PolyLab System

Torque Rheometer:

RheoDrive
Rubber compound formulation

- Rubber: 55%
- Carbon black: 35%
- Activators: 1%
- Antioxidants: 1%
- Oil: 5%
- Sulphur: 2%
- Accelerators: 1%
Polymers are processed in a liquid state: (Extrusion, injection moulding)

The flowability (viscosity) will influence:
Processability
Quality of the final product

Torque Rheometer and its' sensors are:
Down-scaled production machines
(mixers, extruders, dies, postex)

Test procedure similar to production
Who uses Torque Rheometers?

**Application: Material**
- In R+D: for new polymers, small amount of polymers
- In R+D: for new compound developments, polymer alloys
- In R+D for new additives stabilisers, pigments, fillers, lubricants, etc.
- In pilot plants to study formulation and processing characteristics
- In Q.C to control incoming and outgoing materials ISO 9000

**Application: Process**
- In R+D for new machine design to evaluate production process
- In pilot plants to determine optimum process conditions
BASE UNITS faster, higher torque

RheoDrive
RheoDrive + Mixer/Extruder Sensor

= PolyLab OS

RheoDrive

Mixer sensors

Single screw extruder sensors

Twin screw extruder sensor

additional analyzing sensors

Rotors

Feeding systems

Screws

Dies

Postex
PolyLab OS System Components

- HAAKE RheoDrive 4 & 16 kW Drive Unit
- HAAKE PolySoft Control & Application Software
- HAAKE Rheomix Mixer
- HAAKE Rheomex Extruder
- HAAKE PTW 16/24 Parallel Twin Screw Extruder
- HAAKE CTW Conical Twin Screw Extruder
- HAAKE Dies
- HAAKE Postex
- HAAKE Ancillaries
- HAAKE Analyzing Sensors (viscosity, visco-elasticity, spectroscopy)
PolyLab OS

- **RheoDrive**: torque, speed, energy
- **Feeder**: volume, weight
- **Mixer-Extruder**: temperature, frictional heat
- **Die**: dynamic viscosity, MFI / MVI
- **Postex**: conductivity, % additive, dispersity, Xtens viscosity
- **Grinder**: dispersity, film quality, film strength
PolyLab OS – New Design
PolyLab OS – New Design
PolyLab OS – New Design
PolyLab OS - Benefits

"OS" = Open Solutions
- integration of new sensors and systems with CAN bus
  (viscosity, spectroscopy, optical, …)
- open solutions for all the applications
  (expand system with additional sensors)
- open for the future (standardized interfaces, serviceability)
- new sensors for combined measurements or methods
  (viscosity, optic, analytical tests)
- needs only one instrument for polymer testing

plug and measure
- system with user compatibility of components, reliability, ease of use
- connect all our offerings at site, no factory installation required
- connect to PC, network or PDA with TCP/IP (cable-wireless) or USB
PolyLab OS - Benefits

- time saving software
  - software suite for running the complete PolyLab OS platform
- time saving software with job stream option
  (automation of tests, ease of use, unsupervised running)
- serviceability
  - modular in design for service
  - remote diagnostics, exchange of components, minimum downtime
  - remote supervision from the office with browser/PDA monitoring
    alarms/events/data
- traceability of experimental results
  - traceability of experimental results and FDA compliance (21CFR11)
  - prepared for FDA regulations in design and materials used
New Features with PolyLab Hardware

**System:**
Modular system for mixer, single screw extruder, or twin screw extruder to be connected to a single drive unit.

Open System to connect and integrate different components

**RheoDrive:**
New drive concept with intelligent 4 kW & 16 kW motors.

Very smooth and silent running drive system specifically selected for improved mixer test results.

**Docking station:**
New mechanical mechanism to look the docked systems in place; easy to use and safe.

Only one central socket/connector to eliminate cables and wires hanging all over.

Rigid and stable chassis on rolls to travel to different locations in a pilot plant.
**New Features with PolyLab Hardware**

**CAN Open Bus:**
Intelligent CAN bus integrates all peripherals with self-identification and transfer of characteristic data like calibration data table.
Adaptation of all standard CAN open components (thermocouple, pressure sensor, additional sensors) without software modification.
Integration of downstream equipment with standardized interface.

