PVC Mixer Tests- Reproducibility and Influence of Test Conditions

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Mixer Tests in PVC Industry

If it comes to testing of PVC formulations - the fusion behavior, the compound stability, the processing behavior - the laboratory mixer is still the ideal measuring tool.
A laboratory mixer is very sensitive to any changes caused by the compound formulation, or any changes of the Dry Blend components, like the resin itself, the used additives and the added fillers.
Because of the sensitivity of the laboratory mixer, also wrong handling does have a significant influence on the measuring results. So to get reliable test results, a user has to make sure, that he handles the mixer always in a reproducible manner.
This application report should show the good reproducibility of mixer tests, but also give an idea, how changes in the testing conditions will influence measuring results.
For these investigations the following equipment configuration was used:

- Thermo Scientific™ HAAKE™ PolyLab™ OS - RheoDrive™ 7 OS
- Torque sensor for HAAKE PolyLab OS, 400 Nm
- Thermo Scientific PolySoft OS Mixer Test & Data Evaluation Software
- Thermo Scientific™ HAAKE™ Rheomix 600 OS
- Roller Rotors
- Pneumatic Ram

As a sample material, a Dry-Blend of rigid PVC, normally used for the production of window profiles, was selected.

Test procedure and measuring results

Typically a PVC Dry-Blend is a mixture of the basic PVC resin, fillers, processing aids, like softeners, stabilizers, inner and outer lubricants and other additives. Each component has an effect on the processing behavior of the compound.
One important factor is the fusion behavior of such a compound. Changes in the fusion behavior have a direct effect on the degree of gelation of the final product. For a PVC window profile this would result in a deterioration of the mechanical properties, or problems in the welding behavior.
Unlike a normal thermoplastic polymer, PVC does not melt, if it is just exposed to higher temperatures.
PVC needs additional shearing and compression forces to bring the PVC particles to agglomerate and finally to form a homogenous melt.

Fig. 1: Thermo Scientific HAAKE PolyLab OS - RheoDrive with Rheomix

In a laboratory mixer this fusion behavior can be studied by measuring the drive torque, and recording this torque over the mixing time. The PolySoft OS Mixer Test & Data Evaluation Software (Fig. 2) guides the user through this test, records the measurement data and evaluates all points of interest of the resulting measurement curve, by means of dedicated, pre-defined evaluation routines. This software works also as a data base for the measurements, and also allows further statistical studies.

