The influence of carbon black types on the processability of rubber compounds in green tires

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Up to 15 percent of the gas in a car's tank is used to overcome the tires' rolling resistance to the road surface. So with low rolling-resistance tires (aka "green"), fuel economy can improve significantly. To develop more fuel-efficient tires, it is important to understand the processability of new rubber compound formulations. The perfect instrument to study this behavior is a torque rheometer with laboratory mixers and laboratory extruders because it simulates the processing conditions in a small-scale testing environment. The following application note shows how to study the influence of three different types of carbon black on the processability of a rubber compound formulation for tire production.

Test samples

Rubber compound tire formulation, based on a branched cobalt butadiene rubber (Buna® CB 1220 from ARLANXEO), using three different types of carbon black:

- **N326 rubber carbon black**
  - Nitrogen surface area: 78 m^2/g, Iodine adsorption: 82 g/kg
  - Fine reinforcing carbon black with low-structure
- **N234 rubber carbon black**
  - Nitrogen surface area: 118 m^2/g, Iodine adsorption: 120 g/kg
  - Fine reinforcing carbon black with increased structure
- **N339 rubber carbon black**
  - Nitrogen surface area: 91 m^2/g, Iodine adsorption: 90 g/kg
  - Fine reinforcing carbon black with increased structure

Testing equipment

Thermo Scientific™ HAAKE™ PolyLab™ OS Modular Torque Rheometer platform with:

- Drive unit: HAAKE RheoDrive™ 7 OS
- Single screw extruder:
  - HAAKE Rheomex 19/10 OS rubber
  - Screw diameter: 19 mm, length L/D 10, compression ratio 1:1
  - Roll-feeder system for rubber feeding

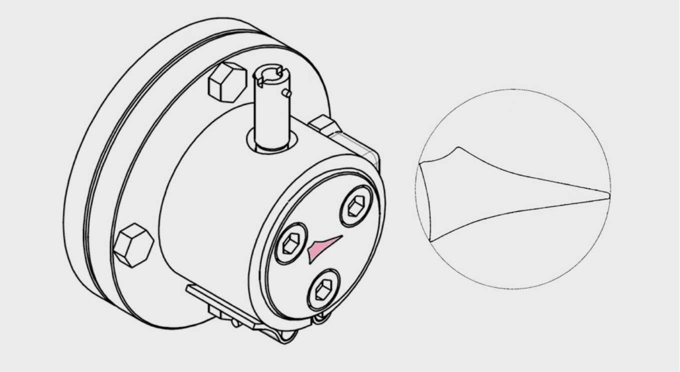


Figure 1: Schematic drawing of a Garvey die and its profile.

Test method 1: Garvey test

For this test, the extruder was equipped with an extrusion die with a Garvey profile according to ASTM D2230 (Figure 1) and a conveyor belt take-off. The Garvey die produces a profile with four different angles, which looks somewhat like a scaled-down version of half of a tire tread. A well-flowing rubber compound will give a smooth profile with no defects, even the smallest corners. A poor-flowing rubber compound will show an uneven, ripped, and swollen profile (Figure 2). The quality of an extruded profile is then ranked according to a ranking system described in the ASTM standard.

The results of the Garvey test with the three rubber compounds are shown in Figure 3, which also shows that the type of carbon black has a significant influence on the profile quality. The carbon blacks with increased structure (CB N339, CB N234) give a much smoother profile compared to the sample with low structure (CB N326).

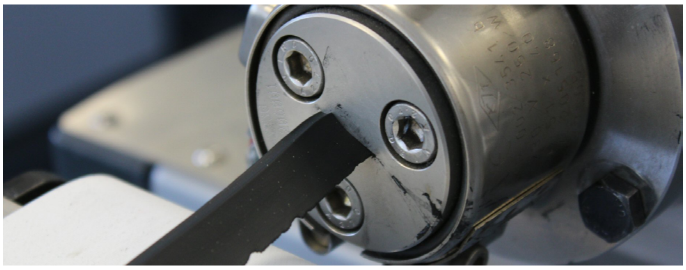


Figure 2: Extruded Garvey die profile.

CB N339:

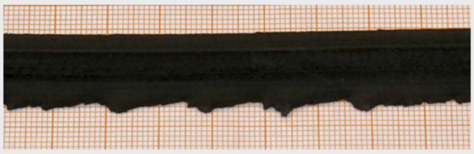
	1	2	3	4
Swelling				x
Edge				x
Surface				x
Corners				x

Edge:	10
Surface:	A

CB N326:

	1	2	3	4
Swelling			x	
Edge		x		
Surface			x	
Corners		x		

Edge:	4
Surface:	B



CB N234:

	1	2	3	4
Swelling				x
Edge			x	
Surface				x
Corners			x	

Edge:	8
Surface:	A

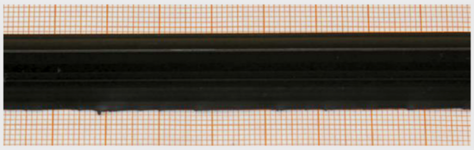


Figure 3: Garvey profile examples of the 3 rubber formulations.

