• feature article

Influence of mortar volume on rheological properties Optimising the workability of fresh high-performance concretes

construction & building industries

This article describes and discusses the methods and results of the investigations into the influence of the mortar volume in concrete on the rheological parameters of fresh concrete and their changes over time. In addition, it was shown that changes in the rheological properties of the fresh concrete over time can be predicted on the basis of tests and measurements of the corresponding mortar properties. From the point of view of workability forecasting and control, it is crucial to measure the rheological parameters of fresh high-performance concrete and their changes over time depending on the materials used while also considering technological factors [1-4]. In recent years, a considerable number of studies on the rheological properties of cementitious materials has been conducted. In this research, it was found that the rheological parameters of the cement paste and mortar, as well as the type and magnitude of their changes over time, mainly depend on the physico-chemical properties of both cement and superplasticizer, superplasticizer content, time of superplasticizer addition, w/c ratio and temperature. Detailed information on the influence of these factors on changes in the rheological properties of fresh concrete is presented in the existing literature [1-7]. At the same time, the



analysis of the studies conducted to date indicates that the type and magnitude of changes in the rheological properties of fresh concrete over time may also be related to the volume of the cement paste or mortar in the fresh concrete [8].

This possible correlation has been thoroughly studied. In the following sections, the methods used for these investigations and their results are described in detail. It was found that the workability of fresh self-compacting concrete can be adjusted and optimized on the basis of the rheological parameters identified for the fresh mortar.

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talk

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news & views Lets get 2009 off on the right foot (or is that paw!)

2008 has been a good year and 2009 has started the same way. Both Evie & Mal will be on holidays in January 2009 so that they can relax and get ready for the year ahead. They wish everyone a happy and successful 2009 and hope that the festive season for those that celebrate Christmas enjoyed it and spent it with friends and family. •



• in this issue

Specific Industry Key



continued from page 1

Experimental program -Measurements of rheological parameters of fresh mortar and concrete

The laboratory studies conducted so far show that the rheological behaviour of fresh concrete mixes may be described with a sufficient degree of accuracy by using the Bingham model, according to the following equation:

$$\tau = \tau_{o} + \gamma \eta_{pl} \qquad [Equation 1]$$

where τ (Pa) is the shear stress at the shear rate γ (1/s), and $\tau_{\rm o}$ (Pa) and $\eta_{\rm pl}$ (Pas) represent the yield value and plastic viscosity, respectively [6, 9, 10]. The yield value $\tau_{\rm o}$ determines the load required to ensure the flow of the mix.

Once the shear stress τ exceeds the yield value $\tau_{_0}$, the mix will flow. The flow resistance depends on the plastic viscosity $\eta_{_{\rm pl}}$, the greater the plastic viscosity $\eta_{_{\rm pl}}$ of the mix, the smaller the speed of its flow.

Zv	w/c /	Superplasticizer content
0.69	0.30	1.5; 2.0; 2.5; 3.0
0.00	0.35	1.0; 1.5; 2.0; 2.5; 3.0
0.64	0.30	1.5; 2.0; 2.5; 3.0; 3.5
	0.35	1.0; 1.5; 2.0; 2.5; 3.0
	0.40	0.5; 1.0; 1.5; 2.0; 2
0.58	0.30	2.0; 2.5; 3.0; 3.5
	0.35	1.0; 1.5; 2.0; 2.5; 3.0

Table 1 Research program.

The rheological parameters of fresh concrete or mortar can be measured by applying two markedly different shear rates N, and by measuring the resulting shear stresses M. The rheological parameters are determined by a regression analysis according to the following equation:

$$M = g + h N$$
 [Equation 2]

where g (Nm) and h (Nms) are rheological constants corresponding to the yield value τ_o and plastic viscosity η_{pl} , respectively. Following the determination of the measurement constants of the rheometer, it is possible, if necessary, to represent the values g and h in physical units. The method used to determine the measurement constants of the rheometer is presented in [9]. The theoretical bases and rules for rheological measurements are discussed in detail in monographs [6, 9, 10].



Fig. 1 Aggregate grading.

Mortar to concrete volume proportion Z_v	Ingredients	w/c ratio		
	[kg/m³]	0.30	0.35	0.40
Mortars		I		-
	c/z	841	807	776
$Z_v = 1$	S	1262	1211	1164
	W	252	282	310
Fresh concretes				
	c/z	570	554	
	S	855	831	
$Z_v = 0.68$	g	855	831	
	W	171	194	
	m/a	1.06	1.10	
	c/z	532	518	505
	S	797	777	757
$Z_v = 0.64$	g	975	949	926
	W	159	181	202
	m/a	0.86	0.90	0.94
	c/z	491	479	
	S	736	718	
$Z_v = 0.58$	g	1104	1077	
	W	147	168	
	m/a	0.70	0.73	

c: cement; w: water, s: sand 0-2 mm; g: aggregate 2-8 mm; m/a: degree of filling the aggregate with mortar

Table 2: Proportioning of mortars and concretes.

Ingredients [%]						Specific surface [m ² /kg]	
SiO ₂	Ca0	AI_2O_3	Fe ₂ O ₃	MgO	Na ₂ Oe	SO ₃	
24.7	56.7	6.3	2.3	2.9	0.70	3.2	325

Table 3: Properties of cement CEM II/B-S 32,5R

Major constituent	Density [g/cm ³]	Concentration [%]
Polyether	1,09	34

Table 4: Properties of superplasticizers.





← continued from page 3

Testing program

In the research, the influence of the mortar volume in the concrete and of the superplasticizer content on the rheological parameters of fresh concrete with different w/c ratios was investigated. In four stages, the voids between the coarse aggregate particles were filled with mortar. This infill was equivalent to mortar-to-concrete ratios of $Z_v =$ 1.00, 0.66, 0.61, 0.56. Two w/c ratios were set: w/c = 0.30 and 0.35. The superplasticizer content was modified within a range of 1 to 3% of cement by mass (w/c = 0.30), and within a range of 1.5%to 3% by mass (w/c = 0.35). The factors considered and their values are shown in Table 1. In addition, the influence of the superplasticizer content on the rheological parameters of fresh concrete of w/c = 0.40 and $Z_v = 0.64$ was investigated. The values of the individual factors were chosen in accordance with the mix design recommendations for high-performance and self-compacting concretes [4, 5, 11].

