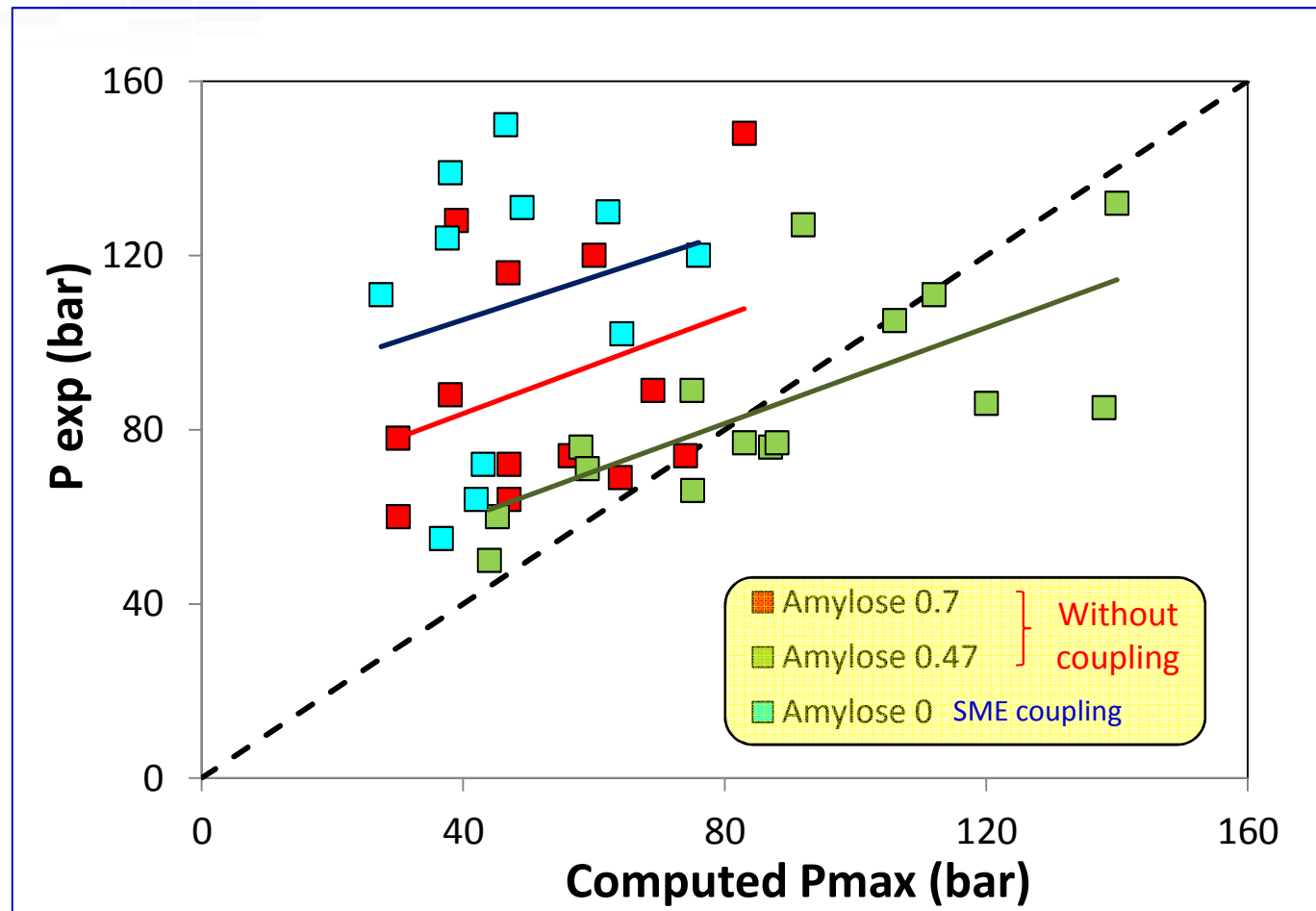
Die exit

Negligible effect of T_m !!!