Test method 2: Die-swell measurement

Die-swell (also known as the Barus effect) is a common phenomenon in polymer and rubber processing. It is where a polymer stream is compressed by entrance into a die and then is followed by a partial recovery or “swell“ back to the former shape and volume of the polymer after exiting the die. For this test, the extruder was equipped with a vertical rod die, a rod die nozzle D = 2 mm, L/D = 0, and a laser die-swell tester.

The system continuously measures the diameter of the expanded strand. The die-swell is calculated from the relation between the measured diameter and the actual diameter of the rod die nozzle.

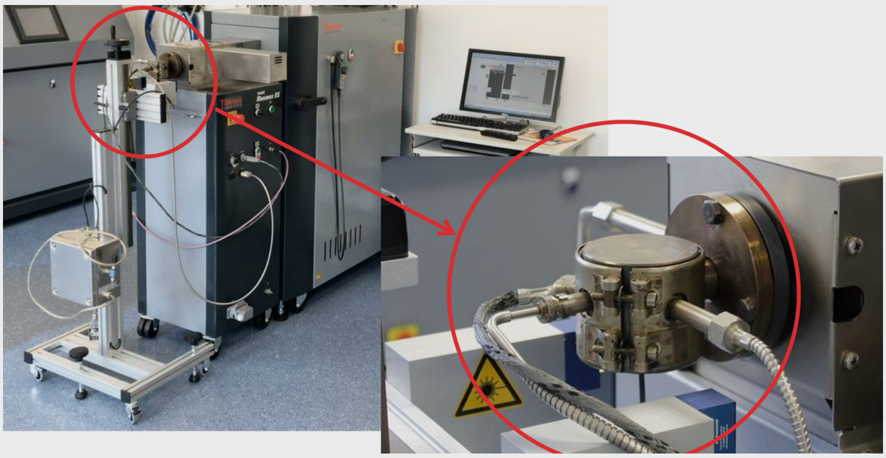


Figure 4: HAAKE PolyLab OS setup with die-swell measurement.

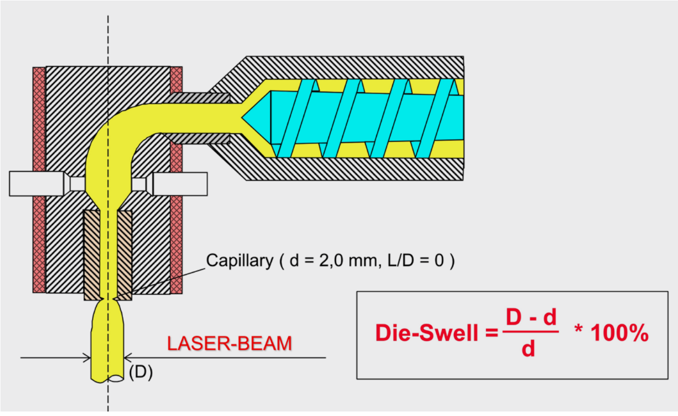


Figure 5: Schematic and calculation of die-swell measurement.

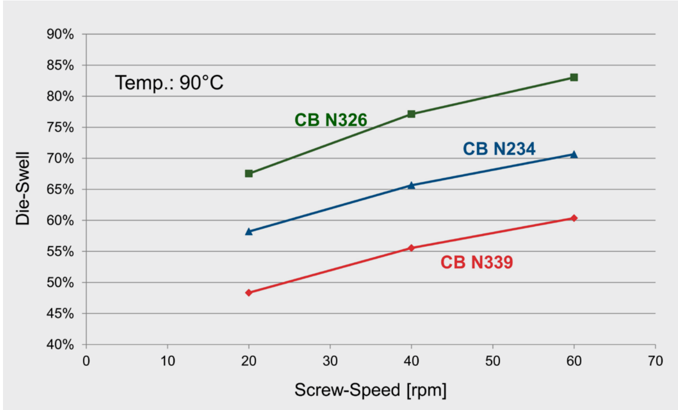


Figure 6: Die-swelling phenomena for three rubber formulations.

The three samples were tested at three different screw speeds (20 rpm, 40 rpm, and 60 rpm). The results of these tests are shown in Figure 6.

Also, in Figure 6, the significant influence of the type of carbon black can be seen. The compounds with carbon black with increased structure (CB N339, CB N234) show a much lower swelling behavior compared to the sample with carbon black with low structure (CB N326). The die-swell test also more clearly shows differences between the compounds with the carbon blacks with the increased structure (CB N339, CB N234).