Materials features and mix compositions

The properties of the cement CEM II/B-S 32,5 R and of the superplasticizer used in the tests are provided in Table 3 and Table 4. The mortars have been designed with a sand/ cement ratio of s/c = 1.5, and they have been prepared from sand with a grading index of $U_k = 3.43$. A 2–8 mm mineral aggregate with a grading index of $U_k = 6.40$ was used for the concrete. The grading curves of the sand and mineral aggregate are shown in Fig. 1. The compositions of the mixes are presented in Table 2.

Testing method

The mixes were prepared in a pan mixer with a capacity of 50 dm³. The rheological parameters were measured using BT2 rheometer (see Fig. 2) for flowable fresh concretes with a slump of at least 200 mm). To perform the measurement, a material sample was placed in the sample container. The BT2 rheometer was positioned in the middle of the measurement container, and one full turn was performed. During this turn, the moment was measured using two probes located at different radii in relation to the centre. On this basis, the baseline values of the rheological parameters of the mix were calculated. The rules and methods applied to measure the rheological parameters using the BT2 device are discussed in detail in [12]. In the tests, a fixed measurement period (i.e. the duration of a complete turn) was defined, which was equal to 15 ± 2 s. Since measurement constants had not yet been determined for the BT2 rheometer, the rheological parameters measured are presented in conventional units. For each mix tested, at least four measurements of rheological parameters



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Fig. 2: Aggregate grading.



Fig. 4: Rheological parameters of mortars and rheological parameters of fresh concrete 5 and 60 minutes after the end of mixing.



Fig. 6: Influence of mortar volume in concrete and superplasticizer content on rheological properties of w/c = 0.35 fresh concrete.

have been carried out. In each of these series, measurements were performed 5 and 60 minutes after completion of the mixing process.

Results and discussion

The rheological parameters of the mortars filling the voids between the aggregate particles in the fresh concretes tested decrease as the quantity of added superplasticizer increases, and increase as the w/c ratio decreases (Fig. 3). The yield value gof the mortars shows a minor increase over time, which is more apparent for mortars with a lower w/c ratio and lower superplasticizer content. The plastic viscosity h of mortars does not change over time. Only if the w/c ratio and superplasticizer ratio are low, the plastic viscosity h slightly decreases over time. It should be noted that the yield value gand plastic viscosity h of fresh concretes are, on average, 40 and 20 times higher than the corresponding fresh mortar parameters.



Fig. 3: Influence of superplasticizer cotent on rheological properties of mortars used in investigation.



Fig. 5: Influence of mortar volume in concrete and superplasticizer content on rheological properties of w/c = 0.30 fresh concrete.



Fig. 7: Influence of w/c ratio and superplasticizer content on rheological properties of fresh concrete of $Z_v = 0.64$

As indicated in the comparisons of the correlations established for mortars (Fig. 3) and fresh concretes (Fig. 5 to Fig. 7), the nature of the changes in the yield value *g* and plastic viscosity *h* of fresh concrete is generally identical to the changes observed for the corresponding mortar. However, in the case of mixes with a low w/c ratio and superplasticizer addition, and a low mortar volume in the concrete, the magnitude of the changes in the plastic viscosity *h* may be different in mortar and concrete. In Fig. 4, linear correlations are shown that enable the conversion of the values of the rheological parameters of the mortar obtained during the measurements to rheological features of the concrete mix with coarse aggregate of a maximum size of 8 mm and with a different volume of the identical mortar (Z_v ratio). In general, these correlations confirm the findings presented in [13] while at the same time demonstrating that the forecast of the plastic



Source of variance	Yield value g		Plastic viscosity h	
	F-ratio	α	F-ratio	α
A: Time	24.208	0.000	15.898	0.000
B: w/c ratio	558.767	0.000	1780.920	0.000
C: Mortar volume	356.452	0.000	1175.613	0.000
D: SP content	92.139	0.000	11.918	0.000
AB	8.406	0.004	7.472	0.007
AC	12.453	0.000	2.065	0.131
BC	143.799	0.000	61.491	0.000
AD	4.841	0.003	0.092	0.964
BD	55.685	0.000	11.622	0.000
CD	21.119	0.000	3.155	0.007

Table 5: Analysis of variance (ANOVA) of influence of time, w/c ratio, mortar volume and super-plasticizer content on yield value g and plastic viscosity h of fresh concrete 5 and 60 minutes after end of mixing.

viscosity h may be incorrect for concrete mixes with a low mortar volume. Moreover, another issue should be considered: A narrow range of changes in the yield value g observed for mortars is contrasted with a very wide range of changes in the same parameter for concrete mixes. This makes it absolutely crucial to determine the yield value g of the mortar very accurately.

The data gathered with respect to the influence of the mortar volume in the concrete and of the superplasticizer content on the rheological parameters of fresh concrete is presented in Fig. 3, 5, 6 and 7. The ANOVA variance analysis of the influence of time, w/c ratio, mortar volume in concrete and superplasticizer content on the rheological parameters of fresh concrete is reflected in Table 5.

An increase in the w/c ratio, mortar volume in the concrete and superplasticizer content results in a decrease in the yield value *q* of fresh concrete (Fig. 5 to Fig. 7). The influence of the increase in the w/c ratio on the decrease in the yield value gis the greater the more mortar the concrete mix contains. An increasing mortar volume causes a considerable decrease in the yield value q of concrete mixes with a w/c ratio of 0.30. A similar trend is seen for concrete mixes with w/c = 0.35, Independently of the mortar volume in the concrete and of the w/c ratio, an increased addition of superplasticizer initially causes a rapid decrease in the yield value *g* of fresh concrete until a certain minimum is reached. Any further increase in the superplasticizer content will change the yield value *g* only to an insignificant extent. As can be seen from the results obtained, the yield value *g* changes considerably in concretes with a low mortar volume and/or low w/c ratio when the superplasticizer ratio is increased, and this change becomes less significant again as the mortar volume in the concrete and/ or the w/c ratio increases. At the same time, the lower the mortar volume in the concrete and/or the w/c ratio, the higher the superplasticizer addition needs to be in order to obtain fresh concrete with a pre-determined yield value g. Also, it should be noted that, if the mortar volume in the concrete mix is low, the manufacture of a fresh concrete with a yield value *g* similar to that of a fresh concrete with a high mortar volume is difficult, even if a very large superplasticizer quantity is added.

