APPLICATION NOTE

Influence of stabilizers on the flow and degradation properties of polyamide

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Introduction

Polyamide (PA), commonly known as nylon, is a popular engineered thermoplastic material. Automobile manufacturers often use it for engine parts such as air intake manifolds or engine covers because the material withstands high temperatures. It is crucial then to manage raw material quality and processing conditions, so the final material property of polyamide is suitable for such applications.

During the processing of polyamide, high temperatures and the presence of a nucleophile such as water lead to the degradation of the polymer chains. This causes a decrease in polymer molecular mass and an increase in polymer end-groups. That leads to a decline of the viscosity of the polymer melt and the mechanical properties of the final product.

It is possible to delay the onset of link decomposition by adding stabilizers. The Thermo Scientific™ HAAKE™ PolyLab™ system offers a quick, reliable method of examining the influence of stabilizers on material processing characteristics.

Test purpose

Test the effectiveness of a stabilizer in Polyamide PA6 processing.

Test equipment

- HAAKE PolyLab OS Torque Rheometer
- Thermo Scientific™ HAAKE™ Rheomix 600 electrically heated laboratory mixer
- Roller rotors
- Thermo Scientific™ HAAKE™ PolySoft Mixer Software

Test conditions

· Mixer temperature: 240 °C
· Rotor speed: 60 rpm
· Sample weight: 52 g

Test material

· Sample 1: PA6 without stabilizer
· Sample 2: PA6 with stabilizer

Test method

The mixer is heated to the desired testing temperature and the drive motor runs at the selected rotor speed.

Before the actual measurement, a calibration routine is conducted, to zero out any possible torque signal generated by the mixer gear box. Then the cold sample material is rapidly added into the heated, running mixer, and the mixer is closed by the feeding ram.

The torque and the melt temperature are recorded over the mixing time, to measure the melting and processing behavior of the sample.

