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The Importance of How Online Rheometers Accurately Indicate Melt Flow Rate in an Extruder

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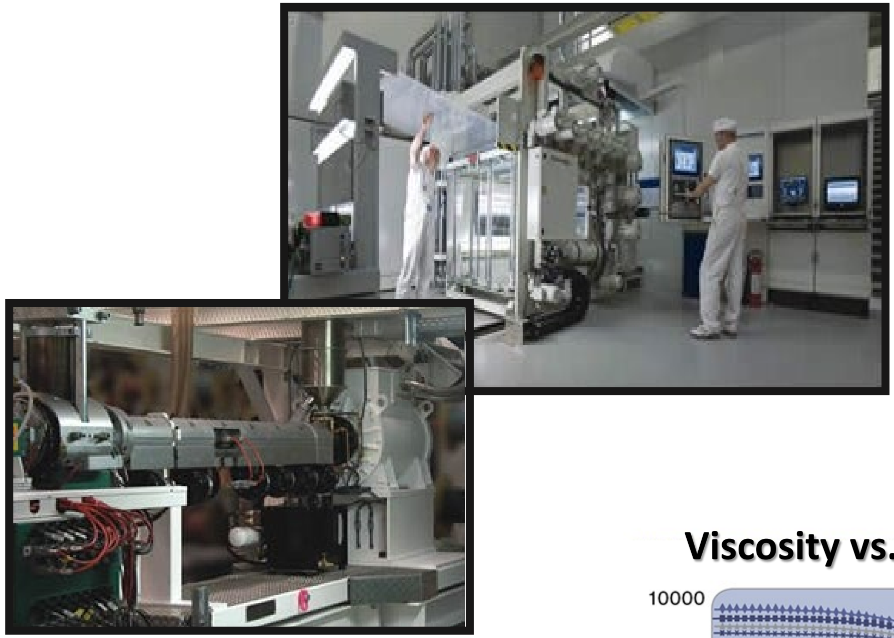
Outline

- Application and benefits of online rheology measurements in plastics processing industry
- ViscoIndicator online rheometer design
- Rheological calculations for correlation of capillary rheometer and melt flow rate (MFR) tester in the online rheometer
- Experimental trials and results from online rheometer

Necessity of Rheological Measurements in Plastics processing

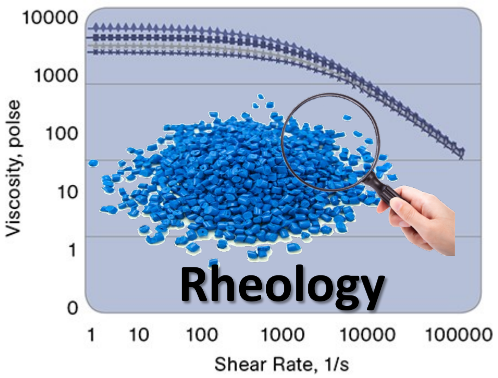


https://www.ptonline.com/articles/high-speed-extrusion

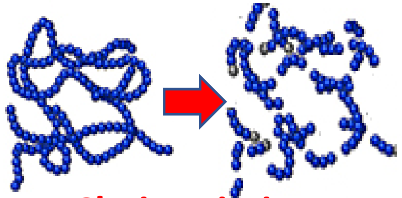
https://www.thoughtco.com/plastics-polymers-science-fair-project-ideas

