

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

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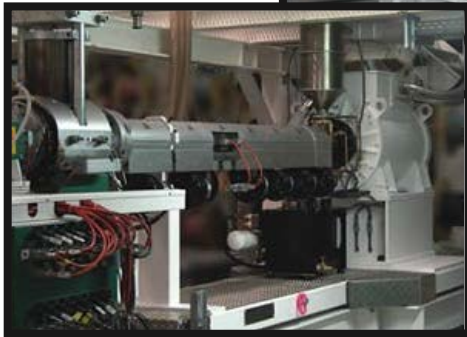
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- ❖ Application and benefits of online rheology measurements in plastics processing industry
- ❖ ViscoIndicator online rheometer design
- ❖ Rheological calculations for correlation of capillary rheometer and melt flow rate (MFR) tester in the online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

Necessity of Rheological Measurements in Plastics processing



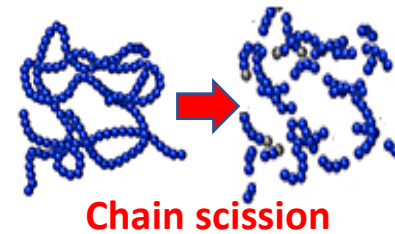
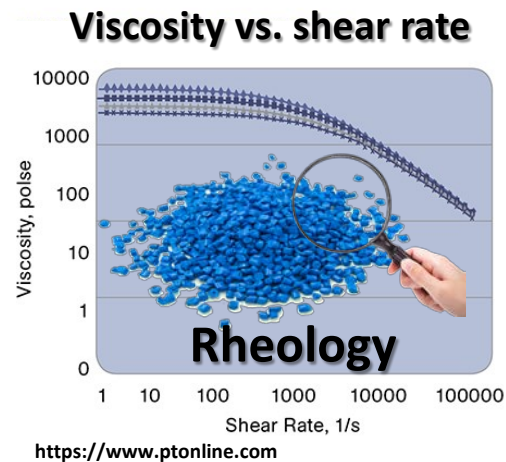
<https://www.ptonline.com/articles/high-speed-extrusion>



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



<http://www.ilcaffe.tv/articolo>



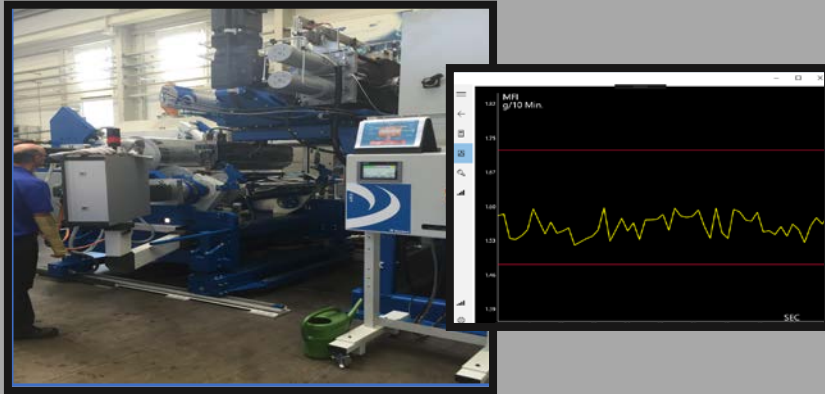
<https://www.victrex.com>



- ❖ 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

SPE ANTEC ORLANDO18 Benefits of Using an Online Rheometer

Window into the Process



In-Process Product Quality Control



<http://www.ebay.com/bhp/plastic-pellets>

Sustainability & Reduce of Waste Stream



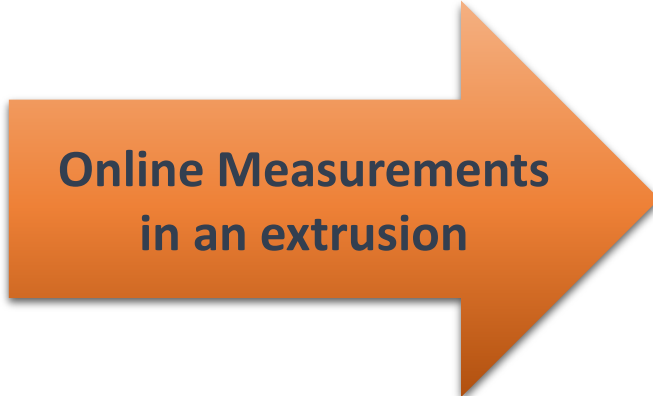
<https://dir.indiamart.com/impacat/plastic-scrap.html>