**Connectivity:**
Simple plug & play computer connection with USB or TCP/IP (option)
Remote controller (cable based) to operate the drive manually
Remote diagnose and status info with Web browser or PDA
PolyLab OS – Central Coupling

- Central coupling for docking Mixers and Extruders
- Connecting the communication and the power supply
PolyLab OS – Remote Control

Control PolyLab OS where it is suitable for you

Optimise the process on site
Never loose visual control on your process
3 torque measurement options:

1) Via motor power
2) Standard torque sensor
3) Multi range torque sensor

<table>
<thead>
<tr>
<th>Torque Measurement</th>
<th>Accuracy</th>
<th>Resolution</th>
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</thead>
<tbody>
<tr>
<td>no sensor (motor power)</td>
<td>+/- 5 % (FSD)</td>
<td>1 Nm</td>
</tr>
<tr>
<td>CANopen torque sensor</td>
<td>+/- 0.15 % (FSD)</td>
<td>0.01 Nm</td>
</tr>
</tbody>
</table>
The PolyLab torque sensor in-between motor shaft and coupling
Multi Range Torque Sensor 400Nm / 120Nm

- Strain gauge principle
- Dynamical Resolution Enhancement
  ≤ 60 Nm : 0.01 Nm
  > 60 Nm : 0.1 Nm
- “This is two torque Sensors”
- Part No.: 567-0101
- The PolyLab Torque Sensors has non contact (no brushes, no collector ring) digital data transmission from shaft to casing. This means no failure of transmission and maintenance free.

- high accuracy: 0.15 % FSD
- high reproducibility: 0.02 %
Strain gauges – operation principle

a) Change of electrical resistance

b) Principle of strain gauge (horizontal stress)

c) Array of 4 single strain gauges affixed with 90° misalignment (Wheatstone Bridge: The changes of strain resulting from the resistance change are detected. These are proportional to the applied loads)
Two software layers:

1) **PolyLab Monitor OS:**
   - Basic software to control and monitor the PolyLab OS
   - Visualization of the attached equipment and sensors
   - Online data export in Excel (XP)
   - No data evaluation

2) **PolySoft Modules:**
   - Application dedicated software modules
     (Mixer Test / Capillary-Rheology / Filter Test / Advanced Extrusion)
   - Job stream structure, incl. data evaluation (similar to Haake RheoWin)
   - The user is guided through the measurement
   - Different user levels
PolyLab Monitor OS

- Plug & Measure
- easy Data monitoring
- Scaleable windows for measured values
- Visualized process values
- online export data to MS Excel (XP)
- Online sensor recognition (“hot plugging”)

**Example Screen:**

- TM_1 Mix: 57.6 [°C]
- M: 2.2 [Nm]
PolyLab Monitor OS

- Graphic monitoring of instrument and test values
- Scaleable windows for measured values
PolyLab Monitor OS

- Heating/Cooling visualization
- Monitor the regulating values (in %) of each control zone
- Get important information for extruder screw-configuration
• On-Screen Remote Control to keep control of the PolyLab even when using other Windows-based software
PolyLab OS – PolySoft Mixer

• PolySoft Mixer SW:
  - Job stream structure analog Haake RheoWin
  - The user is guided through the measurement
  - Project handling: several tests can be saved in a project
PolyLab OS – PolySoft Mixer

- Protocol Element:
  - Documentation of sample information
PolyLab OS – PolySoft Mixer

- **Device Setup:**
  - Automatic recognition of mixer setup
  - Rotor selection
• **Measurement Element:**
  - Setting of the testing parameters
  - Calculation of the sample-weight
  - Programming of sophisticated test steps
PolyLab OS – PolySoft Mixer

- Evaluation Element:
  - Selection of evaluation routine for the determination of the curve maxima, minima and gradients
  - Modification of the evaluation routine
PolyLab OS – PolySoft Mixer

- Post Element:
  - Generation of file name
  - Definition of printouts
PolyLab OS – PolySoft Mixer

- Export Element:
  - Definition of data export
PolyLab OS – PolySoft Mixer

• Statistic function:
  - Statistical evaluation of measurement data
  - Comparison of sample behavior over a longer time period
• **Step choose:**

- Speed-steps from low to high, as well from high to low possible

- Speed-steps from low to high and from high to low possible in one measurement possible
• **Step settings:**

- Adjustments for each step possible

- Possibility for longer step-time at low shear-rates, to get more accurate data

- Possibility for short step-time at high shear-rates, to save time & sample
PolyLab OS – PolySoft Job Controller

148.6 bar

Job-Control

Mode: automatic

Press ... value

State: (6,6) n: 170.0 l/min waiting for equilibrium

Thermo Fisher Scientific

Thermo Scientific
PolyLab OS – PolySoft Regression
PolyLab OS – PolySoft Regression