In all fresh concretes tested, the yield value g increased over time independently of mortar volume, w/c ratio and superplasticizer content. The range of the increase in the yield value g over time is narrower if the w/c ratio, superplasticizer content and/or cement paste volume in the mortar (factor $\phi_{m/p}$) are greater. It is worthy of mention that the manufacture of a fresh concrete with only an insignificant change in the yield value g over time requires a considerably higher quantity of added superplasticizer than attaining the minimum yield value g directly after the end of the mixing process.

The plastic viscosity h of fresh concrete decreases as the w/c ratio and/or mortar volume in the concrete increases. The influence of the mortar volume in the concrete on the plastic viscosity hdecreases as the w/c ratio increases. Unlike the influence on the yield value g, the magnitude of the influence of the superplasticizer content on the plastic viscosity h of fresh concrete depends on the mortar volume in the concrete, and on the w/c ratio.

For concrete mixes with a w/c ratio of 0.30, an increase in the superplasticizer content initially increases the plastic viscosity h. After reaching a certain maximum value, the plastic viscosity h decreases. The range of such changes decreases as the mortar volume in the concrete increases, although this range is generally not too wide, and influences the rheological properties of mortars only to a limited extent. In the case of mortars of w/c = 0.35 and 0.40, the superplasticizer content usually causes a decrease in plastic viscosity *h*. This effect is the lesser the higher the mortar volume in the concrete is. Only for fresh concrete of w/c = 0.35 and low mortar volume ($Z_v = 0.58$), an increase in the superplasticizer content initially causes an increase in plastic viscosity h.

The nature of the changes in plastic viscosity *h* over time of the fresh concrete mainly depends on the w/c ratio. In concrete mixes of w/c = 0.30, the plastic viscosity *h* decreases over time. The range of these changes is the wider the lower the mortar volume in the concrete is, and the higher the superplasticizer content is. When the mortar volume is high combined with a low amount of superplasticizer added, the changes in plastic viscosity *h* over time may be insignificant. In concrete mixes of w/c = 0.35, the direction of changes in plastic viscosity over time is dependent on the mortar volume in the concrete. For concrete mixes with Z_v values of 0.58 and 0.64, the plastic viscosity h decreases over time while it increases in concrete mixes of $Z_v = 0.68$. The range of these changes decreases as the superplasticizer content increases. In concrete mixes with a w/c ratio of 0.40, the plastic viscosity h increases over time.

Conclusions

An increase in the mortar volume leads to a decrease in the yield value *g* and plastic viscosity *h*. The yield value *g* of concrete mixtures increases over time. The range of these changes increases as the mortar volume in the concrete, the w/c ratio and the superplasticizer content decrease. The nature of the changes in plastic viscosity *h* of fresh concrete over time depends on the mortar volume in the concrete and on the w/c ratio. When the mortar volume in the concrete and the w/c ratio are high, the plastic viscosity h increases over time. When the mortar volume in the concrete and the w/c ratio decrease, the plastic viscosity h shows a tendency to decrease over time. In general, the range of changes in the rheological parameters decreases over time as the superplasticizer content increases. Only in the case of w/c =0.30 mixes was a trend towards an increase in the plastic viscosity *h* over time due to increased superplasticizer content observed.

The results obtained for mortars may generally be used to forecast changes in the rheological behaviour of fresh concrete over time. This makes it possible to design, optimize, control and correct the workability of fresh concrete on the basis of the rheological parameters measured for the mortar. However, the research presented in this article was performed within a relatively narrow range. Thus further tests are required that include mortars and mixes with varying ratios of constituents, different rheological characteristics, and aggregates of various types with different grading curves.

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A copy of this article including a complete reference list is available on request by quoting Schl-002 •



industry application Using rheo-optical methods to analyse the waxing of crude oil

chemical & allied industries

Introduction

About 30 billion barrels of crude oil with different chemical and physical properties are currently produced every year which results in a variety of different challenges. For example if in "waxy oils" hydrocarbons and naphthenic hydrocarbons crystallize they can form a wax layer on the inner surface of pipelines which will result in a significant reduction of the cross-section of the transportation channel.

In order to avoid high costs for pipeline cleaning in combination with downtime it is important to know under which conditions the crystallization occurs. The most important parameter is the temperature at which the oil comes from the hot deposit to the much cooler surface.

Temperature dependent measurements on two crude oils were performed using the HAAKE MARS in combination with the RheoScope module. The rheological and optical results are presented here.

Experimental

HAAKE MARS with RheoScope Module The RheoScope is designed as a compact and fully integrated module for the HAAKE MARS rheometer. The temperature range covers from -5°C up to 120°C and is extendable up to 300°C using the high-temperature option consisting of an electrical temperature control in combination with an upper heating system. Polished plates and cones with different diameters up to 60 mm and with ceramic shaft for reduced heat-transfer at high-temperatures are available. Images with a frame rate up to 15 per seconds can be taken with objectives of 5, 10, 20 and 50 times.

Benefits

- All measuring modes (CS, CR, CD) including normal force available with simultaneous recording of microscopic images
- Modularity: individual lenses, light source and camera can be adapted
- Temperature range: -5 up to 120°C (300°C)
- Rheometer, camera and microscope control with one software
- Visualisation and correlation of data and images
 within one software
- Optional image analysis software SPIP for particle size, particle size distribution and structure analysis



Figure 2: HAAKE MARS with mounted RheoScope module

Results

Both oil samples were loaded into a cone/plate geometry, heated up to 85°C, kept at this temperature to fully dissolve any crystal traces and were then cooled down. The rheological properties were monitored in CD oscillation at 1 Hz with 0.3% deformation. Pictures of the oil samples have been taken using a lens with 20 times magnification and crossed polarizers.



Figure 1: Technical Drawing (1a) and rear view (1b) of the RheoScope module

Further parameters of interest are "cloud point" or "wax precipitation temperature" the temperature when crystals start to occur and the "pour point" as the lowest temperature where the oil still flows

Fig. 3 shows the viscosity increase of the homogeneous oil TS 09 below 66°C. Below 30°C (pour point) the oil is completely solid. The solidification happens in two steps. Pictures were taken around 45 °C (cloud point) and in the solid oil, e.g. at 10°C, where small black areas indicate some remaining isotropic domains.