http://www.ilcaffe.tv/articolo

Viscosity vs. shear rate



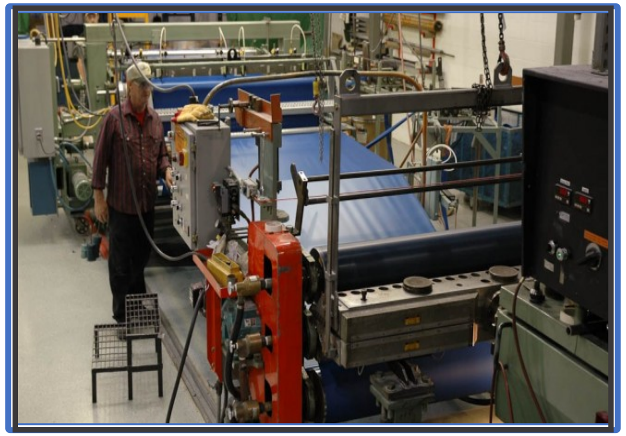
https://www.ptonline.com



Chain scission

https://www.victrex.com

Online Rheometer Applications



- ❖ Continuous data stream of rheological properties at various processing condition
- ❖ Using for automatic processing, product development, quality and process control
- ❖ Incorporating in variety of extrusion lines

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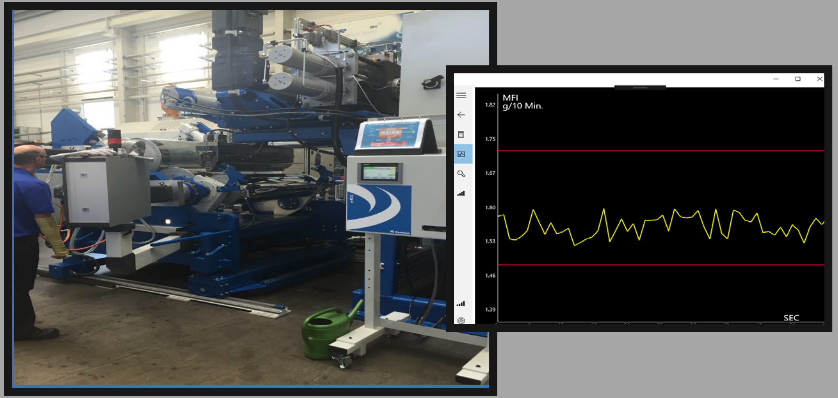
Benefits of Using an Online Rheometer