<http://regainpolymers.com>

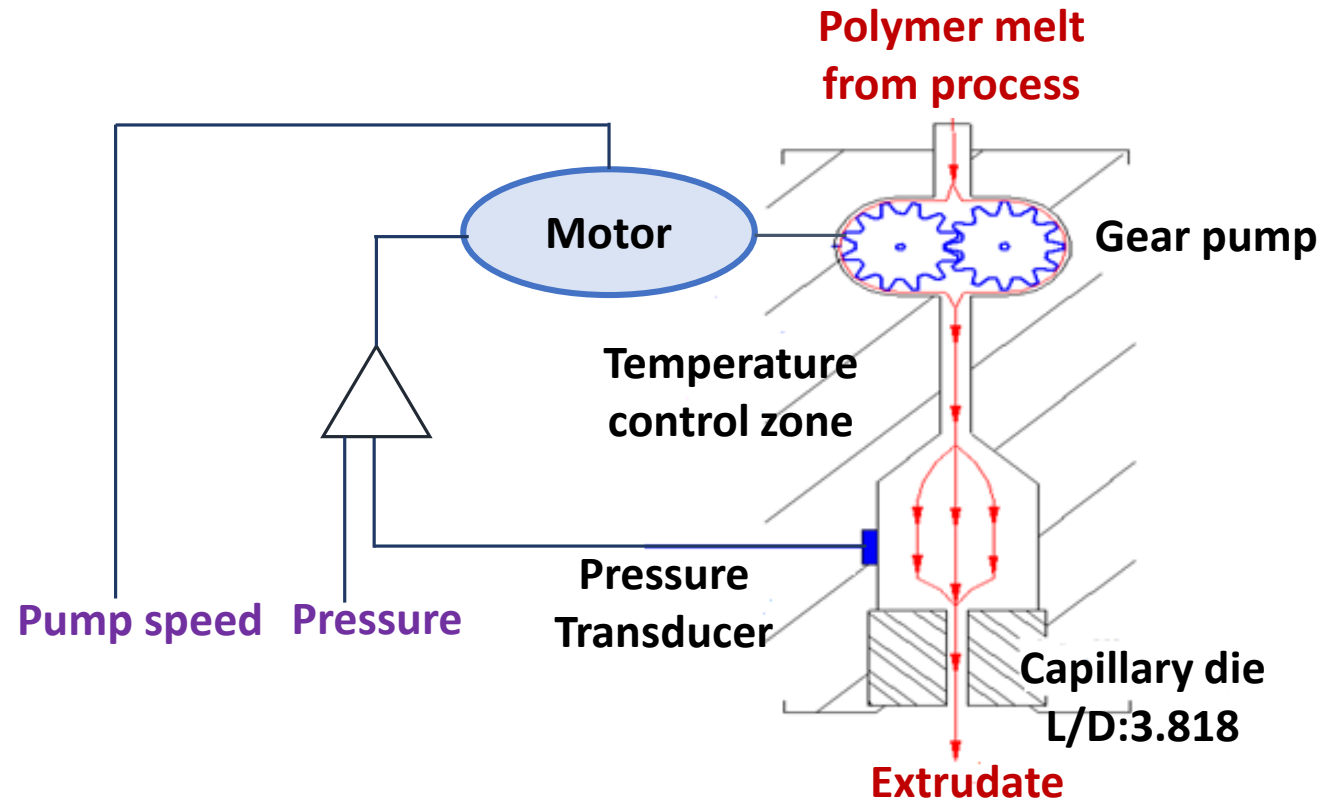
Process Productivity & Efficiency



<https://www.dreamstime.com/>

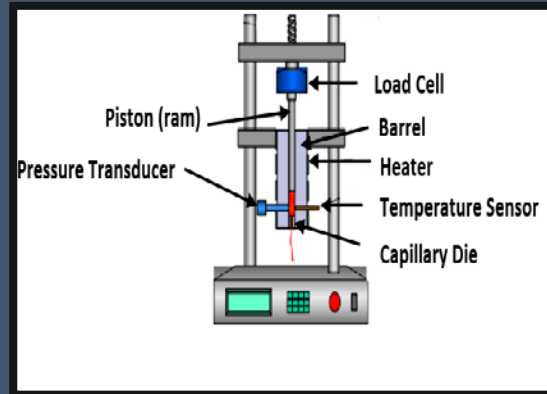


- Controlled shear rate with speed set point for apparent viscosity (η_a) measurements
- Controlled shear stress with pressure set point for melt (volume) flow rate (*MFR* & *MVR*) measurements



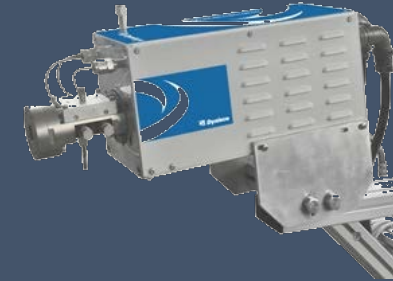
- **Speed Mode:**
maintaining a pre-set speed by manipulating pressure to calculate η_a
- **Pressure Mode:**
maintaining a pre-set pressure by manipulating gear pump speed to calculate MFR

Capillary rheometer



On-line rheometer

System running under rate control



$$\diamond \tau_z(r) = \frac{r}{2} \left(\frac{dP}{dz} \right)$$

Wall shear stress (Pa): $\tau_w = \frac{P}{4(L/D)_{die}}$