Experiments vs Simulations

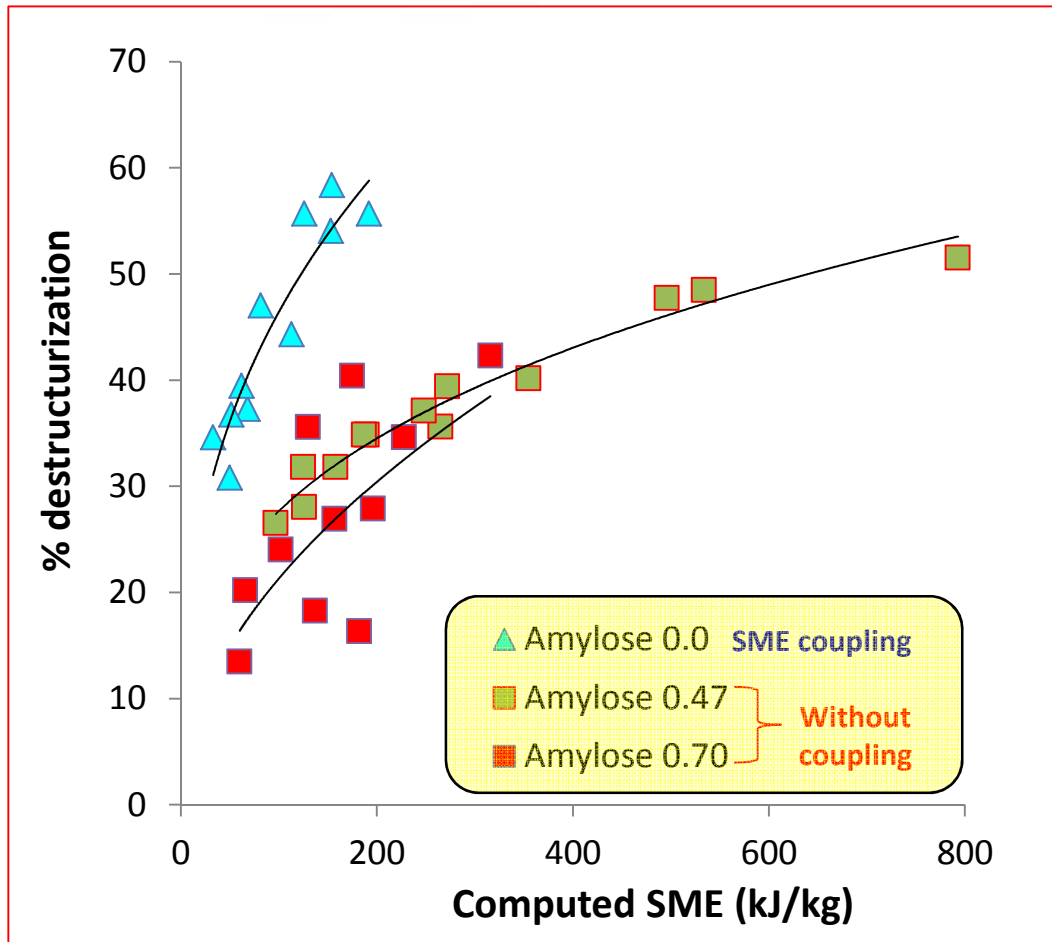


Experiments vs Simulations



Computing of starch destructurization using Ludovic

$$\% \text{ destructurization} = \frac{\Delta \text{ intrinsic viscosity}}{\text{initial intrinsic viscosity}}$$



Native maize starch

Amylose / Amylopectine	Intrinsic viscosity (ml/g)
0 / 100	185
24.5 / 75.5	159
47 / 53	132
70 / 30	104

Amylopectine (higher MW) is more sensitive to thermomechanical treatment than **amylose (lower MW)**



CONCLUSIONS

- The determination of **material properties** is primordial
 - The *rheological behavior* of molten polymer,
f(thermomechanical history)
 - The *thermal physical properties* of material,
f(T , water content)
- The **computed dissipated mechanical energy** is underestimated
 - Well correlation between SME_{computed} and the $SME_{\text{experimental}}$
 - Well correlation between % starch destructurization and SME_{computed}

PERSPECTIVES

- Integration of Ludovic® with phenomenological model of expansion

1) Coupling the model with Ludovic®

- Simulation and validation (experiments)

