#### feature article

### Rheology and micro-structures Close analysis of foam structure recovery

#### pharmaceutical, cosmetic & allied industries

An examination of the rheological behaviour of foam is mostly a descriptive exercise – the complex changes in the structure of the air bubbles that lie behind the behaviour of the foam are difficult to research. Using a rheometer fitted with a microscope unit and with the help of statistical image analysis software, Dr. Klaus Oldörp of Thermo Fisher Scientific has closely examined two very different cosmetic foams.

Products are designed to foam for a wide variety of reasons. In toiletries and personal care a foaming product is easier to dispense and also often feels more pleasant, imparting a sense of "something extra", so a foaming product can convey to the consumer a feeling of value or even luxury. Depending on the product, its application, and the type of packaging, the properties of foaming products can, however, vary enormously. An important characteristic is the mechanical stability of the foam when subjected to shear forces, which must be tailored to suit the way it is to be used or dispensed in order that the foam is not broken down during use.

Ideally a shaving foam, which is dispensed from an aerosol can, under pressure through a narrow nozzle, will be easily and pleasantly spread onto the desired area, and not run down the face. It should stay in place during shaving. These requirements are reflected in the rheological behaviour of a shaving foam. When subjected to shear forces the viscosity breaks down to a considerable extent, but then recovers quickly.

A fundamentally different behaviour pattern is expected from a cleansing foam delivered from a

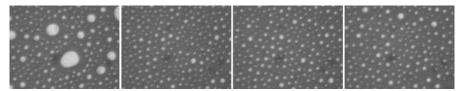
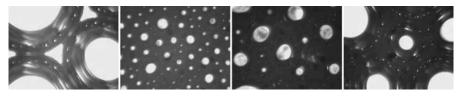


Fig. 1: The shaving foam [in the rheometer before being subjected to the shear action (after 30 s) and during the recovery period (after 80 s, 215 s and 510 s)



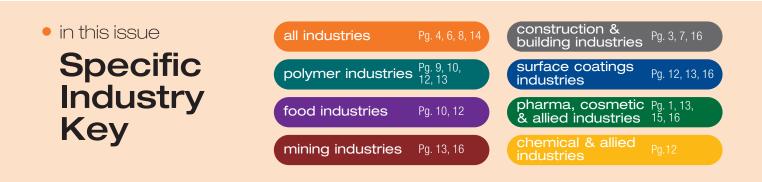
*Fig. 2:* The cleansing foam in the rheometer before being subjected to the shear action (after 30 s) and during the recovery period (after 80 s, 215 s and 510 s)



pump dispenser. When rubbed between the hands the product should foam up in order that the active cleansing ingredients can be spread evenly over as great an area as possible. The foam should also be as rich as possible, although after spreading it should not remain too stable, but should be easy to rinse off. In line with these requirements the rheological behaviour of a cleansing foam is guite different from that of a shaving foam. When the shear action begins its viscosity falls of considerably, then increases to a significant degree and after 15 seconds of shear action reaches a constant value. During the "structure recovery" period the viscosity falls to about one quarter, but over the ten minutes during which it was observed the viscosity remains above the viscosity of the original foam that has not been subject to shearing.

With the help of a modular HAAKE MARS Advanced Modular Universal Rheometer with plate/plate geometry a test was carried out to examine the structure recovery of different foams. The tests were discussed in detail with Rüdiger Brummer of Beiersdorf. The lift of the versatile rheometer, together with the sensitive normal force sensor, allows the measurement geometry to be closed in a gentle and controlled manner during sample loading. The generouslydimensioned lower receiver unit for additional instruments means that there is also room for the microscope unit used here – the Rheoscope module.

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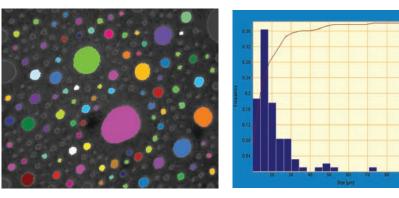


Fig. 3: Computer-aided analysis of the structure of the shaving foam before being subjected to the shear action (left: the air bubbles identified, right: the resultant size distribution)

#### ← continiued from page 1

The viscosity of the unsheared foam was first determined by oscillation measurement, and then the samples were subjected to shearing for 30 seconds at 1,000 revs per second. The progress of structure recovery was then closely followed over a ten minute period, once again using oscillation measurement. With these measurements we were able to confirm that the two foams under investigation exhibited quite different characteristics. What the rheological data do not tell us, however, is what causes this different behaviour.