Figure 3: Temperature curve of crude oil TS09

The sample SO (Fig. 4) shows significant differences in the behaviour, e.g. the initial viscosity is lower by a factor of 10 and a constant viscosity could not be observed. The more inhomogeneous crystal network with bigger crystals and bigger gaps are the explanation for the more gradual changes in the rheological properties.



Figure 4: Temperature curve of crude oil SO

Conclusions

With the HAAKE MARS the rheological properties of two crude oil samples have been monitored over 5 orders of magnitude from the low viscous liquid to the solid state in one measurement using the precise CD mode.

Simultaneously, pictures of the growing crystal structure have been taken from the same samples with the RheoScope module. The two samples could be distinguished by the size of crystals and their distribution as well as the cloud point, where the first crystals appear.

A full copy is available on request •



product news and industry applications New module for torque rheometer with extruder - Sample sizer

polymer industries

The Sample Sizer is a new modular take-off system for the Thermo Scientific HAAKE PolyLab extruder system which allows you to picture an integrated polymer text workflow from compounding to final material testing.

In combination with a HAAKE PolyLab extruder system, polymer mixtures can be directly converted to solid and geometrically well defined test specimen profiles.

This is of specific interest to the polymer industry PVC specifically (including PVC producers, PVC producers of profiles, producers of stabilisers, and producers of additives), polymer R&D labs, analytical labs, and Universities.

Traditionally, polymer compounding and sample preparation are separate processes that require time, manpower and different machinery. The Sample Sizer provides an integrated solution. Difficult samples, eg: high molecular weight mixtures or filled polymer systems cannot be easily injection-moulded, and the Sample Sizer provides an alternative for sample preparation.

Applications

The Sample Sizer allows for the combining of extrusion and compounding tests with the production of small-scale polymer test specimen (standard = sheet profile, hollow and tube profiles on request) for further mechanical, chemical or optical testing.

Instead of re-melting and injection-moulding of polymer mixtures after compounding, the specific sample sizing is an integrated part of the compounding experiments with HAAKE PolyLab extruder systems.



Polymer workflow

After compounding with a HAAKE PolyLab extruder, the polymer melt will be directly transformed into defined solid polymer test specimen.



Calibration

Powered by IDE extrusion – technology. The key part of the Sample Sizer is the vacuum-controlled calibration unit for precise specimen dimensions.



Integrated cutter

With the integrated cutter different sample lengths are adjustable. Programable control unit for setting process parameters.

Typical test methods for polymer profiles from Sample Sizer:

- Tensile tests ISO 527
- Impact resistance ISO 179 etc.
- Bending test ISO 178
- Four-point bending test ISO 53457
- Heat resistance ISO 75-2
- Gloss assessment DIN 67530
- Rheology/Torsional/DMTA

Specifications

Sample Sizer module for HAAKE PolyLab Systems:

Dimensions L x W x H:	1200 x 960
	x 1800 mm
Weight:	450 Kg
Max. hauloff speed:	1.0 m / min
Max. cutting rate:	60 cuts / min
Max. profile dimensions	Ø 30 mm,
W x H:	40 x 15 mm
Max. cutting area	50 mm ²
Electrical connection:	3 x 400 V / 50 Hz / N PE / 35 A
Vacuum pump:	3 kW
Water connections:	4
Vacuum connections:	4
Air pressure connections:	1/4", 6 bar
Water inlet:	3/4", 4 bar
Water outlet :	Ø 75 mm



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product news and industry applications Twin Screw Granulator

pharmaceutical, cosmetics & allied industries

The Thermo Scientific Pharma TSG (Twin Screw Granulator) is a compact continuous processing granulator for pharmaceutical research, development and production.

The TSG continuous granulator with PLC control, data logging and recipe storage ensures reliable and repeatable results.

Features

- Small footprint
- Continuous operation
- High free volume
- Small process inventory
- Segmented screws and barrel
- Removable screws and liners

Benefits

- Reduced clean room area
- Consistent, controllable processing
- Suitable for PAT
- Reduced ATEX risk
- On-demand production
- Minimise scale-up risk
- Reliable cleaning

The TSG have special feeders to optimise dosing of low bulk density and difficult to feed API materials.

The horizontally split barrels have a crevice-fee, GMP design. Quick release clamps give easy access to screws and process contact surfaces for cleaning or configuration changes. The barrel is modular, with segments available for deeding solids and liquids, or for venting.



Fig 1: Removable contact parts for quick product changes.

Thermo Scientific Pharma TSG

Specifications	Pharma 16	TSG	Pharma 24	TSG
Barrel Length (L/D)	25:1	40:1	30:1	40:1
Barrel Bore Diameter	16 mm	16 mm	24 mm	24 mm
Screw Diameter	15.6 mm	15.6 mm	23.6 mm	23.6 mm
Channel Dept	3.3 mm	3.3 mm	5.15 mm	5.15 mm
Root Diamete	9.0 mm	9.0 mm	13.3 mm	13.3 mm
Diameter Ratio (D/d	1.73	1.73	1.77	1.77
Center-line Spacing	12.5 mm	12.5 mm	18.75 mm	18.75 mm
Center-line to Radius Ratio	1.56	1.56	1.56	1.56
Maximum Motor Power	2.5 kW	2.5 kW	7.5 kW	7.5 kW
Maximum Screw Speed	1000 rpm	1000 rpm	750 rpm	750 rpm
Extruder Dimensions				
LxWxH	1.5 x 0.7 x 1.5m	1.5 x 0.7 x 1.5m	2.0 x 0.7 x 1.5m	2.0 x 0.7 x 1.5m
Scale-up Data				
Internal Free Volume	68 cm ³	109 cm ³	274 cm ³	365 cm ³
Periferal Surface Area	316 cm ²	505 cm ²	854 cm ²	1138 cm ²
Services				
Electric Power (Volt/ph/Amp)	400V/3ph+N/20A	400V/3ph+N/20A	400V/3ph+N/40A	400V/3ph+N/40A
Cooling Water 20°C	5 liters per minute	5 liters per minute	20 liters per minute	20 liters per minute
Performance				
Throughput (formulation dependent)	1-10 kg/h	1-10 kg/h	5-50 kg/h	5-50 kg/h





Fig 2: Segmented screws, and barrels for flexibility in R&D with high free volume geometry to maximize intake of low density materials.