Flowcurve

\[ \tau_{\text{app}} = f(\dot{\gamma}_{\text{app}}) \]

\[ \eta_{\text{app}} = f(\dot{\gamma}_{\text{app}}) \]

Carreau
Tests with the PolyLab OS

PolyLab OS

relative Measurements

Mixer tests

Extruder tests

Other CAN Sensors

absolute Measurements

rheological Measurements

Single-screw extruders

Twin-screw extruders

Rod Capillary tests

Slit Capillary tests
Laboratory Mixers:
Laboratory Mixers - measuring principle

Shearing of a test sample in a heated mixing chamber with counter rotating rotors

Test results:
- Torque
- Melt temperature
Electrically heated Rheomix mixer (principle sketch)

1 Back Section
2 Centre Bowl
3 Frontplate
4 Rotor Shafts
5 Bushings
6 Rotors
7 Melt Thermocouple
8 Control Thermocouple
9 Ram
Rotors for Rheomix mixers
### Rotors and Applications

<table>
<thead>
<tr>
<th>Roller Rotors</th>
<th>Mixing of thermoplastics, e.g. Polyolefines, PVC, engineering plastics</th>
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<tbody>
<tr>
<td>Banbury Rotors</td>
<td>Mixing of elastomers, but also for compounding of powder into thermoplastic material. Very common in rubber technology</td>
</tr>
<tr>
<td>Rotors and Applications</td>
<td></td>
</tr>
<tr>
<td>-------------------------</td>
<td></td>
</tr>
</tbody>
</table>
| Delta Rotors | Mixing and crosslinking of Thermosets, used with conical bowl of 540 type mixer  
| Cam Rotors | Thermoplastics, less axial distribution, ceramic compounds, food (sticky, higher torque)  
| Sigma Rotors | Food application and Plastisols limited in torque  

Electrically heated Mixers: Rheomix 600 / 3000

- electrically heated
- 3 heating zones:
  - backplate / centre bowl / frontplate
- temperature range up to 450 °C
- possibility to run temperature programs
- removable rotor blades:
  - Roller Rotors
  - Cam Rotors
  - Banbury Rotors
  - Sigma Rotors
- loading ram for manual or pneumatic operation
Liquid heated Mixers: Rheomix 610 / 3010

- liquid heated for lower temperatures (i.e. mixertests with rubber)
- 3 heating zones:
  - backplate / centre bowl / frontplate
- temperature range below 200°C
- removable rotor blades:
  - Roller Rotors
  - Cam Rotors
  - Banbury Rotors
  - Sigma Rotors
- loading ram for manual or pneumatic operation
Advantage of round feed section

Fr: resulting force, spreading old style mixer bowls

Fm: force created by mixing of the material
Mixer Test Procedure:
Mixer Cleaning:
• Materials:
• Polyolefins:
  (Polyethylene PE
  Polypropene PP)
• Engineering Plastics:
  (PS, PA, PC, PEEK, LCP)
• Point detection:
  • \( L = \text{Loading point} \)
  • \( M = \text{Minimum point} \)
• **Loading Point**
  Detects complete mixer loading.
  Point is only used as time base for further calculations
  Torque value depends on the loading conditions

• **Minimum point**
  Detects lowest melt viscosity
Influence of Stabilizer on Polyamide

Rheomix600, Roller Rotors
Temp.: 280°C, m: 52g, n: 60 rpm

PA6 with Stabilizer
PA6 without Stabilizer
SAN with Carbon Black

Rheomix600, Roller Rotors
Temp.: 230°C, m: 58g, n: 40 rpm

Torque [Nm] vs. Time [min]

SAN & 30% Carbon Black 1

SAN & 30% Carbon Black 2
Materials:
• Polyolefins: PE, PP
• Engineering / high performance Plastics: (PS, PA, PC, PEEK, LCP)
• Compounds of a.m. polymers with organo clays, nano tubes

Point detection:
• $L =$ Loading point
• $M =$ Minimum point (steady state torque)

Observe the melting behaviour and flowability of a compound

“We have noted that a simple indicator of the extent of clay exfoliation within polyethylene is the steady-state torque recorded during melt compounding$^1$.”

$[1]$Ref.: Influence of clay exfoliation on the physical properties of montmorillonite/polyethylene composites
T.G. Gopakumar, J.A. Lee, M. Kontopoulou*, J.S. Parent
Polymer 43 (2002) 5483–5491
The erratic reading in the steps for increased powder load show the limiting powder [%] in the mixture.