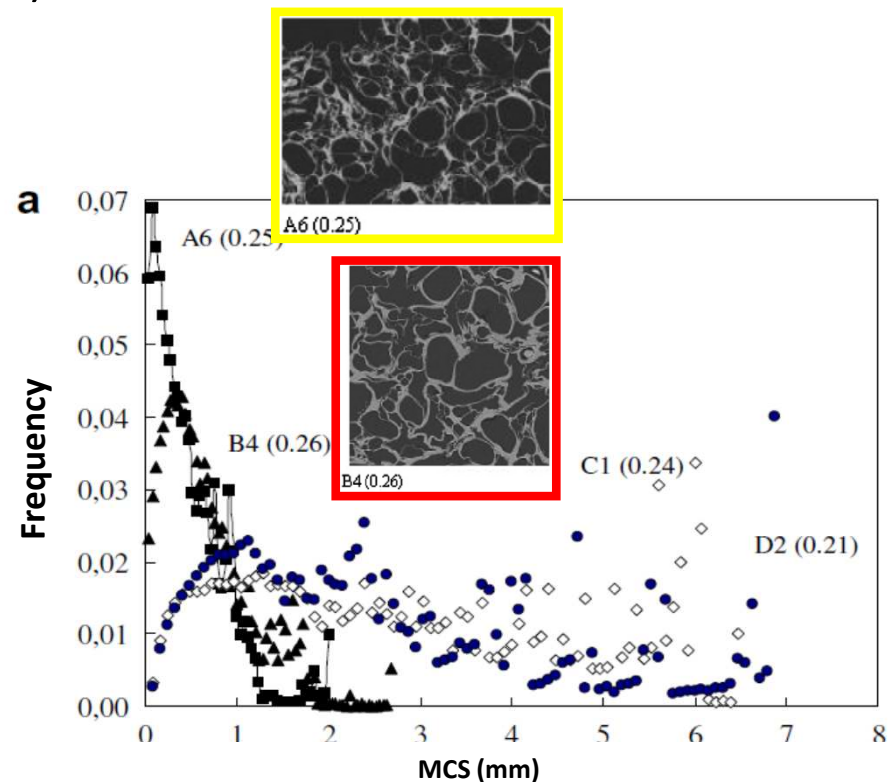
- New Ludovic® outputs:

1) Macrostructure:

- Indices Expansion Volumique (VEI)
- Indices Expansion Radial & Longitudinal
- Anisotropie factor

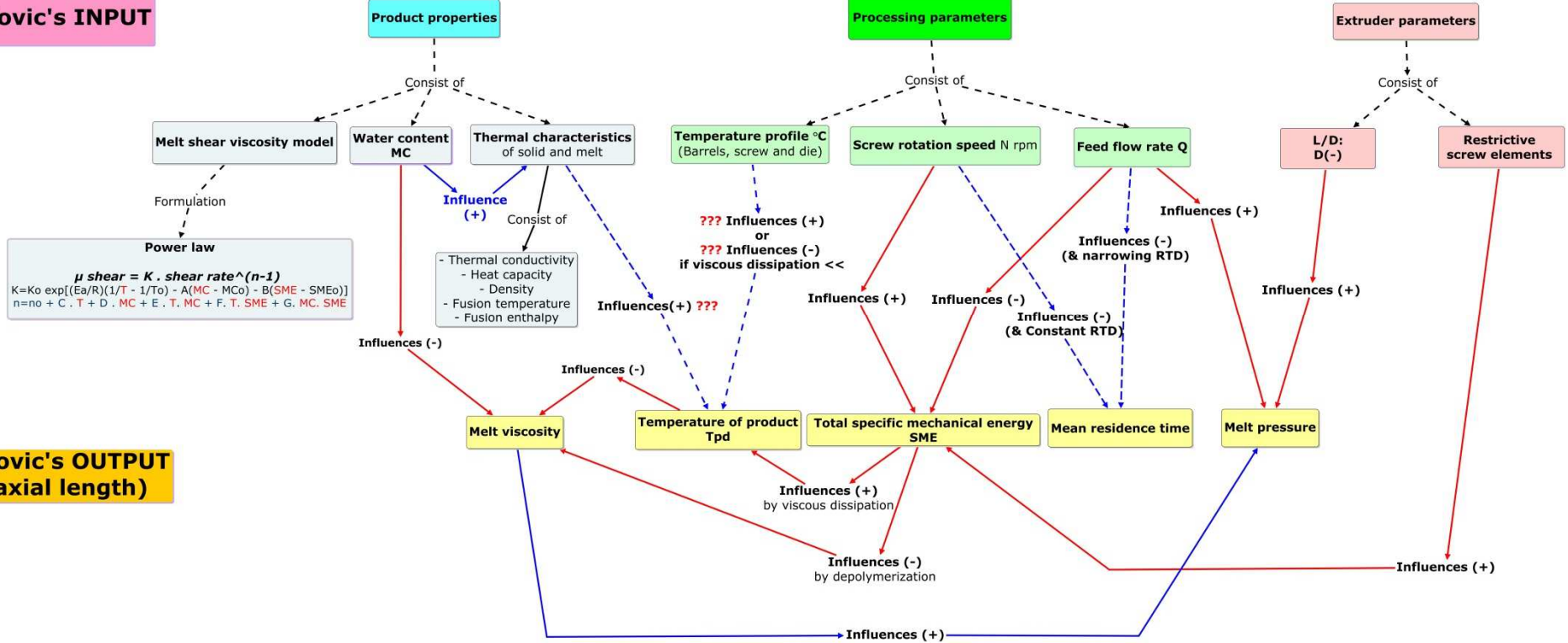
2) Microstructure:

- Mean cell size (MCS)
- Mean cell wall thickness (MCWT)
- Finesse



Concept map: Structure of Ludovic®

Ludovic's INPUT



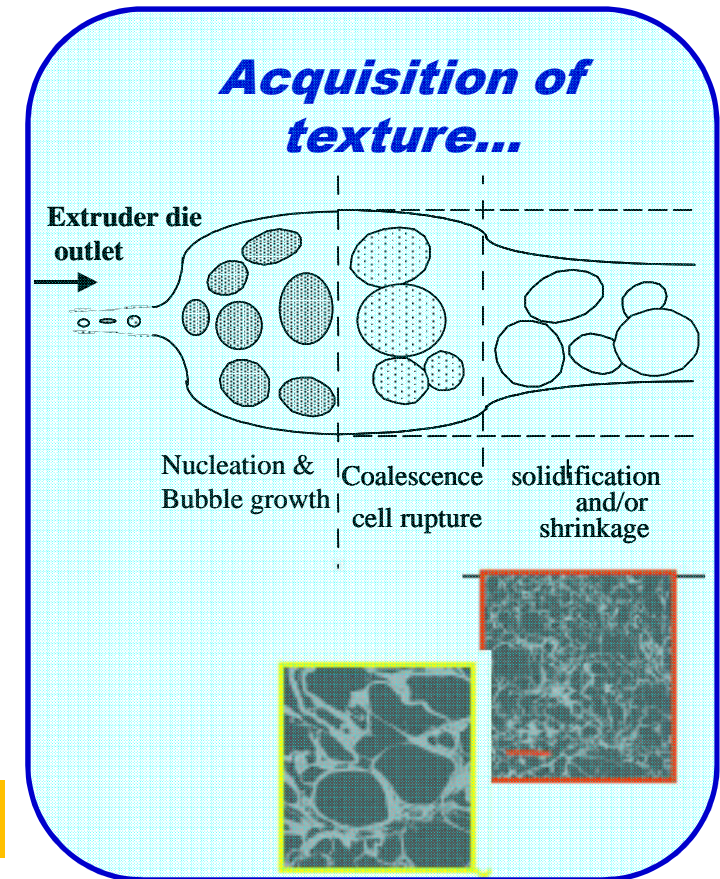
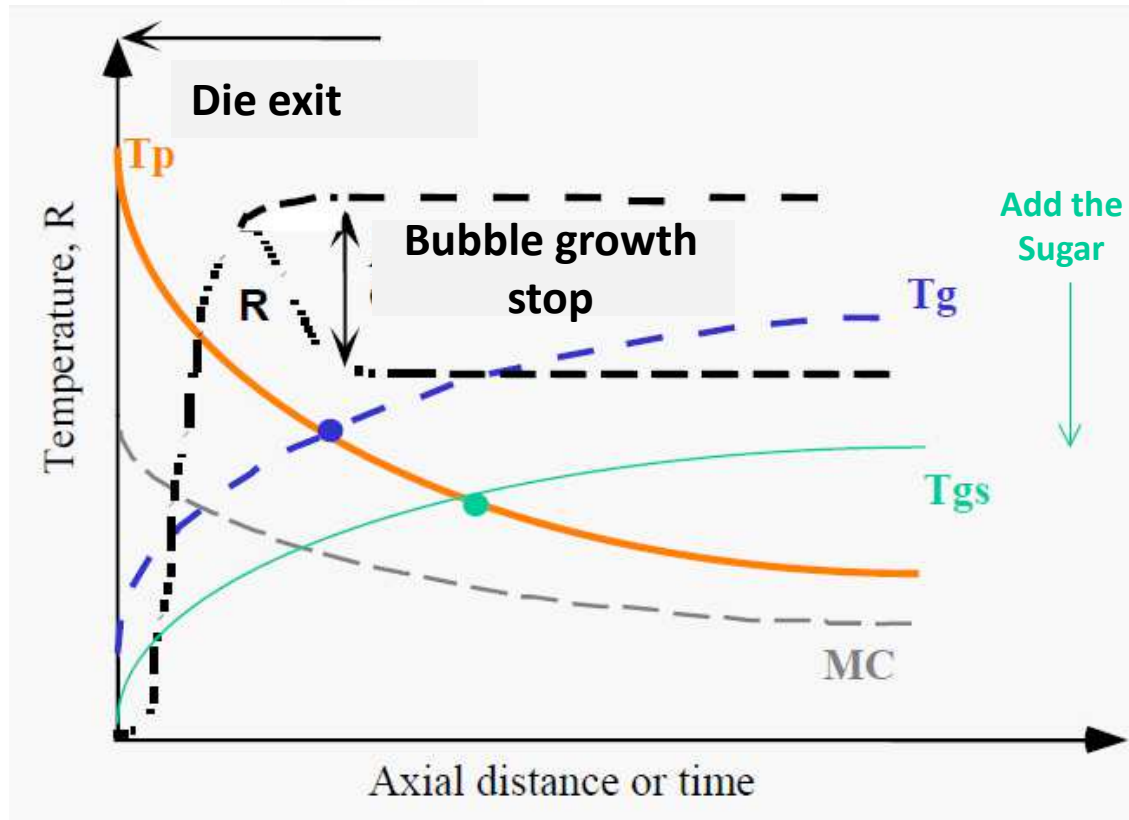
Ludovic's OUTPUT f(axial length)