Window into the Process



In-Process Product Quality Control

Sustainability & Reduce of Waste Stream

Process Productivity & Efficiency

OBJECTIVE
Indication of Rheological Properties in Extrusion

ASTM D1238
ISO 1133

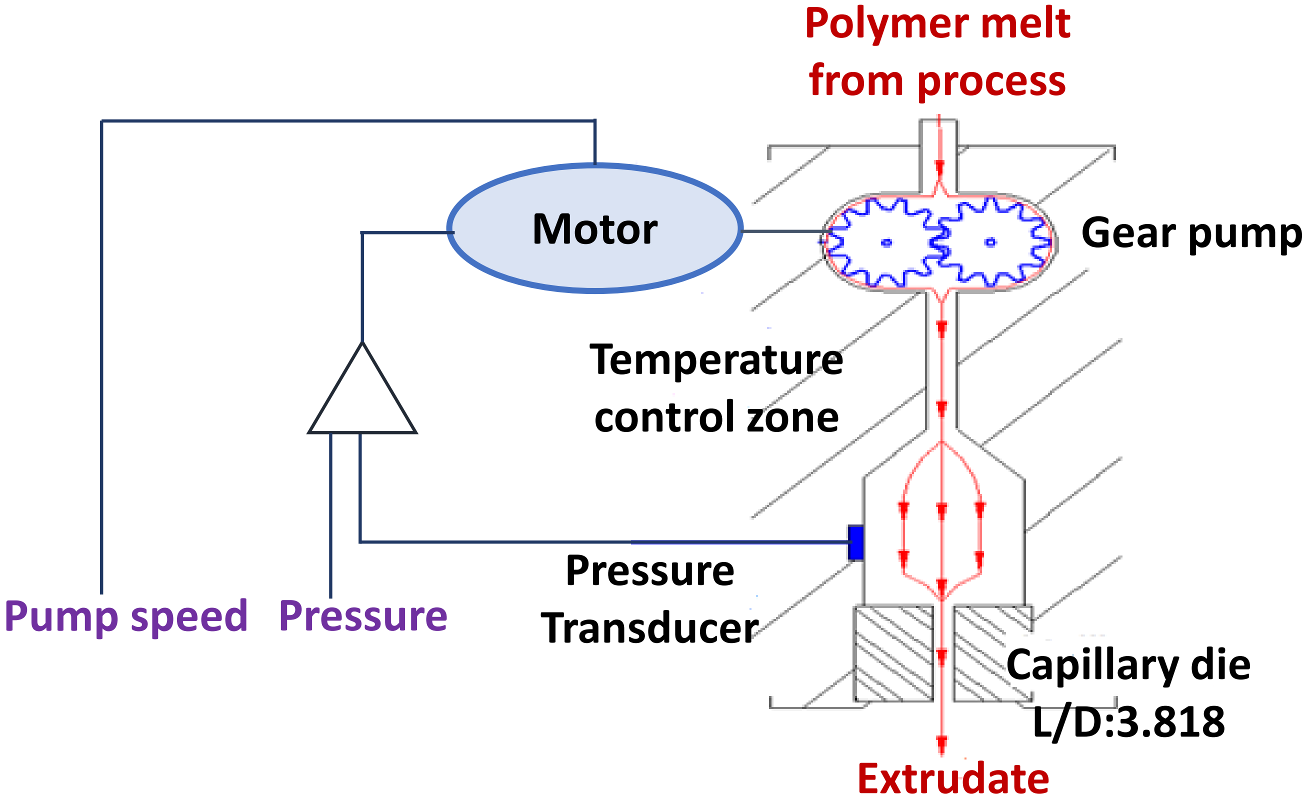
Capillary rheometer

Melt flow rate tester

Online Measurements in an extrusion

- **Controlled shear rate** with speed set point for **apparent viscosity (η_a)** measurements
- **Controlled shear stress** with pressure set point for **melt (volume) flow rate (MFR & MVR)** measurements

ViscoIndicator Online Rheometer Design



- **Speed Mode:**
  maintaining a pre-set speed by manipulating pressure to calculate η_a
- **Pressure Mode:**
  maintaining a pre-set pressure by manipulating gear pump speed to calculate *MFR*

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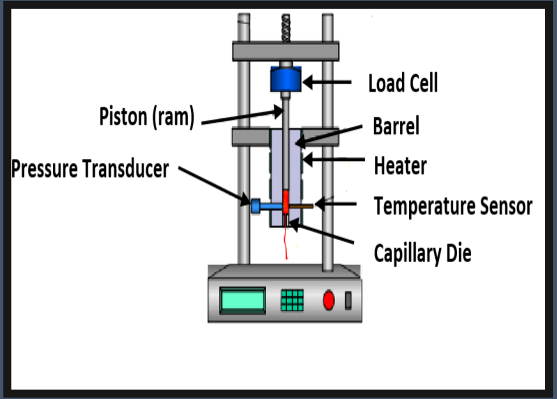
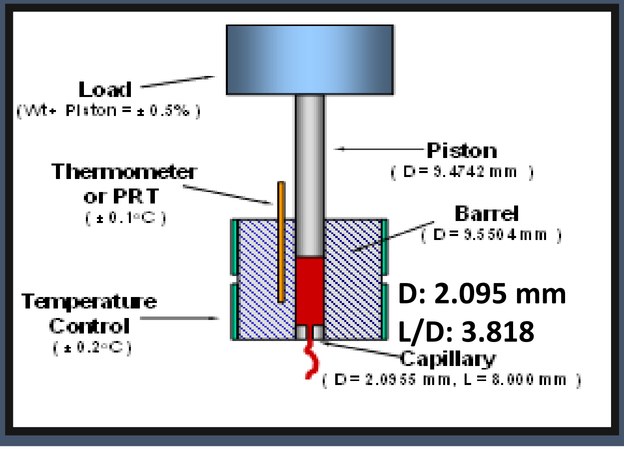
Rheological Calculation from Capillary Rheometer to Online Rheometer

