The Deborah Calculator: Using Rheological Indicators to Predict Film Instability in Extrusion Coating

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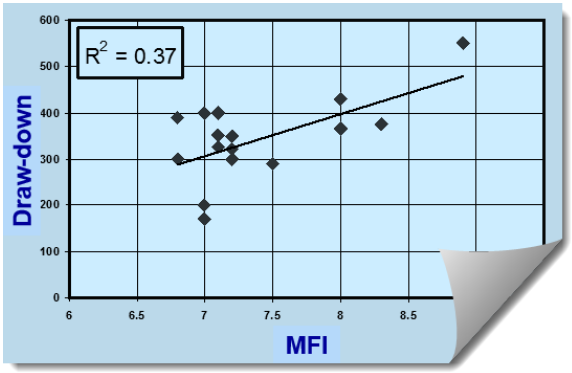
Per-Åke
I'm missing you!

Melt Flow Index

- **OK** to define application e.g. Extrusion coating (MFI = 6 – 9 g/10 min.)
- **NOT** an indicator for performance e.g. Draw-down and Edge instability



Introducing G', Eta0 and Activation energy

"G' – a Rheological Indicator to Predict the Extrusion Coating Performance of LDPE"
TAPPI PLACE Conference 2003 (Rome)

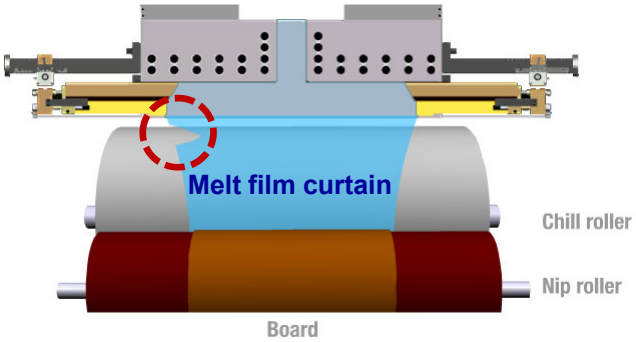
"Rheological Indicators to Predict the Extrusion Coating Performance of LDPE"
TAPPI PLACE Conference 2004 (Indianapolis)

"Monitoring of Rheological Properties of LDPE"
TAPPI PLACE Conference 2005 (Vienna)

"Activation Energy to Predict the Adhesion of LDPE to Aluminium Foil in Extrusion Lamination"
TAPPI PLACE Conference 2015 (Nice)

"Rheological measurements for characterization of polyolefins: low-density polyethylene (LDPE) for extrusion coating"
Test method TAPPI/ANSI T 702 om-19

Edge instability in converting: causing problems in dairy

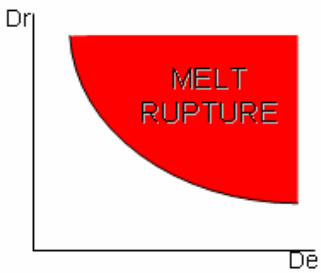


Dimensionless numbers

In literature[1] the performance of coating process can be described with 2 dimensionless numbers, Draw ratio (Dr) and Deborah number (De):

$$\textbf{Draw ratio (Dr)} = \frac{\textbf{velocity at nip}}{\textbf{velocity at die exit}}$$

$$\textbf{Deborah number (De)} = \frac{\textbf{relaxation time of polymer}}{\textbf{residance time in air gap}}$$



The interpretation of this plot is simply:

high Deborah number → low Draw-ratio (elastic response)
low Deborah number → high Draw-ratio (viscous response)

[1] *The Draw ratio-Deborah number diagram: A useful tool for coating applications by S. Bourriguad, G. Marin, V. Dabas, C. Dupuy, D. Silagy*

The equations as published[1]

$$Dr = \frac{\mu_F 2\rho e_0 l_0}{Q}$$

$$De = \frac{Q\lambda(T_0)exp\left[\frac{E_0}{R}\left(\frac{1}{T}-\frac{1}{T_0}\right)\right]}{2\rho e_0 l_0 h}$$

μ_F = drawing velocity
ρ = melt density
e_0 = die gap
l_0 = die half width
Q = mass flow rate
h = air gap
λ = polymer relaxation time
E_0 = activation energy