**Input variables of models =
Extrusion's output variables at die exit (calculated by Ludovic®)**

Context: Starchy material processing using extrusion-cooking

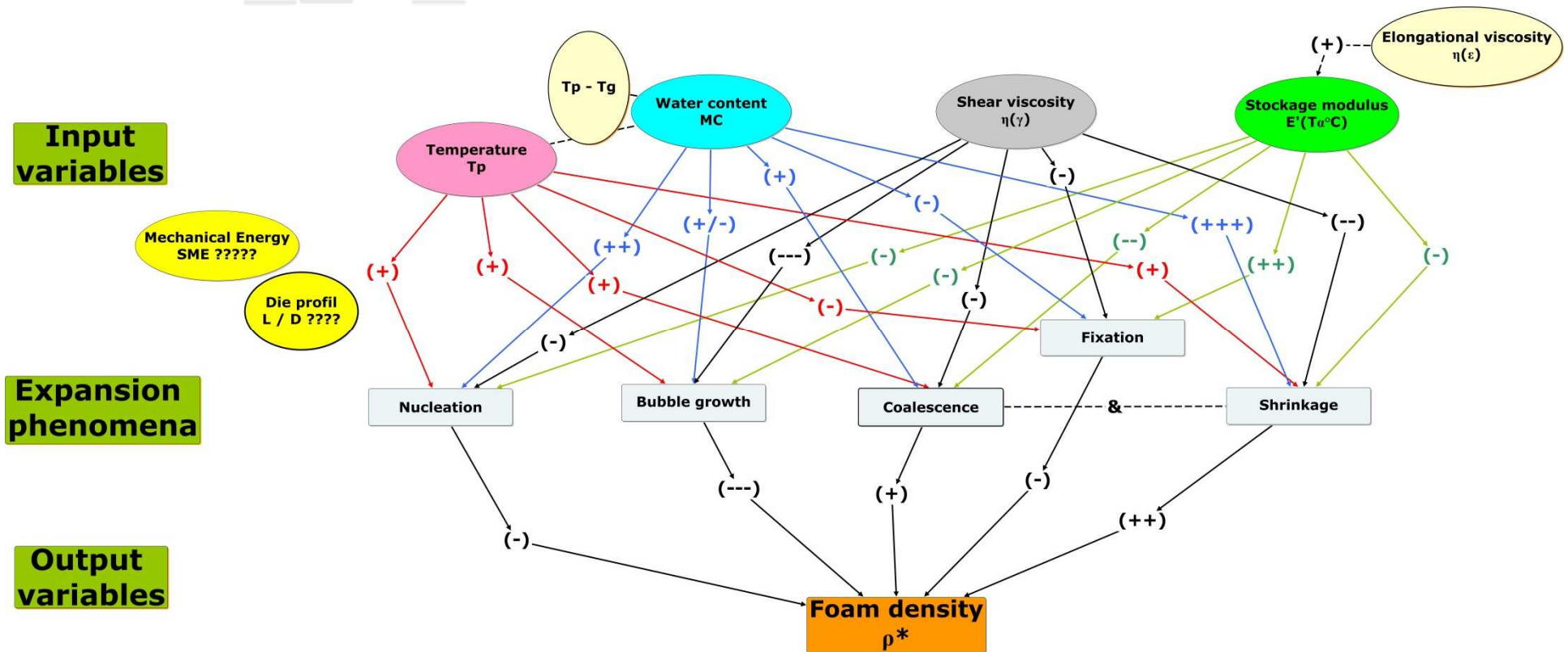
Expansion by extrusion cooking:

Relation with Glass transition temperature T_g



The bubble growth stops (solidification) at $T_p > T_g + 30^\circ\text{C}$