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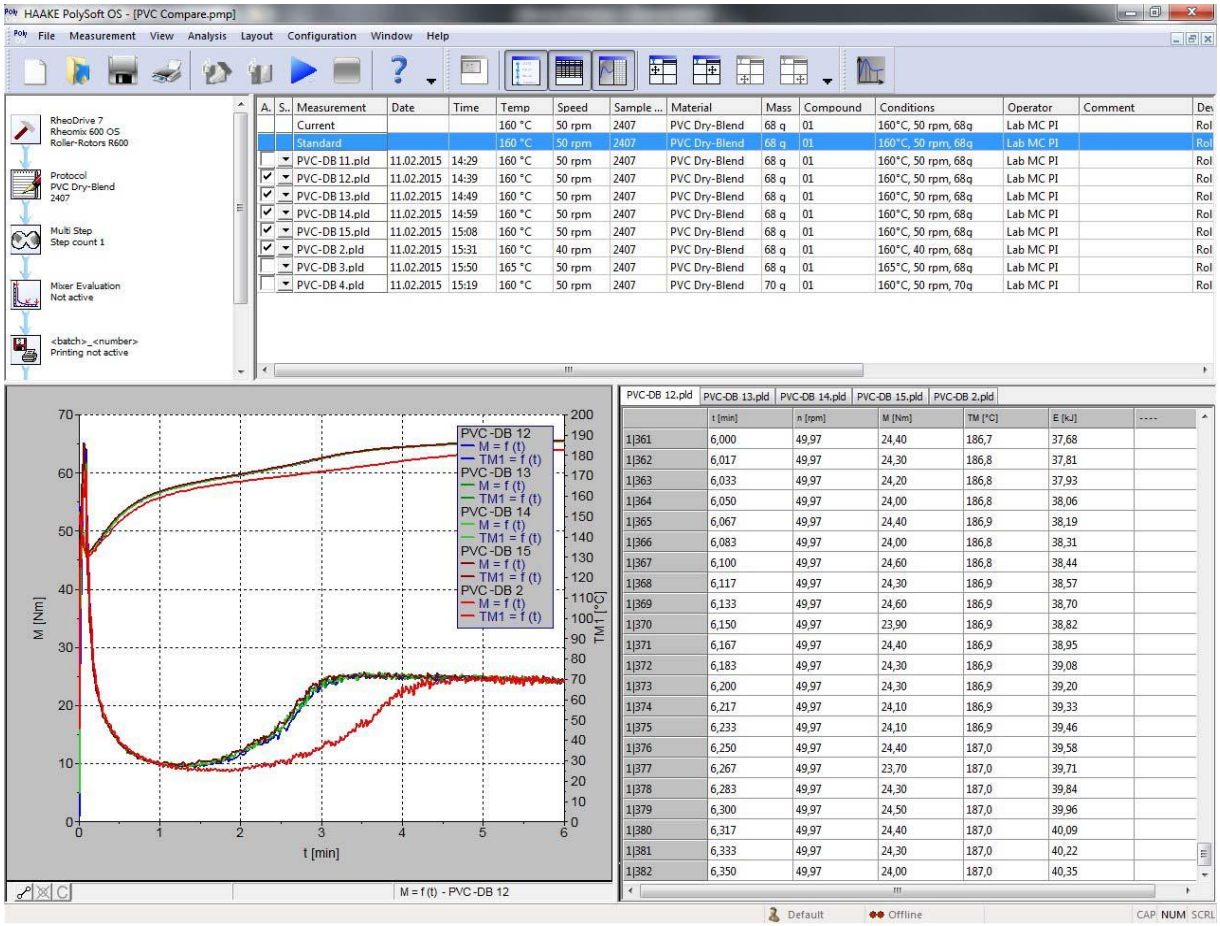

Fig. 2: Screen Capture - PolySoft OS Mixer Test & Data Evaluation Software

The following graph shows an evaluation of such a torque curve (Fig. 3).

At the beginning of the test, the loading of the PVC powder into the mixer causes an instantaneous increase of the torque ("L" - Loading Peak). After that, the powder distributes in the mixer chamber and some parts of the compound (e.g. waxes) melt due to the high mixer temperature. Both effects lead to a drop of the torque and leads to a first minimum ("V" - Valley). Due to the increase of mass temperature and the introduced shear energy, the PVC starts to combine to bigger agglomerates. This causes an increase of viscosity, which leads to an increase of torque. This process results in a second torque maximum ("F" – Fusion Maximum). The PVC Dry Blend forms a homoge-neous melt. Due to additional increase of the sample temperature, caused by frictional heating, the torque drops again until it comes to a constant torque. A balance between the increase of temperature caused by dissipation and decrease of temperature caused by heat