Simple machine settings and polymer properties

[1] *The Draw ratio-Deborah number diagram: A useful tool for coating applications by S. Bourriguad, G. Marin, V. Dabas, C. Dupuy, D. Silagy*

A rheological indicator: Relaxation time of polymer - λ

Temperature dependent

"Lambda [λ] is an average time of the relaxation time spectrum and appears to be the simplest to be measured, still being quite relevant to be correlated to the elongational behavior in processing[1]."

$$\frac{\lambda(T)}{\lambda(T_0)} = exp\left[\frac{E_0}{R}\left(\frac{1}{T}-\frac{1}{T_0}\right)\right]$$

[1] *The Draw ratio-Deborah number diagram: A useful tool for coating applications by S. Bourriguad, G. Marin, V. Dabas, C. Dupuy, D. Silagy*

Lambda - λ

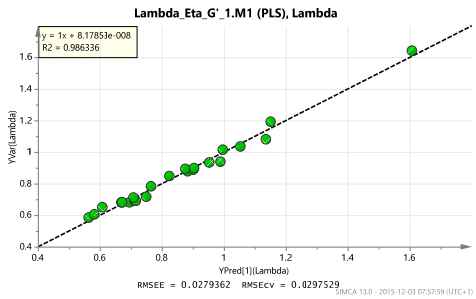
Using Cross 3-parameters equation:

$$\eta^* = \frac{\eta_0}{1+(\lambda\omega)^n}$$

or

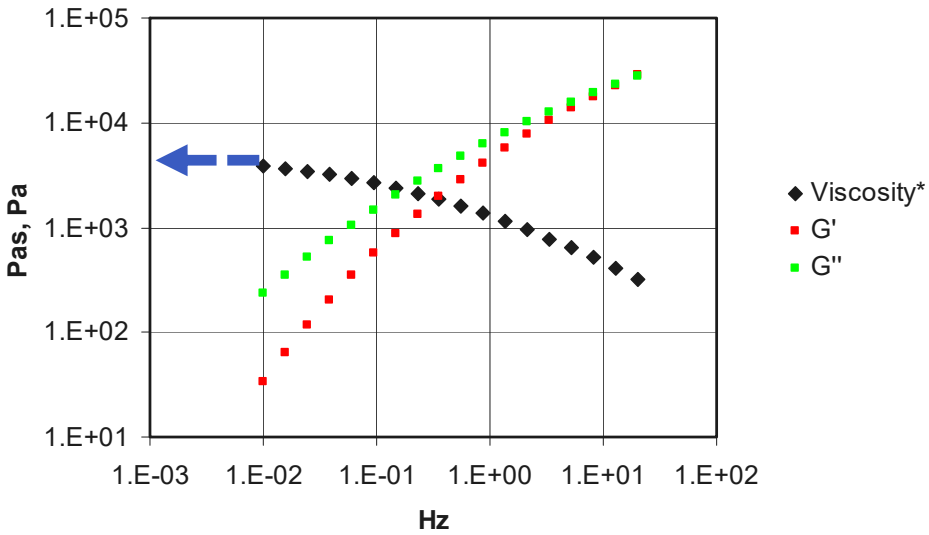
Calculating from Eta0 and G':