#### Causes of structure recovery

Rheology is a purely macroscopic method, i.e. with rheology the sample is always treated as one homogenous piece of material, looked at "from the outside". In order to more closely determine the micro-structural causes of the different rheological behaviour patterns a RheoScope module was added to the Universal Rheometer. This offered the possibility of looking inside the sample during the rheological measurement process.

If one follows the shaving foam test with the microscope module the images are similar to those in fig. 1. The first image was taken of the still unsheared sample, 30 seconds after the beginning of the test. The second image shows the foam after 80 seconds, i.e. well after the end of the shear process. The third and fourth images show the sample during the continued recovery process, after 215 seconds and 510 seconds. It can clearly be seen that the shear process has reduced the size of the larger air bubbles and the foam sample has thus become homogenised. During the recovery period the air bubbles remain almost consistently small.

With the cleansing foam a completely different picture is seen (fig. 2). The original air bubbles in the unsheared foam have a significantly greater wall thickness. The diameter of the air bubbles is dramatically reduced by the shearing action. During the recovery period this structure breaks up and once again large air bubbles are formed, which do not, however, during the period of the test, reach the size of the original air bubbles.

This subjective impression, gained by observing the captured images, can be checked by computer-aided image analysis. Using the SPIP software from Image Metrology A/S, which was specially designed for image analysis, the air bubbles in the image detail were counted and their sizes calculated (see fig. 3).

In the shaving foam, before shearing, 93 percent of the bubbles were of a size less than 40 microns, with a noticeable peak at around 10 microns. The remaining 7 percent consisted of a few larger air bubbles, to just over 100 microns. The larger air bubbles were broken up by the shear action so that the biggest ones were now around 30 microns in size. The frequency peak was at 12.5 microns, but was not so noticeable as the peak prior to the shear action.

From the average size of the air bubbles it can be concluded that by shearing the shaving foam a more stable structure was produced, with bubbles on average about 1/3 smaller and a much tighter statistical distribution pattern.

The images of the cleansing foam were also analysed by computer. In comparison with the shaving foam the structure of the cleansing foam was much more significantly altered during the shearing process as the average bubble size fell to less than 1/10. This sheared structure is however, in comparison with the original foam, not stable, so that during the recovery period the bubbles ran together again and by the end of the test had once again reached about 50 percent of their original size.

#### Dr. Klaus Oldörp

Senior Product Specialist, Thermo Fisher Scientific, Karlsruhe, Germany

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A full copy of this paper is available on request by quoting PTE659 •

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#### news & views

## 2009 Rheology of Building Materials - Conference Report

Following is the report from the 2009 Rheology of Building Materials conference in Regensburg Germany. At the end of the report, a list of all the papers that were presented is provided. These can be supplied on request as PDF's from Rheology Solutions.

#### construction & building industries

## Rheological measurements on building materials

Building materials scientists coming from all over the world appeared not that impressed by the international crisis. Almost the same number of participants as last year found their way to Regensburg, in order to gather information about the results of the latest research in the field of rheology of building materials. Owing to the fact that there were speakers coming from six nations from India to Iceland and visitors from more than 10 countries, it was reasonable for the organisers – the Regensburg University and company Schleibinger – to speak of an international conference.

Peter Ramge<sup>1</sup> of BAM in Berlin (German Federal Institute for Material Research and Testing) presented the influence of different storage conditions of cement on the rheological properties of mortar. The hydration products on the surface of cement particles verified by means of an electron microscope do obviously have an influence on the slump flow and the rheologically determined yield point.

Dr. Stark<sup>6</sup> of the University of Weimar devoted her speech to sand as further starting material of mortar. Reason for her research project is the replacement of natural sand by crushed sand. With the aid of modern optical measurement systems, she was able to investigate beside the particle-size distribution, the particle shape and its influence on the yield point of mortar. The rheological measurements were carried out by using a basket probe according to Prof. Vogel.

By means of modern oscillation measuring systems, Steffen Schneider<sup>2</sup> of Knauf Gips KG investigated the behaviour at rest as well as the processing behaviour of plasters and joint compounds. The behaviour at rest in case of modern concrete is likewise of great interest. If mortar or concrete are not stable at rest, this leads to sedimentation of the aggregates. It is well known that a self-compacting concrete has to have as well thixotropic properties, if it shall flow easily and shall be stable nonetheless.

The effect of modern superplasticizers is depending, among other things, on the temperature. Dr. Golaszweski<sup>3</sup> of the University of Technology in Gliwice, Poland demonstrates that this effect often cannot be detected or may incorrectly be detected in cement pastes. Therefore, a rheological measurement on mortar is indispensable.