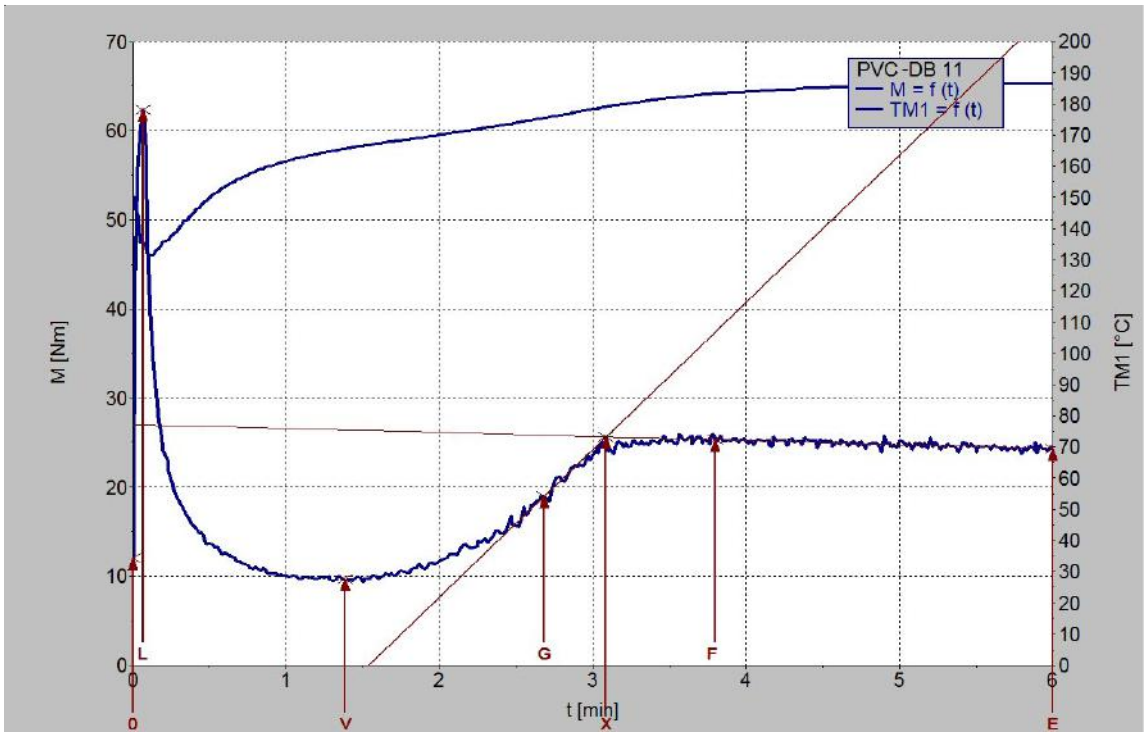

Fig. 3: Evaluation of a Mixer Curve

conduction through the chamber wall is reached. The torque which is adjusting here, is a relative value for the melt viscosity of the sample.
Additionally this graph shows a straight line intersection ("X") generated by the gradient of the point of inflection ("G") and the gradient around the fusion point ("F").

This straight line intersection "X" is often used to compare the different fusion behavior of samples, because this point "X" is easier to reproduce than the Fusion Maximum "F".

The software will then generate a measuring report, which documnets all the results (Fig. 4).

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PolySoft OS - Mixer

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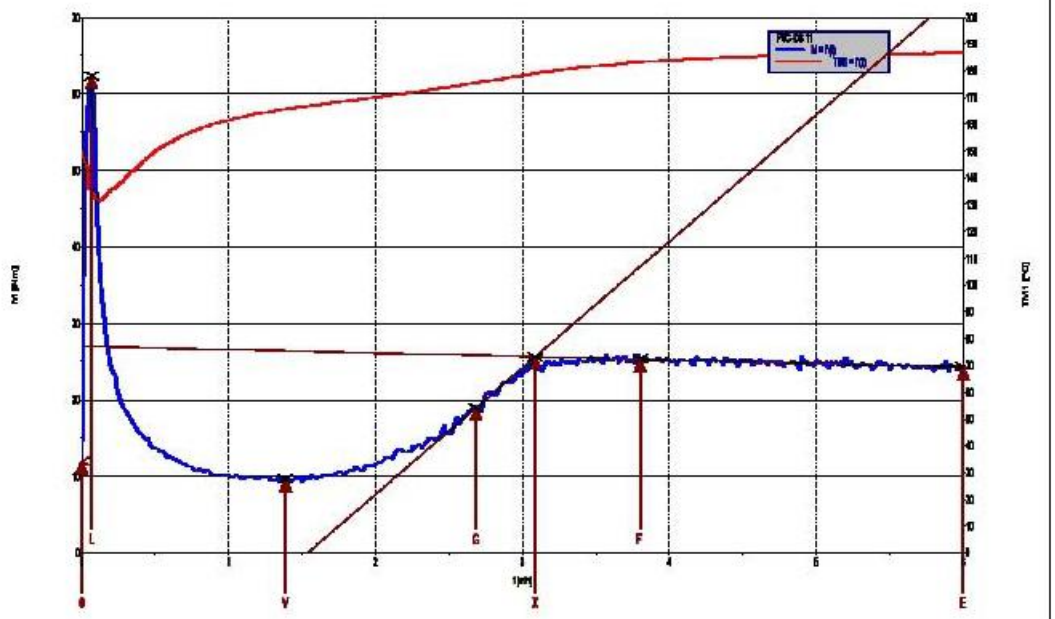
Filename: **PVC-DB 11.pld**