$$\lambda = C + \beta_1 * Eta0 + \beta_2 * G'$$



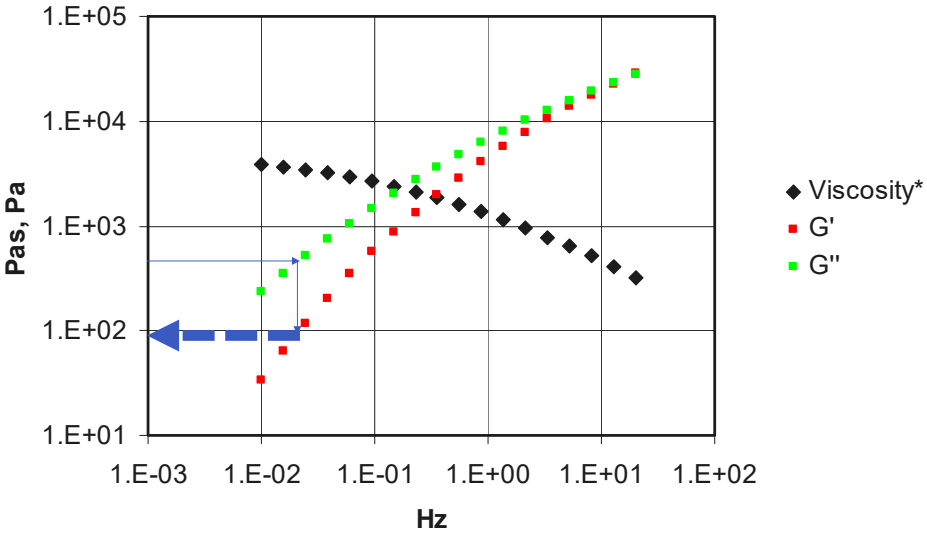
Eta0 is extrapolated to 0 Hz

According to Test method TAPPI/ANSI T 702 om-19



G' is interpolated at G" = 500 Pa

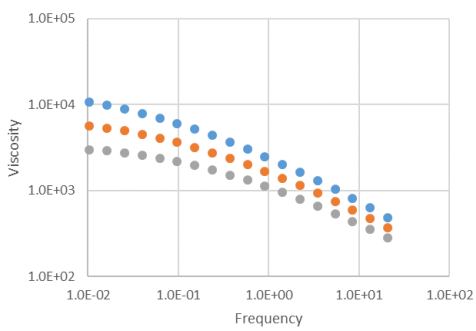
Preferably by using a Cole-Cole plot

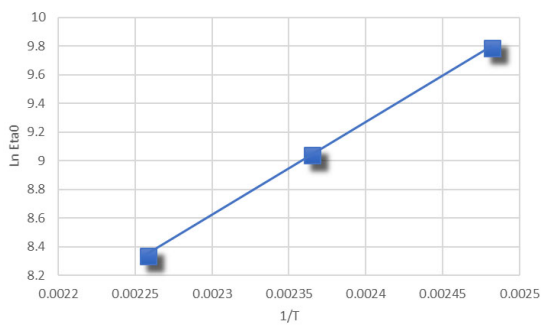


Activation energy – E0

1. Frequency sweeps at 130, 150 and 170°C.
2. Determine Eta0 for each temperature.
3. Calculate E0 from Arrhenius equation:

$$Eta0 = A\, e^{-E0/RT}$$





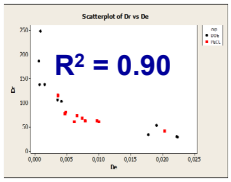
Extrusion coating trials

Confirmation of theory !