➤ P: Capillary Pressure (Pa)

$$\diamond \text{Apparent viscosity (Pa} \cdot \text{s): } \eta_a = \tau_w / \dot{\gamma}_a$$

$$\diamond Q = S \left(\frac{\pi D_b^2}{4} \right)$$

➤ S: Piston speed (mm/min)

➤ D_b: Barrel diameter (mm)

$$\diamond \dot{\gamma} = \frac{du}{dr}, u_z(r) = \frac{2Q}{\pi R^2} \left[1 - \left(\frac{r}{R} \right)^2 \right]$$

Apparent shear rate (1/s): $\dot{\gamma}_a = \left. \frac{du_z}{dr} \right|_{r=R} = \frac{-4Q}{\pi R^3}$

➤ R_C: Capillary radius (mm)

➤ Q: Volumetric flow rate (mm³/s)

$$\diamond Q = S \times V$$

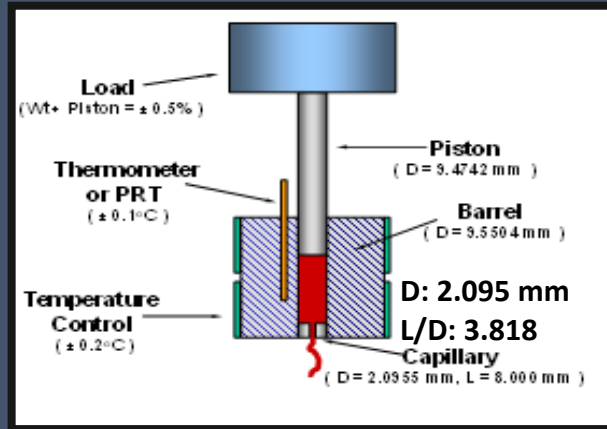
➤ S: Gear pump speed (rpm)

➤ V: Pump volume (mm³/rev)

Assumptions: 1: Fully developed, isothermal, steady state, and Laminar Flow - 2: No radial/circumferential velocity