Material:	PVC Dry-Blend	**Date:**	11.02.2015	14:29
Batch:	01	**Device :**	RheoDrive 7	
Condition:	160°C, 50 rpm, 68g		Rheomix 600 OS	
Mass:	68,00 g		Roller-Rotors R600	
Density:	1400, kg/m³			
SetTemp :	160,0 [°C]	**Company:**	Thermo Scientific	
RotorSpeed :	50,0 [rpm]	**Operator:**	Lab MC PI	
Comment:		**Customer:**		



start point	0	0,0 [min]	12,0 [Nm]	150,4 [°C]	0,0 [kJ]
maximum 1	L	0.1 [min]	62,3 [Nm]	135,8 [°C]	1,04 [kJ]
minimum 1	V	1,387 [min]	9,7 [Nm]	165,8 [°C]	7,27 [kJ]
inflection point 1	G	2,683 [min]	19,0 [Nm]	175,6 [°C]	12,5 [kJ]
maximum 2	F	3,8 [min]	25,3 [Nm]	183,4 [°C]	20,7 [kJ]
end point	E	6,0 [min]	24,3 [Nm]	186,8 [°C]	38,0 [kJ]
straight line intersection 1	X	3,087 [min]	25,6 [Nm]	179,1 [°C]	15,2 [kJ]

fusion time	t(X-L)	3,02 [min]	energy difference	E(X-L)	14,19 [kJ]
fusion rate @G	dM/dt	18,0 [Nm/min]	energy difference	E(V-L)	6,235 [kJ]
torque difference	M(F-V)	15,6 [Nm]	energy difference	E(F-V)	7,952 [kJ]

Fig. 4: Software Report - Fusion Test of a PVC Dry Blend

Reproducibility of Mixer Tests

To check the reproducibility of mixer tests, five tests were done under the same conditions, using the same PVC Dry Blend.
Test conditions:

- Mixer Temperature: 160 °C
- Rotor Speed: 50 rpm
- Sample Weight: 68 g
- Test Time: 6 Minutes