Contact free and fast, high resolution torque sensors have a big advantage over old style “dynamometer” where the reaction of a heavy motor is measured, the erratic reading, here key feature is damped or smoothed.

Ref. LR45-e, Joseph A. Krudys, ThermoHaake 2002
- Materials: PVC (rigid)
- Point detection:
  - L = Loading point
  - V = Valley point
    (starting point of melting process)
  - F = Fusion point
    (melting process finished)
  - M = Minimum point
PolySoft Routine evaluation - Curve Type 2

• Loading Peak:
  Shows when mixer is completely filled and closed. Point is used as time base for calculation only. Torque value depends on kind of loading. It can be manually influenced by loading speed and loading force.

• Valley Point:
  Shows when PVC Dryblend starts to melt. Point is significant for compound formulation.

• Fusion Peak:
  Shows when melting process of PVC is finished. Point is significant for compound formulation. Beyond this point torque starts decreasing caused by friction/heat.

• Minimum Point:
  Shows when material has reached the lowest viscosity.
Influence of the sample weight on PVC fusion

Rheomix 600, Roller-Rotors, Temp.: 160°C, n: 40 rpm

- m: 66 g
- m: 64 g
- m: 60 g
Influence of the sample weight on PVC fusion

m: 60 g
Influence of the sample weight on PVC fusion
PolySoft Routine evaluation - Curve Type 3

- **Materials:** PVC (rigid) Stability test
- **Point detection:**
  - L = Loading point
  - V = Valley point  
    (starting point of melting process)
  - F = Fusion point  
    (melting process finished)
  - M = Minimum point
  - O = Onset of degradation
  - D = Degradation
• Loading Peak
  Shows when mixer is completely filled and closed. Point is used as time base for calculation only. Torque value depends on method of loading.

• Valley Point
  Shows when PVC Dryblend starts to melt. Point is significant for compound formulation.

• Fusion Peak
  Shows when melting process of PVC has finished. Point is significant for compound formulation. Beyond this point torque decreases caused by frictional heat.

• Minimum Point
  Shows when material has reached the lowest viscosity.
• Onset of Degradation
  Shows when material starts to degrade.
  Point is significant for processing stability.
  It can be set as percentage value of torque above minimum torque.

• Stable torque point
  Shows when torque reaches stable torque.
  Stable torque is set with onset of degradation point.

• Stable time
  Time between "S" (stable torque point) and "O" (onset of degradation point).
  Value gives information about processing time and stability of compound.

• Degradation Peak
  Shows when degradation has reached highest value.
  Torque value is not significant and only used for the calculating of the degradation rate.
Gelling of PVC 1st. Phase

- Intact globular structure of PVC particles
Gelling of PVC 2nd. Phase

- Globular structure destroyed
- Flow of primary particles
- Free volume between particles is reduced
Gelling of PVC 3rd. Phase

- Destruction of primary particles
- Flow of subparticles
- Crosslinking molecular Network
- Additives (Stabilisers) visible in network
Application: PVC Stability test

Rheomix600, Roller Rotors
Temp: 170°C, n: 60rpm, m: 65g

a: PVC Dry-Blend with 1,9% Stabilizer
b: PVC Dry-Blend with 2,0% Stabilizer
Correlation Mixertest / Extrusion

Example: PVC-Stability
PolySoft Routine evaluation - Curve Type 4 & 5

Materials:
- PVC – granulate (Stability test)
- X-linking materials (PE, Rubber, Thermoset)

Point detection:
- L = Loading point
- S = Stable Torque
- M = Minimum point
- O = Onset of Degradation
- D = Degradation

Torque vs. Runtime
Loading Peak
Shows when mixer is completely filled and closed. Point is used as time base for calculation only. Value depends on kind of loading. It can be influenced by loading speed and loading force.

Minimum Point
Shows when material has reached the lowest viscosity.

Onset of degradation or cross linking point
Shows when material starts to degrade or cross link. Point is significant for process. It can be set as percentage value of torque above minimum torque.
Stable torque Point
Shows when torque reaches stable value.
Stable torque is set with onset of degradation point.

Degradation Point
Shows when material has been degraded.
Torque value is not significant.
It is used to calculate degradation/cross linking rate.