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Fig 3: Adjustable screw length and flexible configurations to change residence time and processing conditions.



service department report Calibrations & repairs of rheology instruments

all industries

Why Re-Calibrate?

The accuracy of the electronic components used in all instruments drifts over time. The effects of time in service as well as environmental conditions add to this drift. As time progresses changes in component values cause greater uncertainty in your measurements. At some point in time, the drift causes the instruments uncertainty to become undefined, meaning the manufacturer can no longer predict the uncertainty and guarantee measurement results. To resolve this issue instruments must be calibrated at regular intervals as defined by the manufacture.

Calibration is the comparison of an instruments performance to a standard of known accuracy. The result of a calibration may be documentation showing the deviation of a measurement from the known standard or it may also include adjusting the instruments measurement capability to improve accuracy.

The goal of calibration is to quantify and improve the measurement accuracy of your instrument. The benefits of maintaining properly calibrated equipment include:

- Reduced measurement errors
- Consistency between measurements
- · Increases in production yields
- Assurance you are making accurate measurements

Rheology Solutions recommends that you periodically calibrate your hardware to ensure

measurement accuracy and to keep your instrument operation in peak precision.

Calibrations

The past year has been particularly busy in the service department which has resulted in our Calibration schedule occurring in early 2009. To date, we have completed our calibration program in WA. Our service engineer will visit QLD, NSW and SA early in the New Year. All instrument users have been contacted and advised of the schedule for the dates when our service engineer, Richard Donaldson will be visiting their state.

To minimise the cost to our customers, we conduct annual calibrations interstate, however if you wish your Rheometer or Viscometer calibrated outside of the scheduled program, please phone or email Jackie, our Administration Manager at *jackiew@rheologysolutions.com* and she will either organise your HAAKE equipment to be sent in to our office (equipment will have a 48 hour turnaround from the time it arrives in our office), or put you on the list for a site visit to coincide with the schedule.

Pricing* for calibrations in 2009 based on Melbourne metropolitan and scheduled interstate dates are as follows: HAAKE MARS \$1,650 HAAKE RheoScope 1 \$1,650 HAAKE RheoStress 50 \$1,250 HAAKE RheoStress 600 \$1.250 HAAKE RheoStress 6000 \$1.250 HAAKE RotoVisco 1 \$1,650 HAAKE ViscoTester 550 \$825

Notes*

- Melbourne metropolitan customers can have their HAAKE instrument calibrated at any time on site.
- Prices exclude GST, and any parts required for instrument repairs.
- Multiple unit calibrations are priced on a sliding scale if they are serviced at the same time.
 Prices are available on request.

All calibrations are completed by our Service Engineer Richard Donaldson, who is factory trained at the manufacturers. A Calibration Certificate is provided as verification.

Equipment Repairs

This year, our Service Department has been busy with general repairs to a complete array of products. An assessment of the equipment is made and an estimate is provided on the costs including labour and parts, and in consultation with you, any parts are replaced and the repairs made. All repairs are conducted and equipment returned to the customer as quickly as possible.

If any parts are required to be ordered from our suppliers for the repair, it can take 4 - 6 weeks for the parts to arrive. When the parts are on hand the repair can be finalised. We notify you of timing and request that you please be patient if this is the case.

Quotes for all repairs, spare parts can be obtained from us at any time by emailing service@rheologysolutions.com.



Above: Richard Donaldson - Service Engineer





news & views Chocolate processing

food industries

Nestlé cocoa processing and chocolate manufacturing

Nestlé Chocolate Processing Research and Development has a long 100 year history, and is an important part of Nestlé's rich innovation heritage. The 'grandfathers' of Nestlé's chocolate history were François-Louis Cailler, Charles-Amédée Kohler and Daniel Peter. The history of Swiss innovation in chocolate started in the 19th century with Daniel Peter's invention of milk chocolate in 1875. The Nestlé label was used for the first time in 1904, following the merging of Kohler's and Peter's companies. Cailler's company was subsequently added in 1911. We have come a long way since then.

Today, Nestlé manufactures chocolate or chocolate compounds in more than 50 factories world-wide. The earliest was built in the 19th Century by Alexandre-Luis Cailler, the grandson of François-Luis Cailler, in Broc, Switzerland. Most recently, in November 2007 Nestlé acquired the Ruzskaya Confectionery Factory, which is one of Russia's leading premium chocolate manufacturers.

Taste and texture

Chocolate meets a number of emotional needs, including pleasure, reward and sharing. The two characteristics of chocolate that help to meet these needs are flavour and texture. Like tea and coffee, the flavour of chocolate is largely generated during the immediate postharvest phase. This is during the preparation and treatment of the cocoa beans, with additional flavours such as vanilla being subsequently added. Chocolate is uniquely characterised by a texture that is solid at room temperature but which melts at body temperature (37°C). Chocolate is a dispersion of fine sugar, cocoa and milk particles in a continuous fat phase. Therefore, chocolate processing has to take both taste and texture into account.

The consumer ultimately decides whether the chocolate he or she buys has the desired sensory properties. Therefore, qualitative sensory panel testing is an essential part of chocolate research and development. However, quantitative approaches to assess flow and friction in molten chocolate also have their place.

To this end, Nestlé has evaluated the role of rheological or tribological (interaction or friction between different surfaces) measurements to assess the viscous properties of chocolate (Lee et al, 2002). The main factors that influence these properties are the cocoa butter content, the lecithin content and the particle size distribution. Nestlé research has shown that tribological measurements are sensitive to the lecithin content of chocolate, regardless of particle size distribution, whereas rheological measurements are sensitive to lecithin only when particle size is small. By contrast, tribological measurements are sensitive to particle size only when the lecithin content is high, whereas rheological measurements are sensitive to particle size only when the lecithin content is low (Lee et al, 2002) These techniques therefore provide additional research tools for evaluating the sensory properties of chocolate.

More recently, Nestlé has contributed to a detailed review of factors that influence the rheological and textural qualities in chocolate (Afoakwa et al, 2007). This review highlights the need for further research to better understand how processing as well as particle size distribution, and ingredients, influence flavour and texture.

'Chocolate processing has to take both taste and texture into account"

Written by Klaus Zimmermann Head of Product Technology Centres and R&D Centres, Nestec S.A. Hilary Green Head of R&D Communications, Nestlé S.A.