Test method 3: Extruder capillary rheology

To test the rheological properties of the rubber compounds, the extruder was equipped with a horizontal slit capillary die with a measuring geometry of W = 20 x H = 2.0 mm (Figure 7).

To determine the output of the extruder, a balance connected to the HAAKE PolyLab system's control computer with an RS232 connection was used.

A test procedure was programmed using the PolySoft OS Capillary Rheometry software, which executes the measurement sequence automatically after programming. The software runs the extruder at different speed steps. Each speed step measures the pressure drop inside the slit capillary to calculate the shear stress and then uses the output information from the balance to calculate the shear rate. This measurement data calculates the compound viscosity at different shear rates (Figure 8).

Again, the significant influence of the type of carbon black can be seen. The compounds with increased structure carbon black (CB N339, CB N234) show a higher viscosity and a lower shear thinning effect. The rubber compound with low-structure carbon black (CB N326) shows a much lower viscosity, especially at higher shear rates.

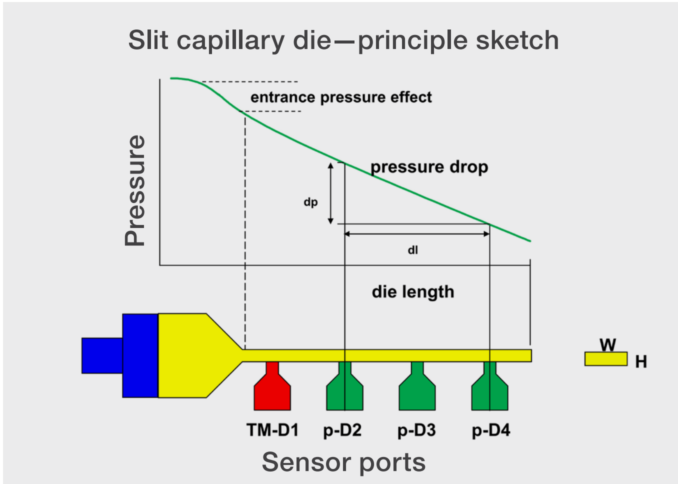


Figure 7: Schematic of a horizontal slit capillary die.

Slit capillary die—calculations for newtonian liquids

Pressure Gradient: $p' = \frac{dp}{dl}$

Shear Rate: $\dot{\gamma} = \frac{6 \bullet Q}{W \bullet H^2}$

Volume flow: $Q = \frac{V}{t}$

Viscosity: $\eta = \frac{\tau}{\dot{\gamma}}$

Shear stress: $\tau = \frac{H}{2} \bullet p'$

Assumptions:
- Laminator, stationary, and isothermal flow
- Wall sticking

Figure 8: Viscosity calculation for 3 different rubber formulations.

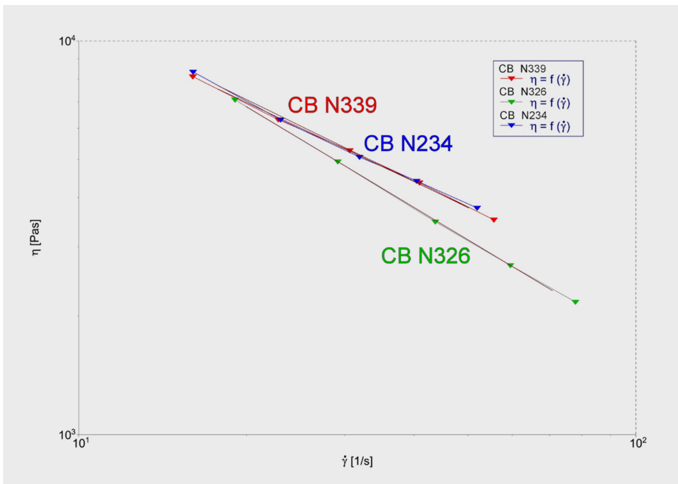


Figure 9: The result of the viscosity measurements.

Conclusion

The increasing demand for fuel-efficient tires at an affordable price increases the need for meaningful, accurate, and simple test methods to develop and determine the processability of new rubber compounds.

This application note shows how the HAAKE PolyLab OS Torque Rheometer system can be practically used to solve the challenges mentioned above. Three different test methods can be performed with one measuring system, and changeover time from one test to another is minimized due to the modular nature of the HAAKE PolyLab system. This time-efficient workflow allows a high number of experiments to be performed in a short amount of time. Linking test results and the real-world production process is vital to success. As a scaled-down production system, the HAAKE PolyLab OS system can achieve meaningful processing parameters on a laboratory scale that relate to the full-scale manufacturing experience. The HAAKE PolyLab OS Rheometer platform makes it possible to formulate compounds, produce test specimens, and characterize samples for processability, all with one system.

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