In Germany SCC is traditionally manufactured with a relatively high binder content. Florian Müller<sup>7</sup>, Innovation Center Iceland, presented a somewhat different mixture design with lower binder content. In this way, a SCC can be produced which flows faster, i.e. being low viscous but nevertheless stable.

The so-called geopolymer concrete presented by Prof. Malathy<sup>6</sup> of the Kongu Eng. College, India, does without any cement at all. For this concrete ash, in particular fly ash, is used together with a chemical activator as binding agent.

The actual concrete pressure when concreting SCC is still not definitely known, therefore the hydrostatic concrete pressure is generally taken as a basis. Carsten Bohnemann<sup>4</sup> of the RWTH Aachen has conducted comprehensive test series in this respect. Interestingly, there is no difference concerning the concrete pressure in case of high-viscosity and low-viscosity SCC. If the concreting speed is not too high, the pressures are even lower than in case of vibrated concrete. This, however, does not apply when pouring the concrete bottom-up by means of pumps. In this case, the hydrostatic pressure actually has to be taken as the basis.

Dr. Kumar<sup>®</sup>, Granit Constr. Company of Abu Dhabi, among other things presented the test methods applied in Abu Dhabi for steel fibre-reinforced SCC. The suitability of alkali-free accelerators for sprayed concrete so far may only be assessed in quite extensive spraying tests. Peter Iff of Bilfinger Berger AG, laboratory Munich, has developed together with the University of Nuremberg a laboratory testing method. Since the processes in this method are running extremely fast, classical rheometer and oscillation tests do not provide definite results. Therefore, further tests were carried out by means of the SEM cryoscopy. In this way, the hydration is interrupted by immersing in supercooled nitrogen. Afterwards, the samples are evaluated in the scanning electron microscope.

At the end of the event, Prof. Yilmaz<sup>9</sup>, Istanbul University, Turkey, presented the project of an underwater pipeline from the Turkish Taurus to Cyprus and expanding to the Middle East. For this purpose, a plastic pipe was anchored in a water depth of about 200 m floating on the bottom of the sea. Owing to many problems, as well in fluidic and rheologic respect, this project becomes an engineering challenge.

After a pleasant get-together and informal exchange of experiences in the evening, a laboratory workshop took place on the 12th of March imparting rheological principles and performing practical building measurements. The papers as outlined in the above report are available on request from Rheology Solutions as a PDF:

 Dipl.-Ing. Peter Ramge (speaker), Dipl.-Ing Wolfram Schmidt, Dr.-Ing. Hans-Carsten Ku.hne,

"Influence of different storage conditions on the rheological properties of cement based materials."

A copy of this paper is available by quoting reference no: Schl - 011

2. Dipl.-Ing. Steffen Schneider Knauf Gips KG, Iphofen

"Material parameters on rheological studies of plasters and joint compounds"

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## A copy of this paper is available by quoting reference no: Schl – 010

 Dr. Ing. (habil.) Jacek Golaszewski Politechnika Slàska Gliwice, Schlesische Technische Universität Gleiwitz Katedra Procesów Budowlanych, Lehrstuhl für Bauprozesse

"The effect of temperature on rheological properties of cement paste and of fresh self compacting concrete." *A copy of this paper is available by quoting* 

reference no: Schl - 009

- Dipl.-Ing. Carsten Bohnemann Institut für Bauforschung (ibac) der RWTH Aachen, Arbeitsgruppe Bindemittel "Influence of the Rheological Properties on the Fresh Concrete Pressure" A copy of this paper is available by quoting reference no: Schl - 008
- Prof. Dr. R. Malathy Kongu Engineering College, Dept. of. Civil. Engineering ,Perundurai, Tamil Nadu, India, "Properties of Fresh and Hardened Geopolymer Concrete" A copy of this paper is available by quoting reference no: Schl - 007
- Dr.-Ing. Ursula Stark (speaker), Dipl.-Ing. Katrin Ostheeren Lehrstuhl Aufbereitung von Baustoffen und Wiederverwertung an der Bauhaus-Universität Weimar "Rheology of SCC-Mortars with sands of different shape" A copy of this paper is available by quoting

reference no: Schl - 006

- Dipl.-Ing. MSc. Florian V. Müller Innovation Center Iceland (ICI), Concrete Division "Rheology of Iow-binder SCC" A copy of this paper is available by quoting reference no: Schl - 005
- Dr. P.T. Sandrosh Kumar Granite Construction Company, Abu Dhabi, UAE
   "The Influence of Steel Fibres on the Rheology of Self Compacting Concrete" A copy of this paper is available by quoting reference no: Schl - 004
- Prof. Dr. L. Yilmaz (speaker), Prof. Dr. N. Mestanzade Atakoy Campus, Istanbul Kultur University und Technical University of Istanbul, Civil Engineering Faculty, Hydraulic Division
   "Influence of vertical vibration on support on the dynamic stability of subsea pipelines." A copy of this paper is available by quoting

4 talk

reference no: Schl - 003 •

#### New product

### Recirculating chiller series extended to include 7,500W & 10,000W cooling capacities

#### all industries

There have been two product configurations added to the Thermo Scientific NESLAB ThermoFlex<sup>™</sup> Series of recirculating chillers. Units are now available with cooling capacities up to 10,000 watts.