The full article was originally printed in New Food, Issue 3, 2008 (pp 9 – 14), and permission has been provided to include a section of this article. www.newfoodmagazine.com

The Thermo Scientific HAAKE MARS, and HAAKE RheoStress 6000 can be provided with a Tribology cell as well as a special device to test the bending and breaking of chocolate bars.

A full copy of the article is available by quoting Rheo55 ●





product news & industry applications

Controlling enzyme reaction temperature with circular baths

food industries

Abstract

Controlling temperature is crucial for enzyme reactions as too high a temperature can bring a reaction to a complete stop. Thermo Electron Corporation's HAAKE Phoenix II heated circulator baths allow technicians to control temperature during enzyme reactions.

Enzyme Reactions and Temperature Control

Enzyme reaction technology is used for a wide variety of analytical applications, including life science, food and beverages, and agriculture. In addition to variables like pH and enzyme concentration, temperature control is one of the most important factors in these reactions.

All chemical reactions speed up as temperature is raised. According to Worthington-Biochem's "Introduction to Enzymes," a ten degree centigrade rise in temperature will increase the activity of most enzymes by 50 to 100%. The text goes on to add, "Variations in reaction temperature may introduce changes of 10 to 20 percent in results."

However, temperature above the enzyme's optimum (that is, where an enzyme exhibits maximum activity) can abruptly render an enzyme ineffective. At this temperature point, the kinetic energy of the enzyme and water molecules is so great that the structure of the enzyme molecules is disrupted.

Systems used to control enzymes temperature must have the ability to:

- React quickly and accurately.
- Remain accurate at the indicated temperature over a long period.
- Have a recirculation pattern in the bath which is equal around the surface of the bath.

Controlling Temperature with HAAKE Circulator Baths

In standard temperature control baths, the approach to reaching the correct temperature is often too fast and results in "overshoot". In a sinusoidal way, the temperature swings around the set temperature before it becomes stabilized. This overshoot may influence the performance of the reaction and may even stop it completely.



Above: The effect of temperature on enzyme catalysed reactions: The velocity of a chemical reaction increases with increasing temperature (A) but because of the increasing denaturation of the protein, the proportion of the active enzyme falls (B). These two processes result in the characteristic temperature profile of an enzyme (C).

HAAKE Phoenix II circulator baths eliminate the problem of overshoot. Basic in its design is the FuzzyStar software, which allows the cooling step and the heating step to work together. The Phoenix II's temperature sensor is placed in the bath, allowing the different cycles react quickly to temperature changes and preventing the bath's contents from going over the set temperature. In addition, the unit's circulation pump assures even temperature conditions throughout the bath, guaranteeing high precision and low turbulence.

The HAAKE bath's single screen display with a "wide angle" view allows users to set the system in any of six languages, including SI and common standards such PSI or °F, using a simple command structure.

Storing sample preparation data is easily accomplished via the Phoenix II's three communication ports. The unit can be connected to a wide range of external controls via an RS232/RS485 and/or Siemens Profibus interface, allowing users to track application performance and making it easy to exclude the temperature control step as a variable when looking at the results.

The Phoenix II's compact design, user-friendly software and reliable temperature accuracy make this an ideal unit for any lab conducting enzyme reactions.

A reprint of this paper is available by quoting reference no: TC05-02e ●





application focus Quality control for the petrochemical industry

polymer industries

chemical & allied industries

Plastics have become indispensable nowadays, and the areas of application are constantly broadened. The level of the requirements has risen in equal measure.

The industry demands for high quality raw material, that needs appropriate "online controls" to guarantee best properties for the fields of polymerisation, processing and application. Therefore it is essential that the extrusion process is checked for function and documented, as the polymer film should be clear of contamination and gels. It is important to detect in-homogeneities as soon as possible so the cause can be eliminated at an early stage of the process.

The rheological behaviour of the polymer melt is defined by the molecular structure and allows conclusions to the extrusion process.

The quality of the granules, eg: the size and shape, has an influence on the pneumatic conveyance and of course also on the material flow in the feeding process and extruded during high performance extrusion.

For further processing the frequency and size of gels and black specs in the film must be determined. These affect the optical appearance of the film and reduce the mechanical stability and ability for treatment, eg: printing, laminating etc.

The surface quality is also an important QC criterion when producing glossy and transparent films. Most thermoplastic material contains additives for processing and preservation, the content of which has to be analysed during quality inspection.

Optical Control Systems (OCS) has developed a modular conception of testing systems and optional devices which can be combined according to requirements:

- · Pellet/powder impurity
- Size and shape distribution
- Gels and contamination
- Additives measurement
- Rheology
- Haze, gloss, thickness measurement
- Pellet transport system

With these instruments all quality control requirements in modern plastic production and plastic conversion are fulfilled, offering tools for flawless history tracking of all critical parameters, for effective process control and for root cause analysis, creating a solid basis for process improvement.

The systems that are available include:

Powder & Liquids

 Power Testing System PT2C is capable of detecting contamination and discoloured powder particles and can be defined with the teaching tool. The irregularities are sorted into different class sizes which are completely user defined. The PT2C can be used for laboratory purposes as well as online inspection. The powder can be submitted as a sample or it can be withdrawn from the production line.

Pellet Inspection Systems

- Pellet Scanning System PS25C is used to analyse transparent, opaque and colour pellets. The material throughput is approximately 40 kg/h.
- High Speed Pellet Scan System PS200C is for online quality control during a production process. This system is an advanced version of the PS25C with an approximate throughput of 250kg/h.
- Pellet Size and Shape Analyser PSSA is a modular inspection system for precise analysis of size and shape of transparent and opaque granules (eg: twins, ripples, dog-bones, tails, fines). The features of the granules. Eg: size, shape, elongation, roundness, roughness and convexity are determined.
- Pellet Size and Shape Distribution PSSD is for the rapid analysis of size and shape distribution of transparent and opaque granules (eg: twins, triples, dog-bones, tails, fines). The system is able to analyse up to 50 kg/hr.
- Pellet Analyser PA66 is a combination of the PSSD and PS25C with real colour measurement (CM) optional.

Blown Film Tower

 The blown film tower BFT 300/400 with film hauloff, extruder and spiral die are used to produce high quality blown films for carrying out optical and physical measurement on the film. The system is for on/-off line measurements such as gels, contamination, haze, gloss etc.

Cast Film

 Chill Roll & Winder Unit CR9/WU9 contains and FT-IR for Online Spectroscopy, Haze, Gloss- and thickness measurement for either laboratory or online.