Concept map: Phenomenological model of expansion



$$\rho^* \approx z \otimes [\eta(\gamma)]^x \otimes [MC] \otimes [Tp] \otimes [E'(T\alpha)]^y$$

where $z, x, y = f(MC, T_p, \dots)$

Volumetric expansion indices VEI

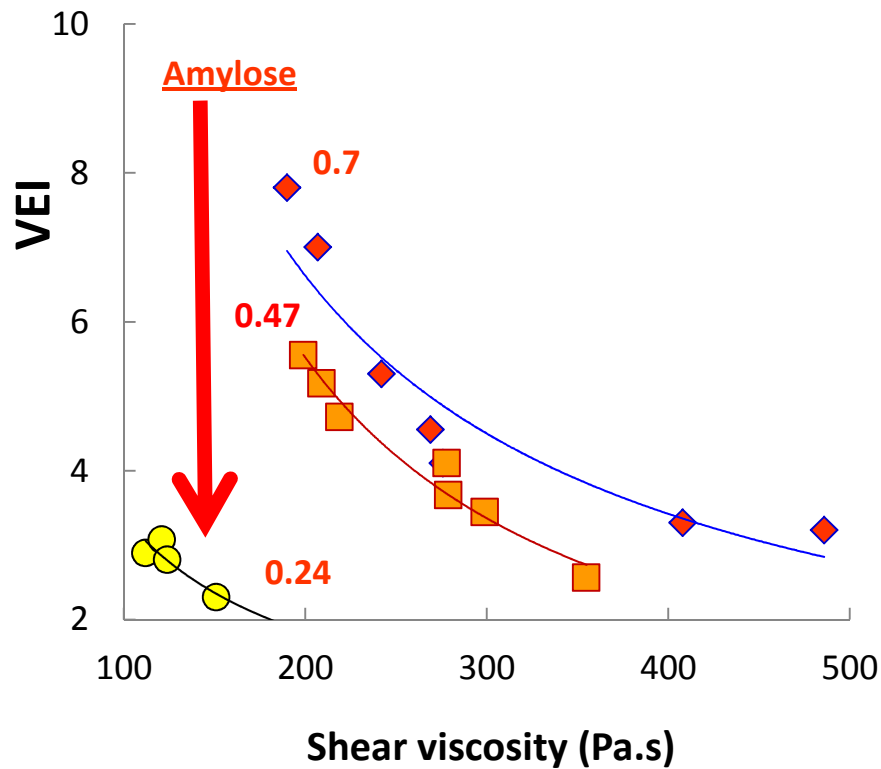
$$VEI = \frac{\rho_m(1 - MC_m)}{\rho^*(1 - MC^*)}$$