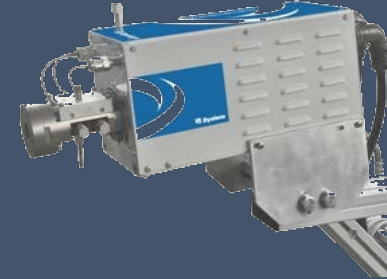
3: Incompressible fluid - 4: No slip at the wall of the die

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 ($g/10\text{ min}$): $MFR = MVR \times \rho_m$

- MVR : Melt volume-flow rate ($cm^3/10\text{ min}$)
- ρ_m : Polymer melt density at test temperature (g/cm^3)

$$\text{❖ } MVR (cm^3/10min) = \frac{10(\pi R^2 L)}{t_B}$$

- L : Piston travel distance (cm) over time t_B
- R : Barrel radius

$$\text{❖ } MVR (cm^3/10min) = 10(SV)$$

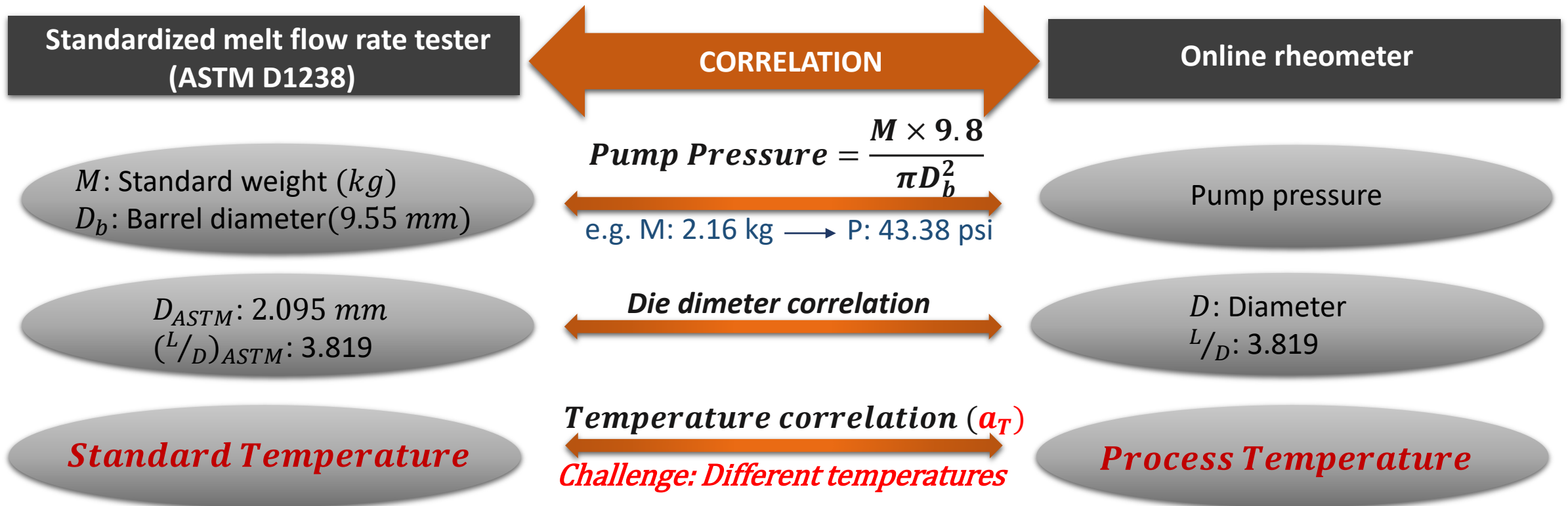
- S : Gear pump speed (rpm)
- V : Pump volume (cm^3/rev)

CHALLENGE !