The ThermoFlex chiller incorporates a new, patented recirculation system with an integrated funnel, full flow filtration, and visual fluid level indication for easy maintenance. Design innovations include an integrated ramp that enables one person to unpack a unit, air and water filters that can be changed while the unit is in operation, as well as a quick-start guide.

A wide variety of options and cooling capacities that can be configured for diverse applications in markets such as industrial, medical, laser, metrology, packaging, pharmaceutical, and semiconductor processing.

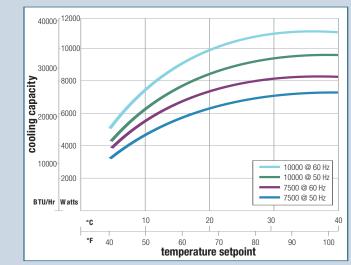
#### Features and Benefits

- Sight tube with level indicator for instant visual awareness of fluid level
- Robust refrigeration and recirculation systems ensure reliable performance and long life
- Installation Flexibility: Can be operated with 2 sides blocked for flexibility when installing



NESLAB ThermoFlex™ Recirculating Chiller

- Flexible options to promote chillers tailored for specific process requirements. Some of the available options include: Water Cooled or Air Cooled Condenser, Standard or High Temperature, Integrated DI, Flow and Pressure Control, SS/Plastic, Auto Refill
- Improved efficiency: up to 10% greater cooling capacity than previous models
- Improved noise quality ensures a pleasant work environment.



Cooling Capacity for NESLAB ThermoFlex 7500 & 10000

Cooling capacity based on units with P2 pumps with no backpressure. Other pumps will affect cooling capacity performance.

#### Options include:

Feature	Benefit		
Pressure Relief	The pressure relief valve allows the user to set the maximum fluid pressure to meet the		
	application requirements and is available as an internal or external option.		
Pressure Relief	The pressure relief valve allows the user to set the maximum fluid pressure to meet the		
with Flow Readout	application requirements. The Flow Readout allows for monitoring the flow rate to the		
	application via controller readout.		
Flow Control	The flow control valve allows the user to adjust the flow to the application. The flow		
with Flow Readout	readout allows for monitoring the flow rate to the application via controller readout.		
Auto Refill	Allows for automatic refilling from a customer-supplied water source to ensure the proper		
	fluid level is maintained.		
Anti Drainback	Prevents fluid from flowing back to the reservoir when the chiller is installed below		
	the application.		
DI Water	Partial flow internal DI cartridge minimizes footprint and provides fluid resistivity		
	between 1 and 3 mOhm.		
RS232 & RS485	Provides digital communication for remote operation, monitoring and data logging.		
Digital Communication			
Analog I/O	Provides analog communication for remote operation and monitoring. Includes a remote		
	sensor port which allows for remote temperature control of an application when used with a		
	remote sensor (available as an accessory).		
Global Voltage	Allows the user to select the appropriate frequency and voltage to enable operation anywhere		
	in the world.		
Air-Cooled Condenser	Uses ambient-temperature room air to remove application heat.		
Water-Cooled Condenser	Uses facility water to remove application heat.		
SEMI S2 Compliance	Compliant with S2-0703, S8-0705, S14-0704, F47-0706.		