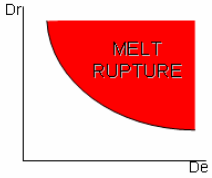
Factors → Responses → Plot → Theory

- LDPE grades
- Air gap
- Die gap opening
- Temperature
- Line speed

- Edge instability
- Draw-down speed



$$Dr = \beta_1 De^{-\beta_2}$$

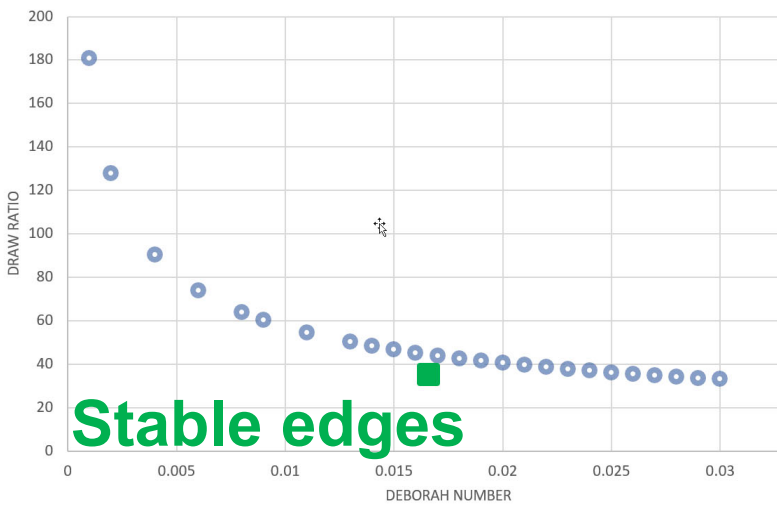


@ 2 different coating lines
@ 2 different ocasions

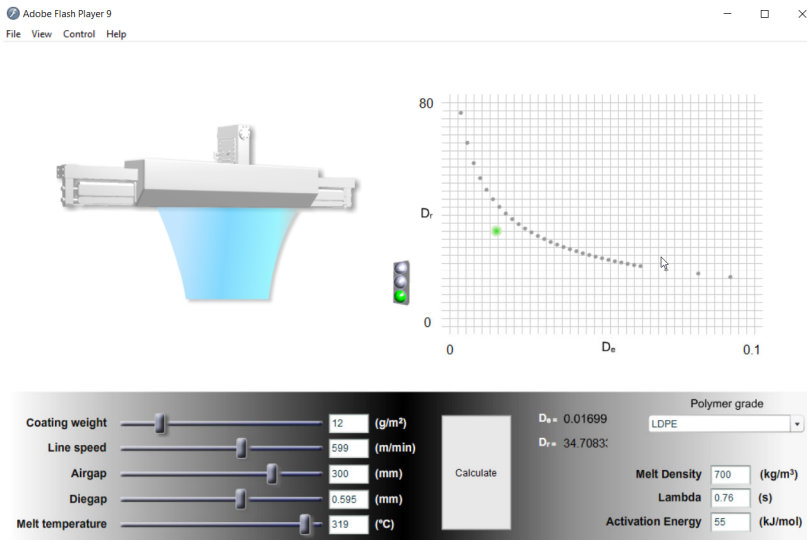
The Deborah calculator

Stable conditions

DEBORAH CALCULATOR FOR LDPE		
Input Material Properties:		
Eta0 @ 170°C	[Pas]	**4200**
Eta0 @ 150°C	[Pas]	**8500**
Eta0 @ 130°C	[Pas]	**18500**
G' @ 170°C	[Pa]	**110**
Calculations Material Properties:		
Lambda @ 170°C	[s]	0.76
E0 from 130-150-170°C	[kJ/mol]	55.0
Machine & Process Settings:		
Die gap opening	[mm]	**0.60**
Coating thickness	[gsm]	**12**
Air gap	[mm]	**300**
Film temperature	[°C]	**320**
Line speed	[m/min]	**600**
Lambda	[s]	*0.76*
E0	[kJ/mol]	*55.0*
Dr	[-]	*35.0*
De	[-]	*0.017*



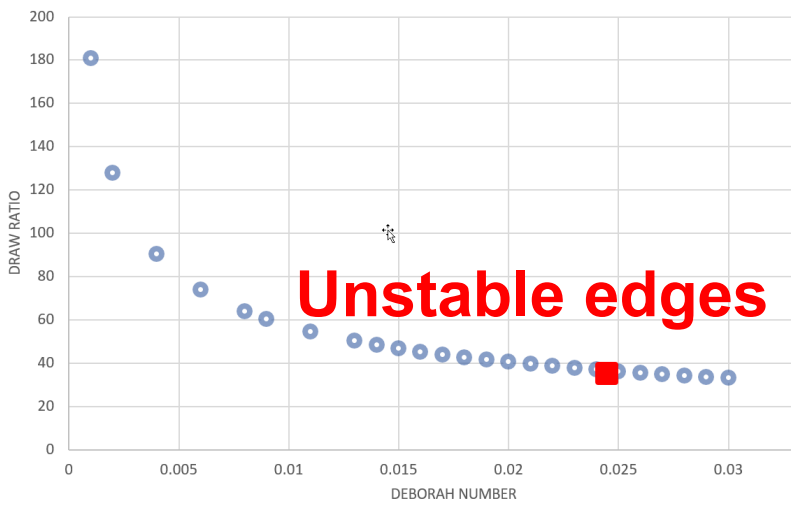
Deborah calculator in Adobe Flash Player