Correlation of Online Rheometer to Standard Condition in MFR Tester

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

$$MFR_{Online\ rheometer} = 10(VS\rho_m) \left(\frac{D_{Standard}^3}{D^3}\right) a_T$$

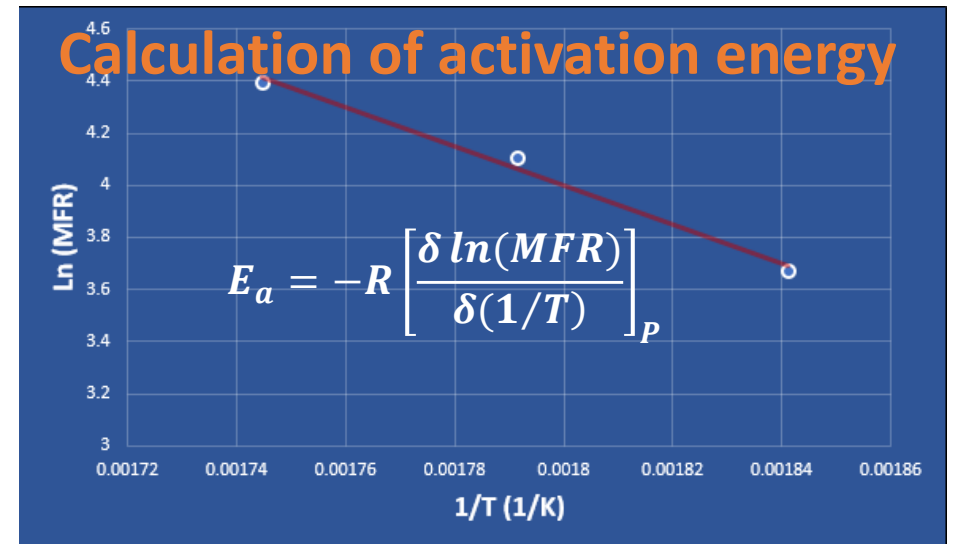


$$MFR_{Standard} = (MFR_0) e^{\frac{-E_a}{R} \left(\frac{1}{T_{Standard}} - \frac{1}{T_0} \right)}$$

$(T > T_g + 100)$

where

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





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

Human Machine Interface (HMI)

Rheological Sensing Unit (RSU)

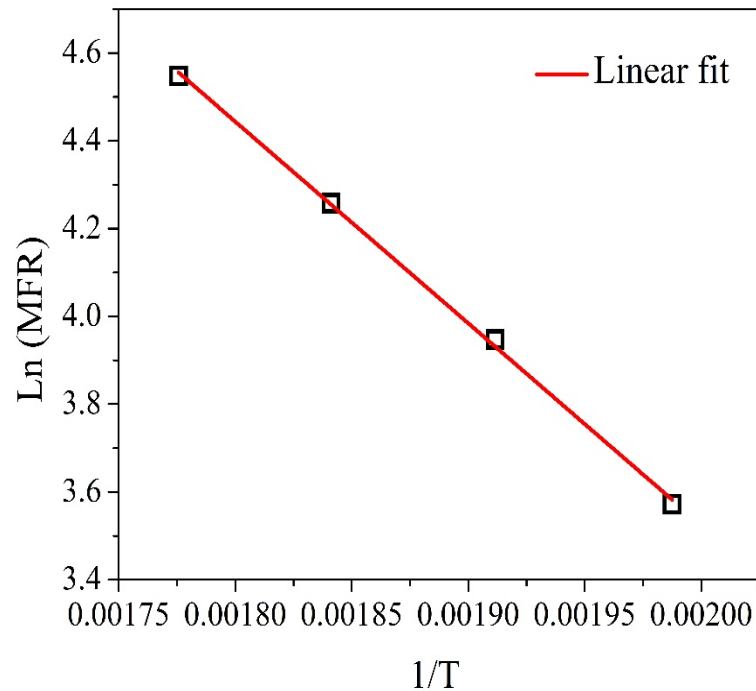
Rheological Control Unit (RCU)