Capillary rheometer	On-line rheometer
	**System running under rate control** 
❖ $\tau_z(r) = \frac{r}{2}\left(\frac{dP}{dz}\right)$ **Wall shear stress** (Pa): $\tau_w = \frac{P}{4(L/D)_{die}}$ ➢ P: Capillary Pressure (Pa) ❖ **Apparent viscosity** $(Pa-s)$: $\eta_a = \tau_w/\dot{\gamma}_a$	❖ $\dot{\gamma} = \frac{du}{dr}, u_z(r) = \frac{2Q}{\pi R^2}\left[1 - \left(\frac{r}{R}\right)^2\right]$ **Apparent shear rate** $(1/s)$: $\dot{\gamma}_a = \left.\frac{du_z}{dr}\right\|_{r=R} = \frac{-4Q}{\pi R_C^3}$ ➢ R_C: Capillary radius (mm) ➢ Q: Volumetric flow rate (mm^3/s)
❖ $Q = S\left(\frac{\pi D_b^2}{4}\right)$ ➢ S: Piston speed (mm/min) ➢ D_b: Barrel diameter (mm)	❖ $Q = S \times V$ ➢ S: Gear pump speed (rpm) ➢ V: Pump volume (mm^3/rev)

Assumptions: ***1:*** *Fully developed, isothermal, steady state, and Laminar Flow -* ***2:*** *No radial/circumferential velocity*
3: *Incompressible fluid -* ***4:*** *No slip at the wall of the die*

Dynisco

Rheological Calculation from MFR Tester to Online Rheometer

Melt flow rate tester	On-line rheometer
	System running under pressure control 

ASTM D1238/ISO 1133 – Test Method B (Based on melt volumetric displacement)

- ❖ **Melt flow rate** $\left({}^{g}/_{10\ min}\right)$: $\boldsymbol{MFR = MVR \times \rho_m}$
  - ➢ MVR: Melt volume-flow rate $\left({}^{cm^3}/_{10\ min}\right)$
  - ➢ ρ_m: Polymer melt density at test temperature $\left({}^{g}/_{cm^3}\right)$

Melt flow rate tester	On-line rheometer
❖ $\boldsymbol{MVR\left({}^{cm^3}/_{10min}\right) = \frac{10(\pi R^2 L)}{t_B}}$	❖ $\boldsymbol{MVR\left({}^{cm^3}/_{10min}\right) = 10(SV)}$
➢ L: Piston travel distance (cm) over time t_B	➢ S: Gear pump speed (rpm)
➢ R: Barrel radius	➢ V: Pump volume $\left({}^{cm^3}/_{rev}\right)$

CHALLENGE !
Correlation of Online Rheometer to Standard Condition in MFR Tester

$$MFR_{Online\ rheometer} = MVR \times \rho_m$$

$$MFR_{Online\ rheometer} = 10(VS\rho_m)\ (\frac{D^3_{Standard}}{D^3})\ \boldsymbol{a_T}$$

Standardized melt flow rate tester (ASTM D1238)	CORRELATION	Online rheometer
M: Standard weight (kg) D_b: Barrel diameter$(9.55\ mm)$	$\boldsymbol{Pump\ Pressure = \frac{M \times 9.8}{\pi D_b^2}}$ e.g. M: 2.16 kg ⟶ P: 43.38 psi	Pump pressure
D_{ASTM}: $2.095\ mm$ $(^L/_D)_{ASTM}$: 3.819	***Die dimeter correlation***	D: Diameter $^L/_D$: 3.819
Standard Temperature	$\boldsymbol{Temperature\ correlation\ (a_T)}$ ***Challenge: Different temperatures***	***Process Temperature***