Example # 1

Melt temperature too low

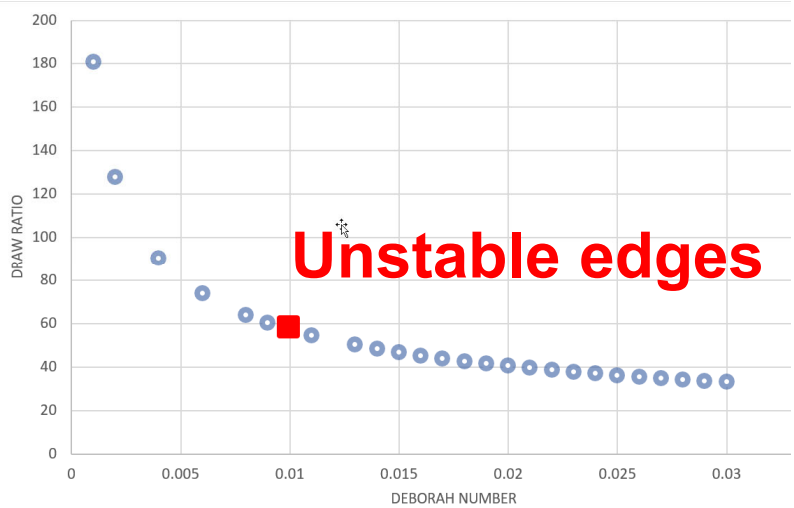
DEBORAH CALCULATOR FOR LDPE		
Input Material Properties:		
Eta0 @ 170°C	[Pas]	**4200**
Eta0 @ 150°C	[Pas]	**8500**
Eta0 @ 130°C	[Pas]	**18500**
G' @ 170°C	[Pa]	**110**
Calculations Material Properties:		
Lambda @ 170°C	[s]	0.76
E0 from 130-150-170°C	[kJ/mol]	55.0
Machine & Process Settings:		
Die gap opening	[mm]	**0.60**
Coating thickness	[gsm]	**12**
Air gap	[mm]	**300**
Film temperature	[°C]	**300**
Line speed	[m/min]	**600**
Lambda	[s]	*0.76*
E0	[kJ/mol]	*55.0*
Dr	[-]	*35.0*
De	[-]	*0.024*



Example # 2

Die gap opening too wide

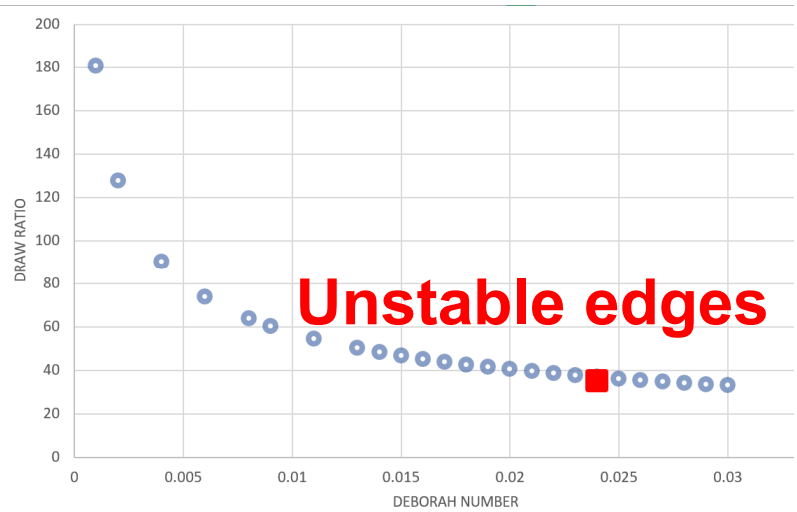
DEBORAH CALCULATOR FOR LDPE		
Input Material Properties:		
Eta0 @ 170°C	[Pas]	**4200**
Eta0 @ 150°C	[Pas]	**8500**
Eta0 @ 130°C	[Pas]	**18500**
G' @ 170°C	[Pa]	**110**
Calculations Material Properties:		
Lambda @ 170°C	[s]	0.76
E0 from 130-150-170°C	[kJ/mol]	55.0
Machine & Process Settings:		
Die gap opening	[mm]	**1.00**
Coating thickness	[gsm]	**12**
Air gap	[mm]	**300**
Film temperature	[°C]	**320**
Line speed	[m/min]	**600**
Lambda	[s]	*0.76*
E0	[kJ/mol]	*55.0*
Dr	[-]	*58.3*
De	[-]	*0.010*