Stable time
Time between "S" (stable torque point) and "O" (onset of degradation/cross linking point).
Value gives information about processing time and stability of testing material.
Rubber-Compounds with the same Mooney Viscosity

Rheomix600, Banbury Rotors
Temp.: 130°C, n: 60 rpm, m: 63 g
Thermoset Mixer Rheomix 540 (principle sketch)

- **Backwall**
- **Conical Rotors**
- **Bushings with O-rings**
- **Conical Centre bowl**
- **Frontplate with melt thermocouple**
Crosslinking of PES-Resins

Rheomix540, Delta Rotors

*Temp: 120°C, n: 50rpm, m: 63g*

Sample 1

Sample 2
Mixer – Application:

PVC Plastisol

*Rheomix 600p, Rollerrotor, T = 70°C, m = 80g, n = 50rpm*

- PVC/Plastisol = 100/40
- PVC/Plastisol = 100/60
- PVC/Plastisol = 100/80

**Diagram:**

- **Red Line:** PLASTI6_M1_t1
- **Blue Line:** PLASTI8_M1_t1
- **Green Line:** PLASTI7_M1_t1

**X-axis:** Zeit [min]

**Y-axis:** Drehmoment [Nm]
Mixer – Application:

Batch differentation with speed-program

2 Rubber-Compounds

Rheomix 600, Banbury-Rotoren
Temp.: 130°C, m: 80g

Time [min] Torque [Nm]

Compound 1

Compound 2

n = 70 rpm

n = 5 rpm
**Mixer – Application:**

**Production-Improvement with Lab-Mixers**

### Production of Hot-Melts:
- Procedure 1: A + B + C
- Procedure 2: A + C + B

**Graph Details:**
- Torque [Nm]
- Energy [kJ]
- Time [min]
- Lines and annotations for Torque 1, Torque 2, Energy 1, and Energy 2.
Gas Flow Sensor for Rheomix

Application:
- foaming Compounds
- degradation tests
Measuring results Gasflowsensor:
Mixing Behaviour of Instant-Soup-Compounds

Torque

Temperature [°C]

Energy [kJ]

Sample A
Sample B
Sample A (Repro)
Planetary mixer for *PolyLab* system

**Application:**
Determination of plasticizer absorption in vinyl chloride (VC) polymers (PVC-Dryblends)

Mixing of solid powders with liquids

Preparation of a PVC paste for testing purposes under controlled conditions

**Standards:**
DIN 54 800 / DIN 54802 / ISO 4612
Planetary mixer for *PolyLab* system

- **Technical Specifications**
  - mixer bowl volume: app. 2.500 cm³
  - temperature control: liquid (water or oil)
  - temperature range:
    - Water: $T_{\text{max}} = 95°C$
    - Oil (Synth 210): $T_{\text{max}} = 150°C$
  - $n_{\text{max.}}$ (PolyLab) = 200 1/min
    - gear ratio for planetary rotor: $i = 1:4$
    - gear ratio for skimmer: $i = 3:4$
  - $Tq_{\text{max.}} = 50$ Nm
Planetary Mixer curve - Plasticizer absorption

- Speed 100 rpm
- 400g PVC Powder
- 200g DOP Plasticizer
- Temperature variation

Torque vs. Time

- 50°C
- 90°C

T2 Drypoint
A new approach by combining traditional methods with new technologies
The electrical conductivity of rubber compounds as a measure for the dispersion of carbon black is most important. Studies on the final compounds by use of classical laboratory instruments have been reported (e.g. Carbon Co., Fort Worth).

Getting those information during the mixing process can be of great importance for quality control and waste reduction.

Thermo Fisher Scientific has now successfully integrated a electrical conductivity sensor in a laboratory scaled mixer.
Electrical Conductivity Measurement for Rubber Compound

**Test 1:**
- Test material: SBR 1500 with 32 vol.% Carbon black N220
- Carbon Black characteristics: relatively fine with high specific surface

**Test 2:**
- Reproducibility of Test 1

**Test 3**
- Test material: SBR 1500 with 32 vol.% Carbon black N330
- Carbon Black characteristics: relative to N220 the N330 has bigger particle sizes and thus a lower specific surface
Electrical Conductivity Measurement for Rubber Compound

Test material: SBR 1500 with 32 vol.% Carbon black N220
Electrical Conductivity Measurement for Rubber Compound

Reproduction: SBR 1500 with 32 vol.% Carbon black N220
Electrical Conductivity Measurement for Rubber Compound

Test material: SBR 1500 with 32 vol.% Carbon black N330
Change from Mixer to Extruder