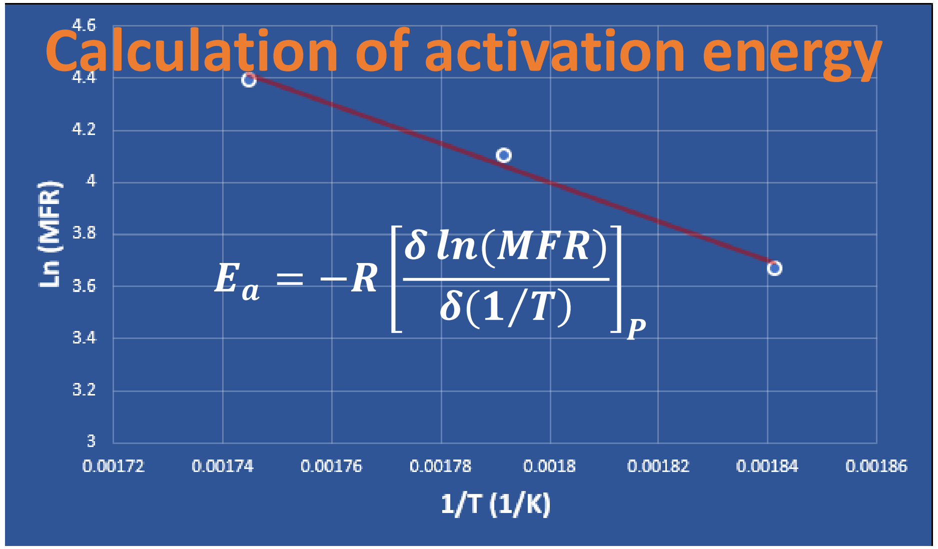
Arrhenius relationship for Temperature correlation

$$MFR_{Standard} = (MFR_0)e^{\frac{-E_a}{R}\left(\frac{1}{T_{Standard}}-\frac{1}{T_0}\right)}$$

$(T > T_g + 100)$

where

- E_a: Flow activation energy $({}^{kJ}/_{mol})$
- R: Universal gas constant $(8.314 \times 10^{-3}\ {}^{kJ}/_{mol.K})$
- $T_{Standard}$: Standard temperature (K)
- T_0: Processing temperature (K)



Experimental

Material

- Polypropylene (PP)
- manufactured by BRASKEM S.A
- MFR (2.16 kg/230 °C): 35.5 g/10min

Off-line measurements

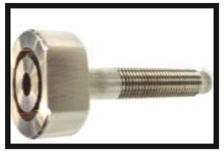
- Dynisco® LMI5000 melt flow rate tester
- Test method: Method B (2.16 kg/230 °C)
- Flag length: 6.35 mm
- Die dimensions: L/D: 3.818, D: 2.095 mm

Process

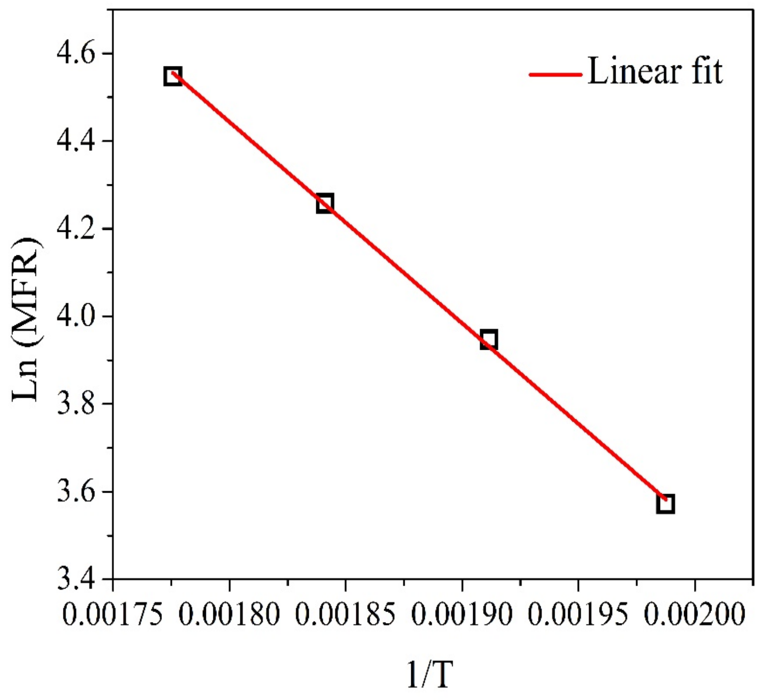
- Dynisco® REX single screw extruder
- Barrel L/D: 20:1
- Head pressure: 4.1 MPa
- Barrel temperatures: 230 °C, 250 °C, 270 °C, and 290 °C
- Collecting about 40 g of extrudates at each temperature