On-line measurements

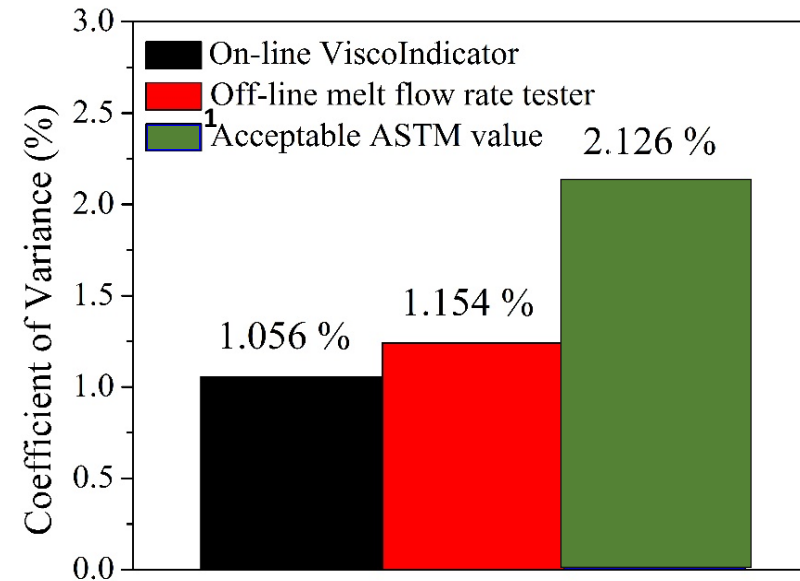
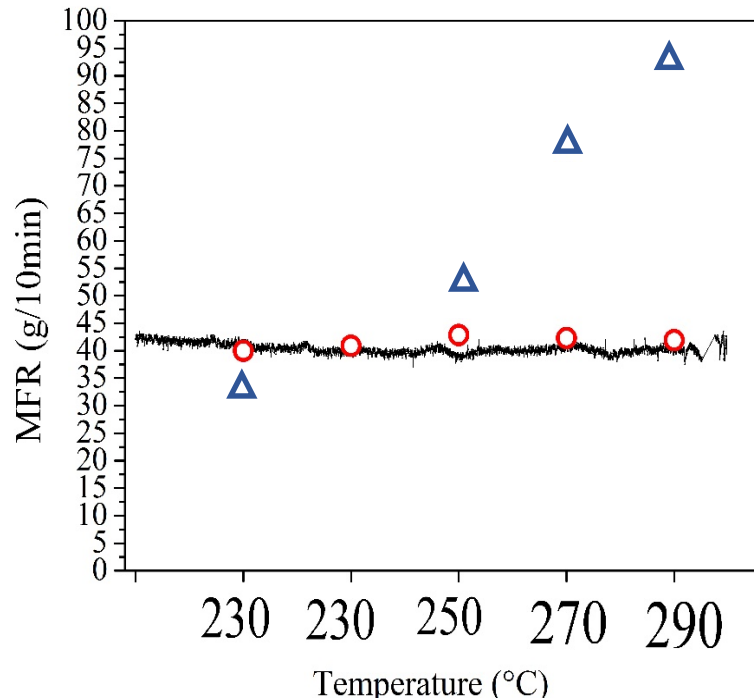
- ❖ Dynisco® ViscoIndicator Rheometer
- ❖ 90 degrees heated adaptor
- ❖ ½-20 mounting port
- ❖ Pump pressure: 43.38 psi (pressure mode)
- ❖ Dynisco® Vertex Mercury pressure transducer





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

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

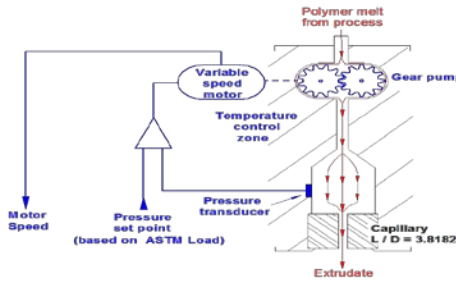


¹ Harban, A. A., McClamery, R. M. (1963) "Limitations on Measuring Melt Flow Rates of Polyethylene and Ethylene Copolymers by Extrusion Plastometer," *Materials Research and Standards* 3: pp. 906.