$$VEI = \left(\frac{\eta(\dot{\gamma})}{a} \right)^n$$

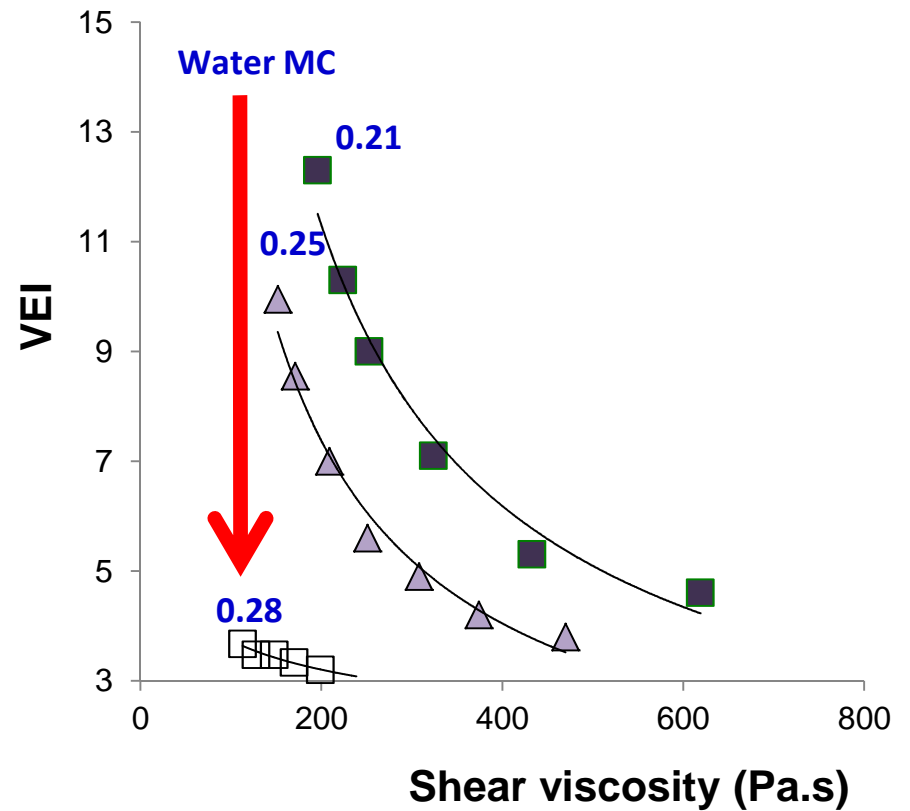
$$\left. \begin{aligned} a &= a_1 MC + a_2 T_p + a_3 MC \cdot T_p + a_4 \ln(E'(T_\alpha)) \\ n &= (n_1 MC + n_2 T_p + n_3 MC \cdot T_p) \cdot \ln(E'(T_\alpha)) \end{aligned} \right\}$$

Water = 0.245, 185°C, 150 kWh/t, h die = 3 mm

Higher Amylose% → higher $E'(T_\alpha)$ → higher $\eta(\epsilon)$



Amylose 70%, 165°C, 200 kWh/t, h die = 3 mm



Thank you....



Discussion....



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