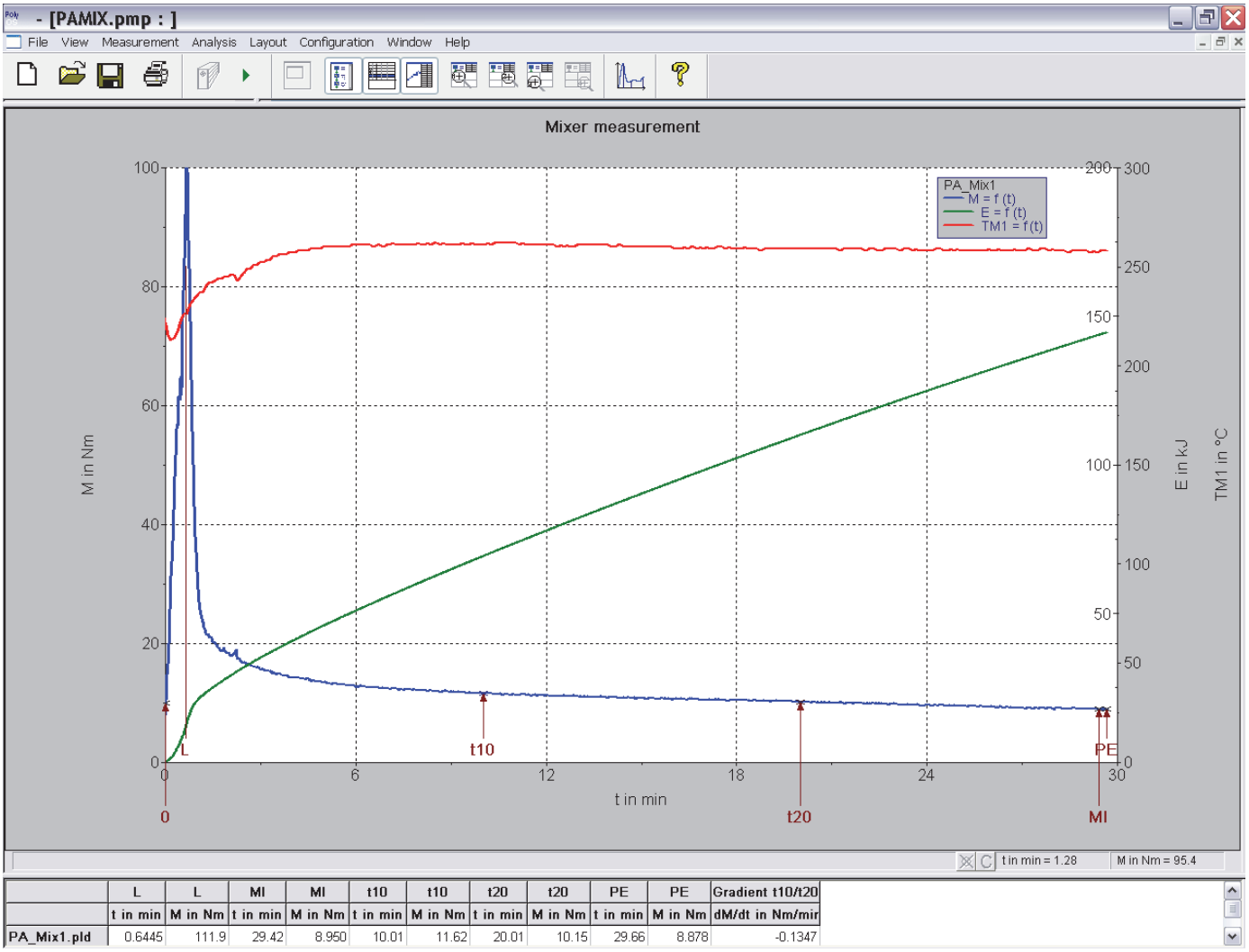
Test results

Figure 1 shows the torque-time curve of a polyamide without the addition of a stabilizer. The graph shows the torque (M, blue), the melt temperature (TM1, red) and the energy consumption (E, green) as a function of test time.

Basic curve discussion

The initial filling of the mixer with the rigid PA sample results in a significant rise in torque, the so-called Loading Peak (L). This peak serves as the starting time for the calculation of the substance's various characteristics.

After the sample material has become totally molten, the torque-time curve shows a continuous decrease in torque. This decrease in torque is caused by the reduction of the melt viscosity due to the degradation of the un-stabilized sample.



	L	L	MI	MI	t10	t10	t20	t20	PE	PE	Gradient t10/t20
	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	dM/dt in Nm/min
PA_Mix1.pld	0.6445	111.9	29.42	8.950	10.01	11.62	20.01	10.15	29.66	8.878	-0.1347

Figure 1: Rheogram of mixer test without stabilizer.