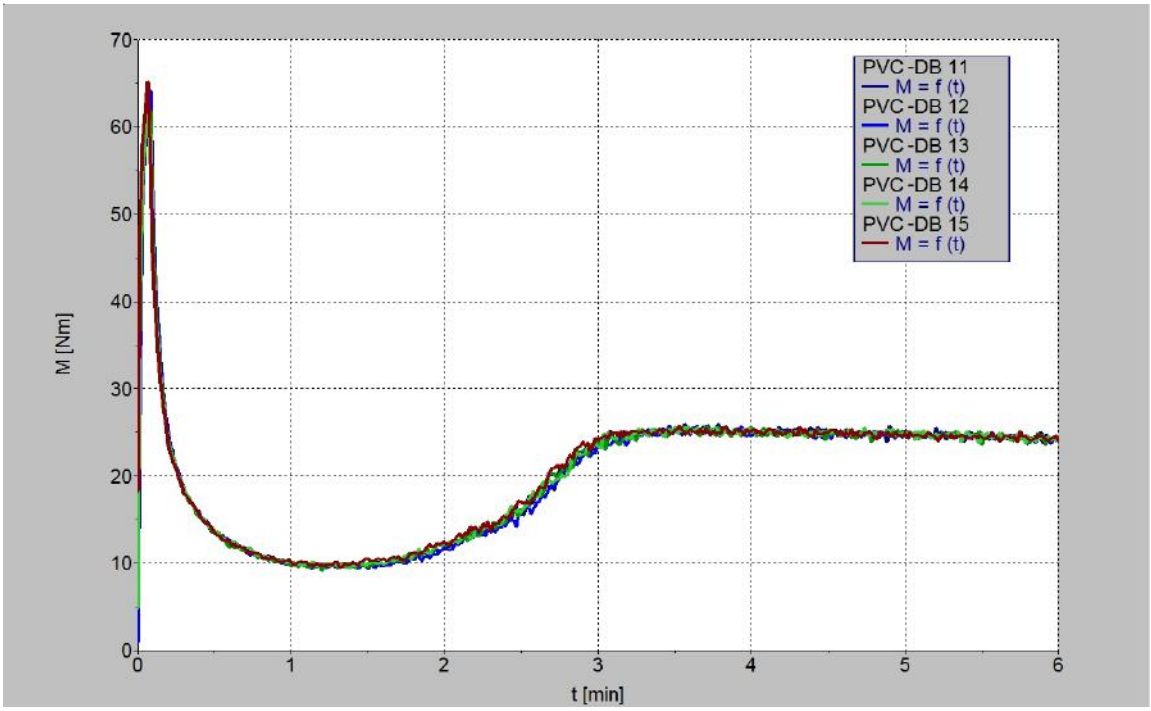


Fig. 5: Reproduction of five mixer tests

Fig. 5 shows the fusion curves of these five mixer tests in one graph. Clearly it can be seen, that the torque curves of these five tests are nearly identical, which shows the good reproducibility of the test method.
To get such a good reproducibility, it is important, that a user has to use the mixer always in the same manner. Changes in handling or mixing conditions will have an immediate effect on the test results.
To show, how small changes in the mixer settings or the testing conditions will influence the measuring results, the following tests were done with the same PVC Dry Blend, but with defined changes in the mixing conditions.

Influence of the Mixer Speed

Test conditions:

- Mixer Temperature: 160 °C
- Rotor Speed: 40 rpm, 50 rpm
- Sample Weight: 68 g
- Test Time: 6 Minutes