Example # 3

Activation energy too low

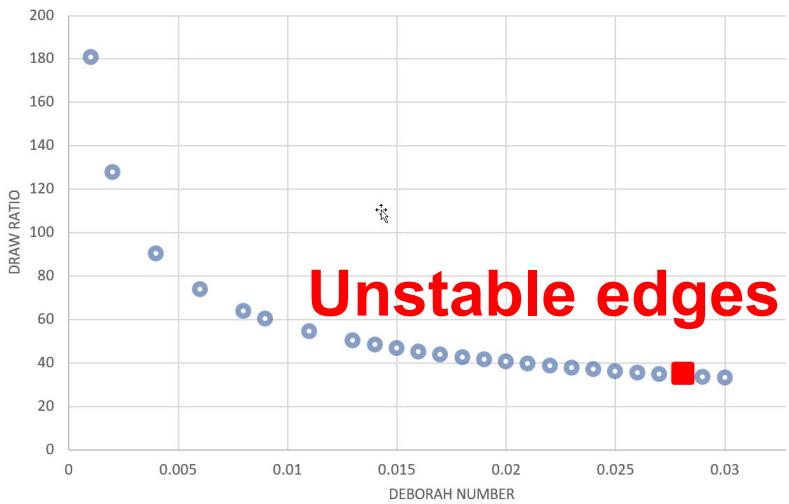
DEBORAH CALCULATOR FOR LDPE		
Input Material Properties:		
Eta0 @ 170°C	[Pas]	**4200**
Eta0 @ 150°C	[Pas]	**8200**
Eta0 @ 130°C	[Pas]	**16000**
G' @ 170°C	[Pa]	**110**
Calculations Material Properties:		
Lambda @ 170°C	[s]	0.76
E0 from 130-150-170°C	[kJ/mol]	**49.6**
Machine & Process Settings:		
Die gap opening	[mm]	**0.60**
Coating thickness	[gsm]	**12**
Air gap	[mm]	**300**
Film temperature	[°C]	**320**
Line speed	[m/min]	**600**
Lambda	[s]	*0.76*
E0	[kJ/mol]	*49.6*
Dr	[-]	*35.0*
De	[-]	*0.024*



Example # 4a

G' too high

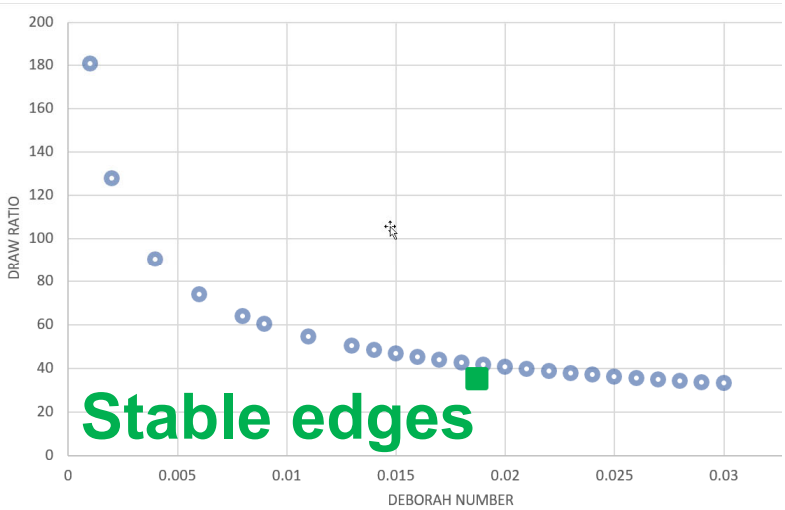
DEBORAH CALCULATOR FOR LDPE		
Input Material Properties:		
Eta0 @ 170°C	[Pas]	**4200**
Eta0 @ 150°C	[Pas]	**8500**
Eta0 @ 130°C	[Pas]	**18500**
G' @ 170°C	[Pa]	**140**
Calculations Material Properties:		
Lambda @ 170°C	[s]	1.29
E0 from 130-150-170°C	[kJ/mol]	55.0
Machine & Process Settings:		
Die gap opening	[mm]	**0.60**
Coating thickness	[gsm]	**12**
Air gap	[mm]	**300**
Film temperature	[°C]	**320**
Line speed	[m/min]	**600**
Lambda	[s]	*1.29*
E0	[kJ/mol]	*55.0*
Dr	[-]	*35.0*
De	[-]	*0.028*