- Online rheometer at various processing temperatures
- Offline MFR test at standard testing condition on extrudates collected at each processing temperature
- △ Offline MFR test on raw PP at various testing temperatures

❖ Application and benefits of online rheology measurements

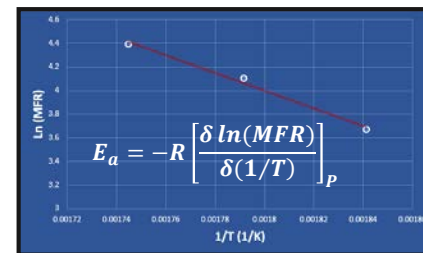
❖ ViscoIndicator online rheometer design



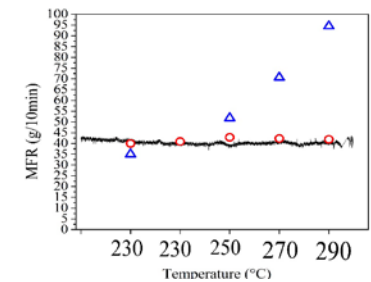
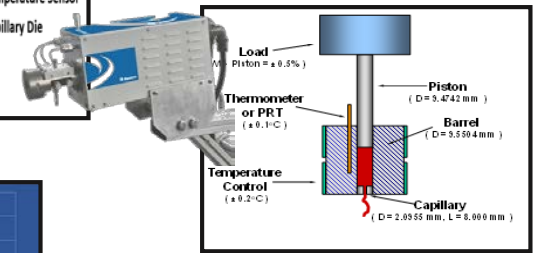
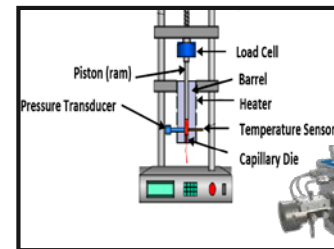
❖ Rheological calculations from capillary rheometer (constant rate) and MFR tester (constant stress) to online rheometer

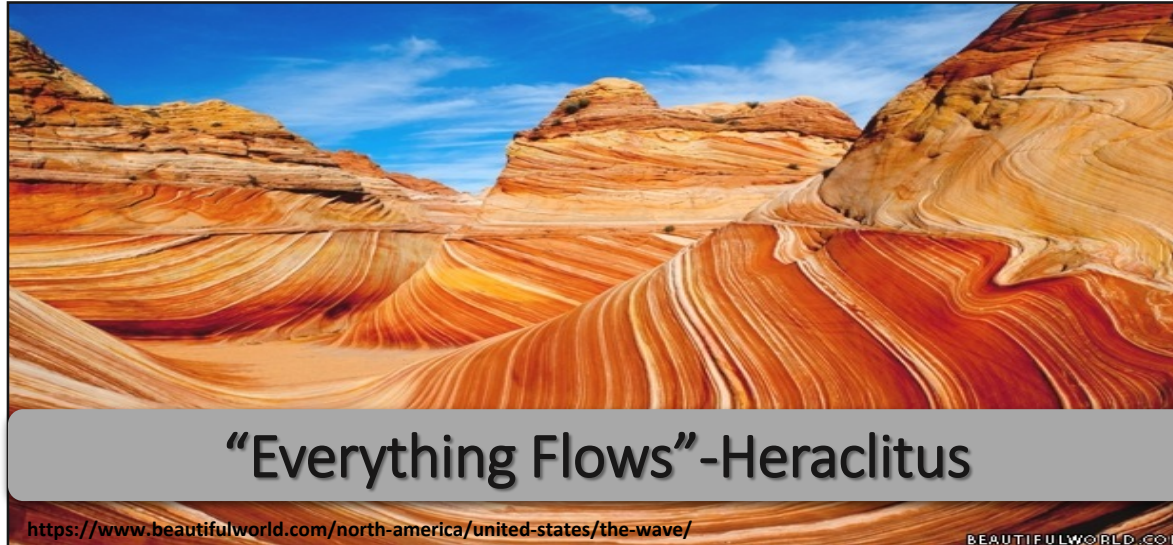


❖ Temperature correlation and calculation of activation energy



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





Thank You !