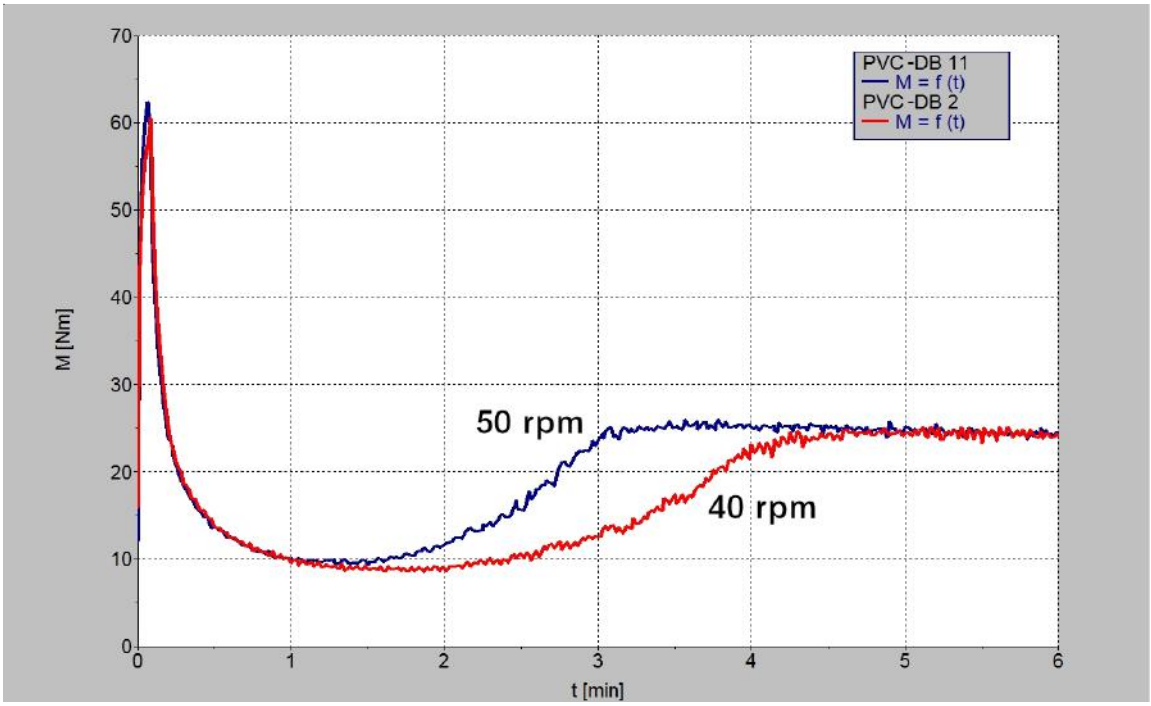


Fig. 6: Comparison of different Rotor Speeds

Fig. 6 shows the influence of the mixer speed on the measuring results.
The reduction of the rotor speed results in a much longer Fusion Time, because the slower turning rotors introduce less shear energy into the sample.

Influence of the Mixer Load

Test conditions:

- Mixer Temperature: 160 °C
- Rotor Speed: 50 rpm
- Sample Weight: 68 g, 70 g
- Test Time: 6 Minutes

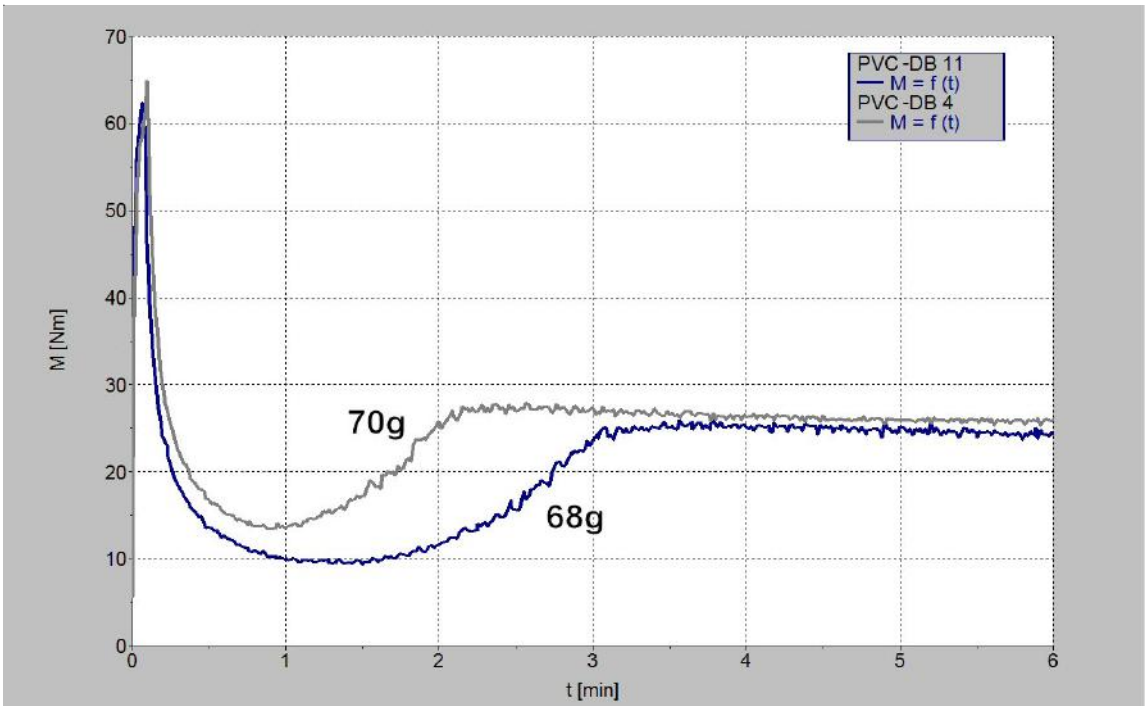


Fig. 7: Comparison of different Mixer Loads

The higher sample weight of just additional 2 g, causes a much shorter Fusion Time (Fig. 7).
This is because the higher sample volume results in more shearing.
So small mistakes made during the weighing of the sample, do have a significant effect on the measurement.