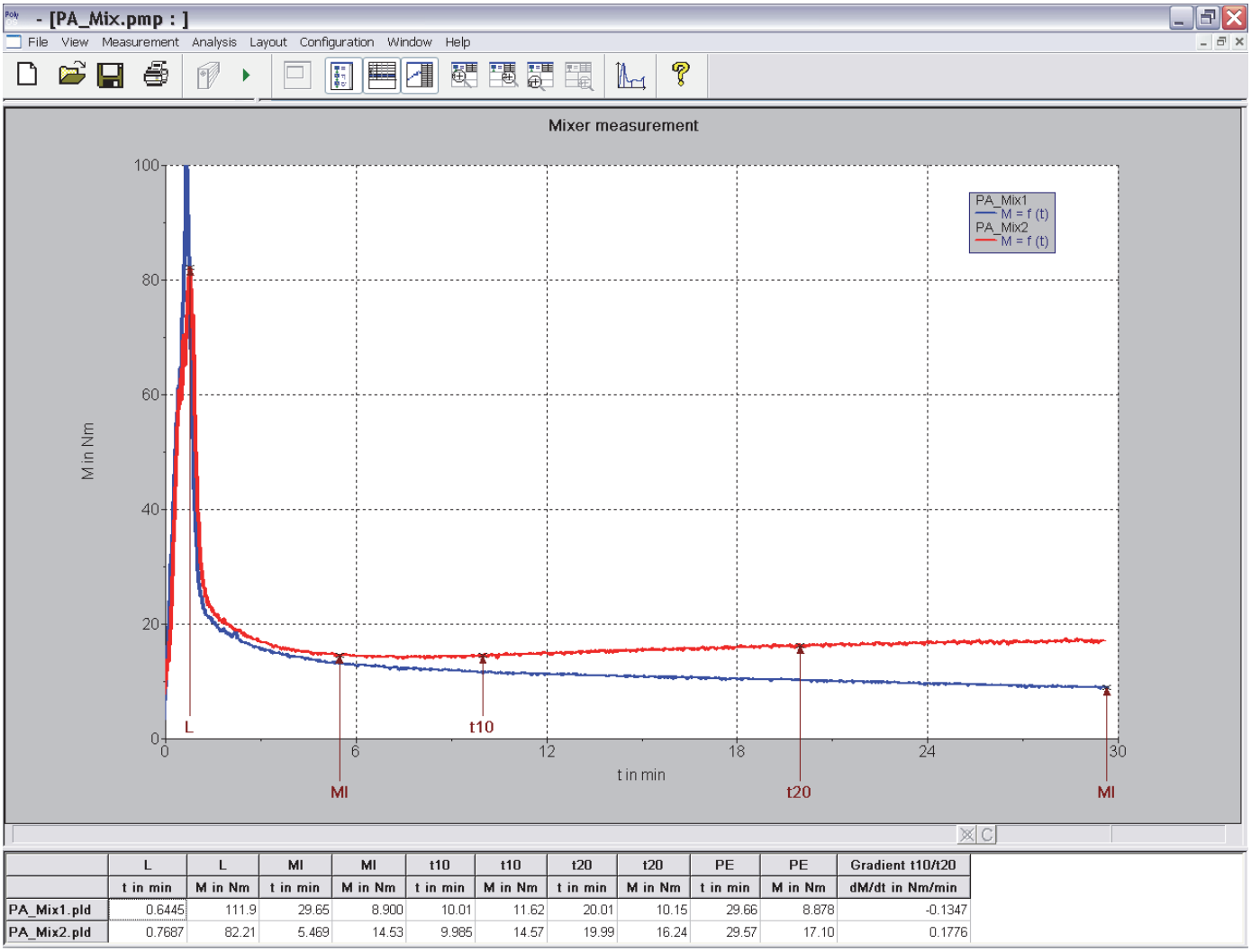
The evaluation of the characteristic curve points is done using the table shown below the graph in figure 2 . Apart from registering the time, torque, energy and melt temperature values, the gradient between the torque value after 10 minutes and 20 minutes was calculated as a measure of the degradation speed.

Another mixer test was carried out with sample 2, a PA6 polymer compound that contains stabilizer. The stabilizer helps to conserve the polymer end-groups under harsh temperature conditions and thus should result in an improved material stability. Therefore, with the second mixer test, a stable torque value is expected.

Comparison of test results

Figure 2 shows the torque curves of the mixer tests for the PA with and without stabilizer in one graph.

The stabilizer influence is clearly visible. The sample with added stabilizer (red curve) shows a higher torque value in the molten state. Also the torque does not decrease further over the course of the measurement. It can be concluded that the stabilizer prevents the breaking of the polymer chains and thus counteracts the degradation of the polyamide.



	L	L	MI	MI	t10	t10	t20	t20	PE	PE	Gradient t10/t20
	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	dM/dt in Nm/min
PA_Mix1.pld	0.6445	111.9	29.65	8.900	10.01	11.62	20.01	10.15	29.66	8.878	-0.1347
PA_Mix2.pld	0.7687	82.21	5.469	14.53	9.985	14.57	19.99	16.24	29.57	17.10	0.1776

Figure 2: Torque curves of mixer tests for PA with and without stabilizer.

Conclusion

The tests show that the HAAKE PolyLab Torque Rheometer with an attached laboratory mixer is ideal equipment to study the flow and processing behavior of polymer compounds. It can help to establish a QC routine for PA applications for easy, fast and reliable determination of final product quality attributes.

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