Film Testing

• Film Surface Analyser FSA100 can be used in the laboratory or production. The film quality (either transparent or opaque) is assessed

optoelectronically and the data is stored in an inspection report for later analysis. The system recognises and classifies defects, and every defect detected is transferred with the defect image to the measurement protocol with its feature vector (position, size, shape ...)

 Sample Tester ST4 is for the precise inspection of surfaces (transparent films, plates, glass and non-transparent/opaque materials such as paper, metal or textiles) with a small area. The system detects impurities, holes, scratches and other surface irregularities.

Rheometer

• OP5 online Rheometer measures Melt Index and/or Polydispersity of small solid polymer samples.

Additive Measuring

 Online FT-InfRared Spectrometry APLAIRS (Analysis of PLAstics by InfraRed Spectrometry) measures additives, physical properties, co-polymer and multi-layer composition, thickness as well as degree of polymerisation simultaneously.

Filtra Test

• Filtra Test ME FT 20 (according to DIN En 13900-5) can determine the Filter Pressure Value FPV.

Pelletising

 The laboratory scale strand pelletiser consists of an extruder (20/25 D V3), strands die, water bath and strand pelletiser with adjustable speed.

Thickness, Gloss, Haze and Colour Measurement

- Hazemeter Gamma 12 (according to ASTM 1003) calculates the haze value from the ratio of the scattered light and total transmitted light.
- Glossmeter GM (according to ASTM D 523 / DN 6730) determines the gloss characteristics of the manufactured film.
- Thickness Measurement TM9 is used for continuous measurement of the film thickness with the aid of measuring rolls.

Wide Web Inspection System

• FSP600 is a surface inspection system for use in production. Every defect is transferred with the defect image to the measurement protocol together with its feature vector (position, size, shape ...).

Pellet Transport System

 The Pellet Transport System PTS is a continuous and automatic transportation of pellets between the production lines and measuring systems.

Benefits of continuous granulation for pharmaceutical research, development and manufacture

pharmaceutical, cosmetics & allied industries

The pharmaceutical industry uses numerous batch processes. Recently, because of its many advantages, a trend toward continuous processes is becoming more prevalent. Continuous granulation offers opportunities for product and process development using small quantities of materials with minimized risks in scale-up. Characterisation of the granulation process using Design of Experiments techniques can be achieved using less API. In the development of new drugs/excipients, small scale continuous systems reduce time to market and employ processes comparable to production. In manufacturing, continuous granulation can deliver increased efficiency and a higher but flexible throughput. It also allows for the monitoring of processing parameters in the granulation process, which is particularly important for Process Analytical Technology (PAT). The following article compares batch and continuous processes with similar output rates. The cost of producing one sample is compared, and the product risks associated with each method are discussed.

A full copy of this paper is available on request by quoting LR-63 ●

application notes & technical articles Automatic detection of the thermal degradation of a polymer

polymer industries

Similar to any other material, the properties of polymeric materials are closely linked to their chemical nature.

Similar to any other material, the properties of polymeric materials are closely linked to their chemical nature. What makes polymers so different from low-molecular-weight substances is the huge influence of their molecular weight (M_w) and their molecular weight distribution (MWD) on their macroscopic behaviour. Without changing its chemical nature, we can e.g. "select" the rigidity or elasticity of a polymeric material just by varying the parameters of the polymerisation process.

Thus, to make a polymer with the desired properties we have to be able to quickly determine the right moment to stop the polymerization process in order to always get the same M_w and MWD. Also, these two parameters can be used to decide whether materials delivered meet the specifications or not.

One widely accepted method makes use of the fact that the crossover frequency determined with a frequency sweep (see Figure 1) depends on the $M_{\rm W}$ of a polymer, whereas the crossover modulus is related to its MWD.

From the crossover modulus the polydispersityindex (PI) can easily be calculated: PI = 100.000/Crossover Modulus

The HAAKE MARS is a high-end rheometer, which has a number of important advantages for polymer analysis. Amongst them is the sensitive normal force sensor, which allows the measurement of forces down to 0.01 N. In combination with the precise lift control this is a great tool to ensure the reproducible loading and axial relaxation of samples. Another unique feature of the HAAKE MARS is the Controlled- Environment-Chamber (CTC). Its powerful combination of convection and radiation heating guarantees the quick adjustment of temperature and low temperature gradients in the sample.

A full copy of this paper is available on request by quoting V-237 •



Fig. 1: Schematic crossover dependency on $M_{\rm W}$ and MWD



Above: The HAAKE MARS equipped with the CTC



application notes & technical articles

Effect of aggregation on shear and elongational flow properties of acrylic thickeners

surface coatings industries

chemical & allied industries

pharmaceutical, cosmetics & allied industries

The effect of intermolecular aggregation induced by hydrophobic and electrostatic interactions on shear and elongational flow properties of aqueous acrylic thickener solutions is discussed.

Complex shear modulus is determined at frequencies up to 10⁴ rad/s employing oscillatory squeeze flow. Extensional flow behaviour is characterised using Capillary Break-up Extensional Rheometry. Aqueous solution of poly (acrylic acid) (PAA)/poly (vinylpyrrolidone-co-vinylimiadazole)

(PVP-VI) mixtures exhibit unusual rheological properties described here for the first time. Zero-shear viscosity of the mixtures increases with decreasing pH and can exceed that of the pure polymers in solution by more than two orders of magnitude. This is attributed to the formation of complexes induced by electrostatic interaction in the pH range, where both polymers are oppositely charged. PAA/PVP-VI mixtures are compared to the commercial thickener Sterocoll FD (BASF SE), which is a statistical co-polymer including (meth) acrylic acid and ethylacrylate (EA) forming aggregates in solution due to "sticky" contacts among hydrophobic EA-sequences. PAA/PVP-VI complexes are less compact and more deformable than the hydrophobic Sterocoll FD aggregates. Solutions of PAA/PVP-VI exhibit a higher zero-shear viscosity even at lower molecular weight of the aggregates, but are strongly shear-thinning in contrast to the weakly shearthinning solutions of Sterocoll FD. The higher ratio of characteristic relaxation times in shear and

elongation determined for PAA/PVP-VI compared to Sterocoll FD solutions reflects that the chargeinduced complexes provide a much stronger resistance to extensional flow than the aggregates formed by hydrophobic interactions. This is most likely due to a break-up of the latter in extensional flow, while there is no evidence for a break-up of complexes for PAA/PVP-VI mixtures. These flexible aggregates are more suitable for the stabilisation of thin filaments in extensional flows.