Example 4b

Too high G' means lowering of line speed

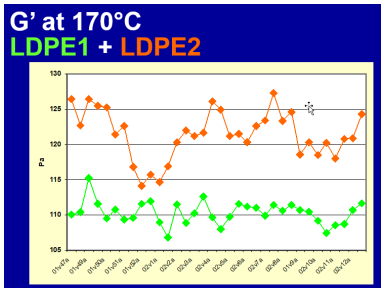
DEBORAH CALCULATOR FOR LDPE		
Input Material Properties:		
Eta0 @ 170°C	[Pas]	**4200**
Eta0 @ 150°C	[Pas]	**8500**
Eta0 @ 130°C	[Pas]	**18500**
G' @ 170°C	[Pa]	**140**
Calculations Material Properties:		
Lambda @ 170°C	[s]	1.29
E0 from 130-150-170°C	[kJ/mol]	55.0
Machine & Process Settings:		
Die gap opening	[mm]	**0.60**
Coating thickness	[gsm]	**12**
Air gap	[mm]	**300**
Film temperature	[°C]	**320**
Line speed	[m/min]	**400**
Lambda	[s]	*1.29*
E0	[kJ/mol]	*55.0*
Dr	[-]	*35.0*
De	[-]	*0.019*



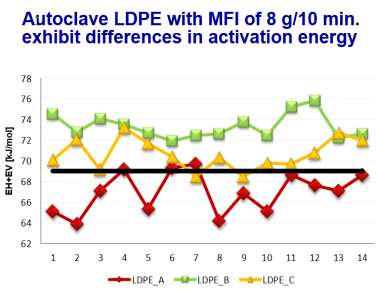
Capability to control rheological indicators

LDPE1 = LDPEB = LDPEY ??

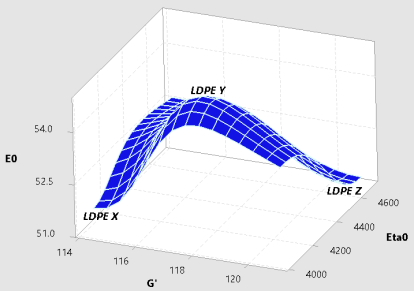
ROME 2003



NICE 2015



PORTO 2019

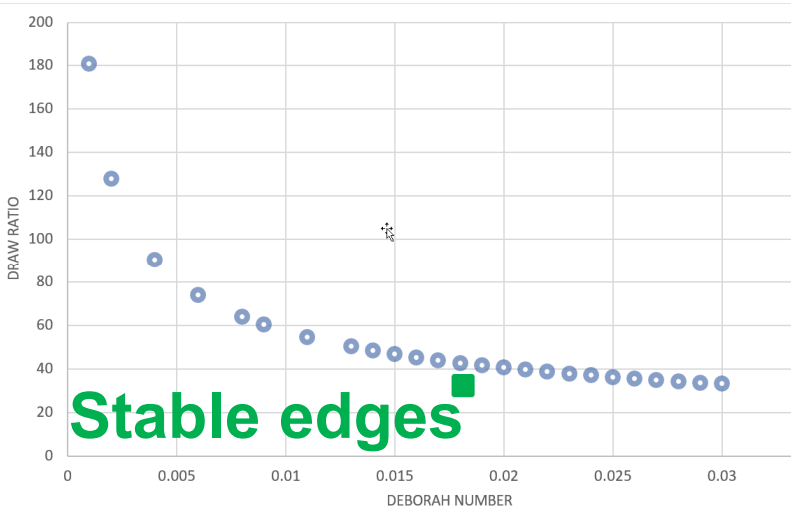


1000 m/min – is it (it is) possible ? (!)

Optimization of machine settings and polymer properties

DEBORAH CALCULATOR FOR LDPE

Input Material Properties:		
Eta0 @ 170°C	[Pas]	
Eta0 @ 150°C	[Pas]	
Eta0 @ 130°C	[Pas]	
G' @ 170°C	[Pa]	
Calculations Material Properties:		
Lambda @ 170°C	[s]	
E0 from 130-150-170°C	[kJ/mol]	
Machine & Process Settings:		
Die gap opening	[mm]	
Coating thickness	[gsm]	**12**
Air gap	[mm]	
Film temperature	[°C]	
Line speed	[m/min]	**1000**
Lambda	[s]	
E0	[kJ/mol]	
Dr	[-]	*32.1*
De	[-]	*0.018*



Summary

Future vision !

Polymer suppliers having **capability to control**
molecular weight (**Eta0**),
molecular weight distribution (**G'**) and
long chain branching (**E0**) of LDPE
enabling **converter** to secure an **efficient** and **stable extrusion coating operation.**

THANK YOU !

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