Influence of the Mixer Temperature

Test conditions:

- Mixer Temperature: 160 °C, 165 °C
- Rotor Speed: 50 rpm
- Sample Weight: 68 g
- Test Time: 6 Minutes

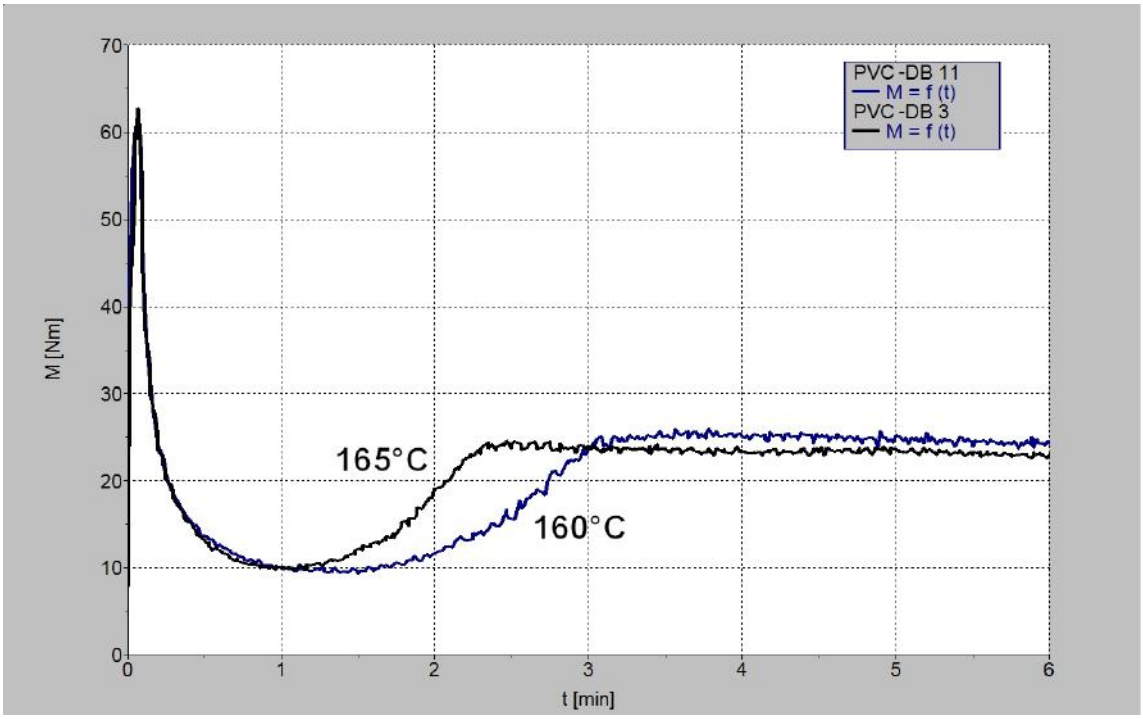


Fig. 8: Comparison of different Mixer Temperature

An increase of just 5 °C causes a much faster fusion of the sample (Fig. 8).
For a user this means, that he has to make sure, that he always feeds the sample at the same temperature.
To ensure this, the user has to take special care, that his cleaning procedure and cleaning time is reproducible.

Summary

These tests show that test with the laboratory mixer are well reproducible, very sensible to changes in compound formulations, but also very sensitive to changes in handling and measuring conditions.
When handled in a reproducible manner, the HAAKE PolyLab System in combination with a laboratory mixer is a perfect and reliable tool to characterize the processing behavior of PVC Dry Blend.

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