On-line measurements

- Dynisco® ViscoIndicator Rheometer
- 90 degrees heated adaptor
- ½-20 mounting port
- Pump pressure: 43.38 psi (pressure mode)
- Dynisco® Vertex Mercury pressure transducer



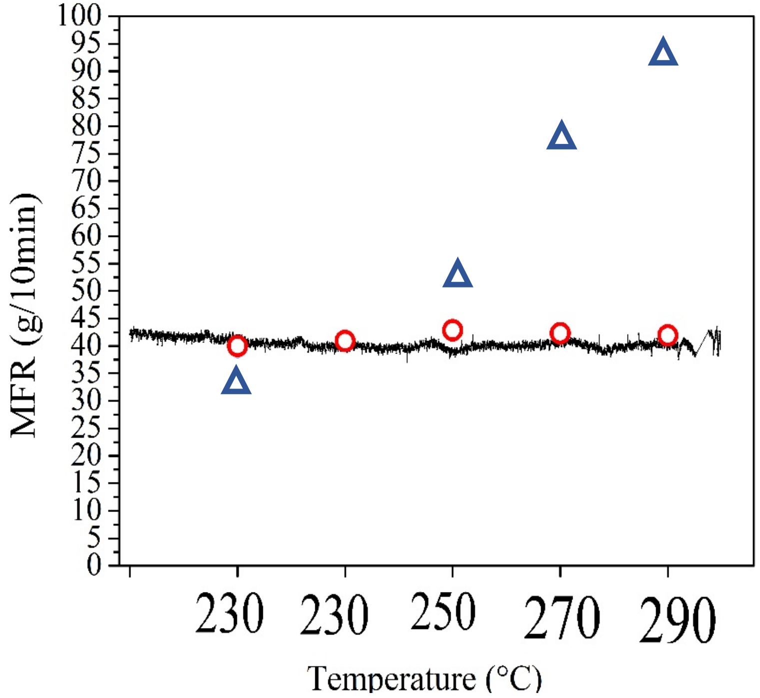
Calculation of Activation Energy

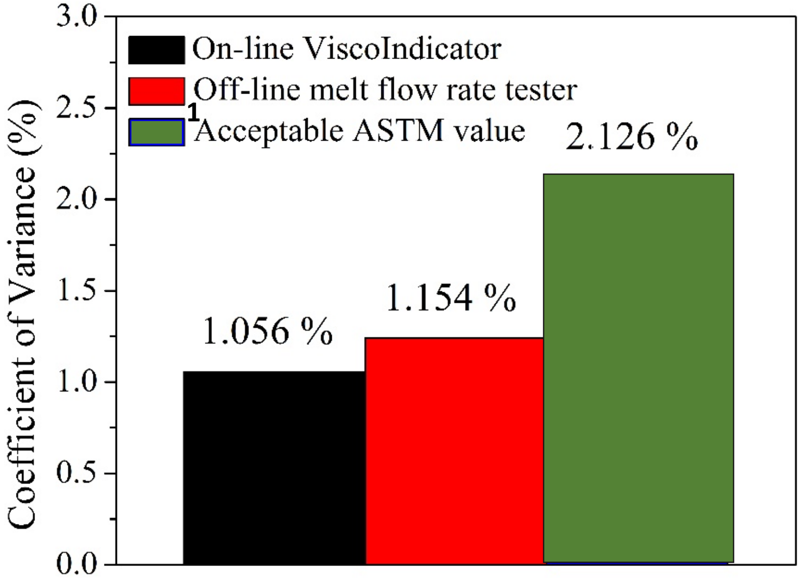


$$\ln(MFR) = \frac{-E_a}{R}\left(\frac{1}{T}\right) + \ln(A)$$

Linear fitting equation: y = a+b*x	
Adj. R-Square: 0.99	
Slope (b = -Ea/R)	-4595.4 ($\pm$92.21)
Activation energy (kJ/mol)	38.2

Comparison of Online Measurements and Laboratory MFR Measurements





[1] Harban, A. A., McClamery, R. M. (1963) "Limitations on Measuring Melt Flow Rates of Polyethylene and Ethylene Copolymers by Extrusion Plastometer," *Materials Research and Standards* 3: pp. 906.

— Online rheometer at various processing temperatures

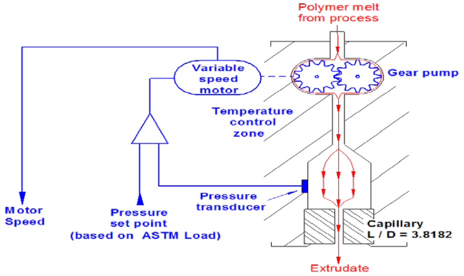