Keywords

Thickener, acrylate, aggregation, complexation, shear rheology, elongational rheology

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A full copy of this paper is available on request by quoting PTE 671 •

application notes & technical articles Flow & levelling for water-borne coating systems

surface coatings industries

pharmaceutical, cosmetics & allied industries

In order to reduce solvent emissions to the environment water-borne coatings systems have been developed for a multitude of applications.

Many examples can be found in the coating of wood, plastic and metal substrates including car bodies. As their solvent-borne counterparts these coatings enables the end-user to fulfil protective and decorative functions simultaneously.

In practice it is often overseen that apart from the technical performance the application safety is the real key success factor to guarantee a defect-free coating quality. Unfortunately there are many

possible film defects that are able to deteriorate film performances as shown following.

Coating Defects

.....

•	Orange	peel	•	Fisheves
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- Craters Crawling
- Benard Cells Floating

Orange peel is an undesired surface waviness that

looks like the skin of an orange, fish-eyes are flat depressions in the film. Craters can be described as bowl-shaped depressions in the film that often have drops or bands of material at their centre and raised circular ridges. The depression can reach out to the layer underneath.

Crawling or retraction is a phenomenon related to de-wetting of the coating from the substrate and can often be observed at the edges of a panel. Benard Cell formation has its reason in an induced circulation inside the applied wet coating film, in some cases the formation of hexagonal cells can be observed after the curing process.

Floating is a consequence of Benard Cell formation in solid colours and is expressed as mottled, streaked or blotchy film appearance. The driving force results from differences in density of combined pigments especially in grey colour tones where organic and inorganic pigments are present at the same time.

All film defects as detailed are related to flow and levelling problems of the coating system. To get control over these film defects it is necessary to know the parameters that have influence upon them. As such five items can be identified as follows:

Influence of flow and levelling

- Surface tension
- · Surface roughness of substrate
- · Wet film thickness
- Rheological behaviour after application
- Drying time or open wet time of the film

A full copy of this paper is available on request by quoting Rheo039 •



application notes & technical articles

Influence of the incorporation sequence of different components on the energy consumption of the mixing process

polymer industries

In a production process it is of major interest to reduce energy consumption by optimising the mixing process.

The following example shows how a laboratory mixer can be used to optimise the incorporation sequence of different components of a mixing process.

Test Purpose

Optimising of the mixing process of a hot-melt adhesive regarding the energy consumption.

Test Equipment

- Torque rheometer system Thermo Scientific HAAKE PolyLab
- Electrically heated laboratory mixer Thermo Scientific HAAKE Rheomix 600 (Fig.1)
- Cam rotors (Fig.2)
- Thermo Scientific HAAKE PolySoft mixer software

Test Material

A hot-melt adhesive consisting of three components:

- A: Polymer (40%)
- **B:** Filler (33%)
- **C:** Resin (27%)

Test Procedure

Incorporation sequence of the components:

- Test 1:
- **A** (2 min)+**B** (3 min)+**C** (5 min)
- Test 2:
 - **A** (2 min)+**C** (3 min)+**B** (5 min)

Test Conditions

- Mixer temperature: T=150°C
- Rotor speed: n=40 rpm
- Sample weight: m=75 g
- Total test time: t=10 min

Test Results

The graph below shows the torque curves and the energy curves of both mixer tests against the test time (Fig. 3). The first test segment (0 - 2 min) shows the melting of the polymer component. The congruence of the measuring curves in this test segment confirms the good reproducibility of the measuring method.



Fig. 1: HAAKE Rheomix 600



Fig. 2: Cam rotors

In the second test segment (2 - 5 min) the filler was added in the test 1 (red curve). In test 2 the resin was used instead (blue curve). After this second component was mixed in and the torque value came to a constant level, it can be seen, that the compound with the filler generated a nearly 4 times higher torque.

After five minutes the mixer was opened again and the third component (the resin in test 1 and the filler in test 2) was added into the mixer. After about 2 more minutes the torque curves of both measurements are congruent again. This



Fig. 3: Mixer test torque and energy curve versus mixing time

means that the melt viscosity of both compounds is identical.

A comparison of the energy curves of the two test shows that the energy consumption of procedure 1 was about 40% higher than the energy consumption of procedure 2. For the production process this means that the incorporation sequence of procedure 2 would safe a significant amount of energy compared to the procedure 1.

A reprint of this paper is available on request by quoting LR-24 $\, \bullet \,$



• application notes & technical articles

Practical determination of the pasty sewage sludge flow curve on the basis of the main interactions characterisation

mining industries

chemical & allied industries

Due to the increases in sludge production resulting from an increasing volume of treated wastewater, the need for regulating the production and optimising treatment process become crucial.

A complete characterisation, chemically and mechanically is required for controlling process or designing parameters for storing or spreading operations. Rheological parameters are needed but rheometric tests often need sophisticated apparatus and require careful experimental procedures, which does not suit to in situ measurements inside a treatment plant. So, in order to quantify behaviours observed in real processes, engineers and operators have thus resorted to empirical tests that appear to correlate resorted to empirical tests that appear to correlate with full-scale operations. However, these tests are not often justified on a rigorous basis. The aim of this paper is to demonstrate rigorously that for pasty biosolids, we can determine the parameters of the flow curve without a rheometer, simply from the knowledge of the man interactions between the components (solid or liquid).

A full copy of this paper is available on request by quoting RF-010802-Fr-01 ●



application notes & technical articles Examining the influence of stabilisers on the flow characteristics of polyamide

polymer industries

During processing high temperatures and the presence of a nucleophile such as water lead to the degradation of polyamide.

This causes a decrease in polymer molecular mass and an increase in polymer end-groups. This finally results in a decline of the mechanical properties of the final product. It is possible to delay the onset of link decomposition by adding stabilisers.

The PolyLab system offers a quick and reliable method of examining the influence of stabilisers on material processing characteristics.

A full copy of this paper is available on request by quoting LR-18 \bullet





Further information is available by contacting **Rheology Solutions**:

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Controlling Enzyme Reaction Temperature with Circulator Baths – TC05-02e

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