o Offline MFR test at standard testing condition on extrudates collected at each processing temperature

Δ **Offline MFR test on raw PP at various testing temperatures**

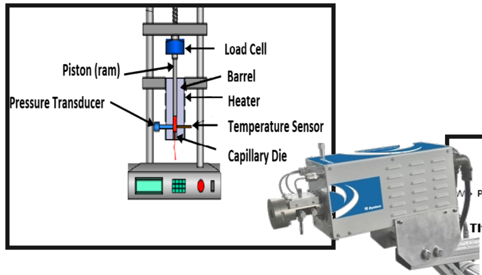
Conclusion

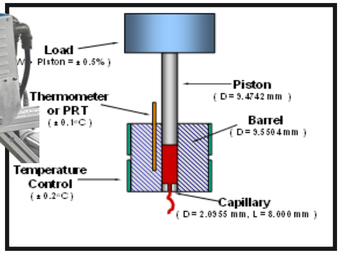
- Application and benefits of online rheology measurements

- ViscoIndicator online rheometer design

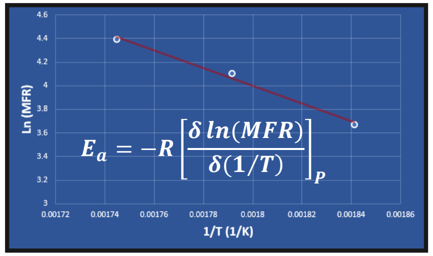


- Rheological calculations from capillary rheometer (constant rate) and MFR tester (constant stress) to online rheometer

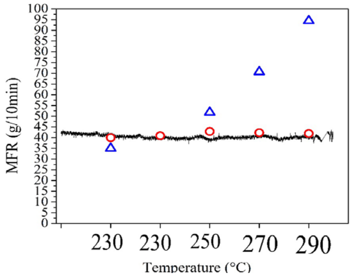




- Temperature correlation and calculation of activation energy



- Good agreement between online and offline MFR measurements at standard conditions




"Everything Flows"-Heraclitus
https://www.beautifulworld.com/north-america/united-states/the-wave/
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Thank You !