#### Specifciations:

		NESLAB ThermoFlex 7500	NESLAB ThermoFlex 10000
Setpoint Temperature Ran	ge	+5°C to +40°C	+5°C to +40°C
Ambient Temperature Range		+10°C to +40°C	+10°C to +40°C
Temperature Stability		±0.1°C	±0.1°C
Setpoint Cooling Capacity			
	50 Hz at +20°C	6425 W / 21910 BTU	8500 W / 28985 BTU
Reservoir Volume		17.9 liters	17.9 liters
Refrigerant		R407C	R407C
Physical Dimensions (H x	W x D)		
	Air-Cooled	132.7 x 63.9 x 85.6 cm	132.7 x 63.9 x 85.6 cm
	Water-Cooled	116.6 x 63.9 x 85.6 cm	116.6 x 63.9 x 85.6 cm
P2 - Positive Displacemen	t Pump		
	50 Hz 3.3 gpm @ 60 psig	12.5 lpm @ 4.1 bar	12.5 lpm @ 4.1 bar
P3 - Centrifugal Pump**	50 Hz 10 gpm @ 20 psid	37.9 lpm @ 1.4 bar	37.9 lpm @ 1.4 bar
P5 - Centrifugal Pump**	50 Hz	75.7 lpm @ 2.4 bar	75.7 lpm @ 2.4 bar
Unit Weight	(by pump type)	P2-AC: 356 lb (161.5 kg)	P2-AC: 356 lb (161.5 kg)
		P2-WC: 315 lb (143 kg)	P2-WC: 315 lb (143 kg)
		P3-AC: 372.5 lb (169 kg)	P3-AC: 372.5 lb (169 kg)
		P3-WC: 331.5 lb (150 kg)	P3-WC: 331.5 lb (150 kg)
		P5-AC: 405.5 lb (184 kg)	P5-AC: 405.5 lb (184 kg)
		P5-WC: 364.5 lb (165 kg)	P5-WC: 364.5 lb (165 kg)
Voltage Options	400 V/50 Hz/3 phase	Available	Available
400-460 V/50-60 Hz/3 phas	e Global Voltage	Available	Available
Standard Compliance		(for all ThermoFlex recirculatin	g chillers)

Specifications obtained at sea level using water as the recirculating fluid, at a  $+20^{\circ}$ C process setpoint,  $+25^{\circ}$ C ambient condition, at nominal operating voltage. Other fluids, process temperatures, ambient temperatures, altitude or operating voltages will affect performance. Cooling capacity based on units with P2 pumps with no backpressure. Other pumps will affect cooling capacity performance. Specifications subject to change.

\*\*Pressure values for centrifugal pumps are differential pressures between the inlet and the outlet of the unit.

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## Key Features:



Patented full flow filter ensures clean fluid to protect your application and maximize recirculation system life.



Easily removable condenser grill and air filter allow for quick and simple cleaning to optimise chiller performance and maximize component life.



Patented integrated funnel design allows for spill proof filling.



Easy-to-view controller with two display options



#### news & views

### Temperature Control Systems Sealed systems vs open bath circulators

#### all industries

Thermo Scientific HAAKE DynaMax<sup>™</sup> temperature control systems dissipate heat from exothermic, chemical reactions much faster than other products in its class. The system is designed to provide high performance and safety with greater ease of use and lower maintenance requirements.

The architecture of a closed system provides many enhanced safety features for the user, the application and also the environment. Following is an overview of the HAAKE DynaMax range of closed temperature control systems.

- Sealed architecture prevents the creation of fumes at high temperatures
- No unpleasant odours in the lab
- No oily film

- No flammable fumes
- Longer fluid life
- No hot surfaces at high setpoints (other than the hose connections)
- Limits the likelihood of receiving a burn
- Prevents damage to the controller
- Sealed architecture prevents condensation forming and getting into the fluid. Condensation can:
- Cause instability and loss of cooling performance
- Cause overflow as it adds to the fluid volume
- Raise the freeze point when using glycols or alcohols
- Create a frozen, time consuming mess when using silicon oils
- More powerful pumping than bath circulators
- Up to 25 lpm and 1.5 bar (with water)

- Up to 20 lpm and 1.4 bar (SIL 180)
- Longer fluid life, lowers the cost of ownership
- Hot fluids in contact with air limits fluid oxidation
- Cold fluids do not contact air therefore do not get fouled with atmospheric moisture
- Eliminates the need for nitrogen purging
- Less downtime required resolving fluid issues

HAAKE DynaMax<sup>™</sup> is suitable for high-end laboratory & pilot applications that combines rapid temperature changes with incredible stability.

Typical applications include:

- Jacketed reactor vessels
- Large rotary evaporators
- Reaction calorimetry
- Fermentation
- Analytical instrumentation cooling •

#### Technical Data:

Specifications	HAAKE DynaMax 1200	HAAKE DynaMax 1700
System Performance		
Temperature Range	-40°C to 150 °C	-45°C to 150 °C
Cooling Capacity at 20 K (50 Hz)	1.2 kW	1.7 kW
Cooling Capacity at 0 K (50 Hz)	900 W	1.25 kW
Cooling Capacity at -20 K (50 Hzz)	250 W	550 W
Cooling Capacity at -40 K (50 Hz)	-	150 W
Heating Capacity (230 V - 50 Hz)	2 kW	2 kW
Temperature stability	+/- 0.01 K	+/- 0.01 K
Pump Performance (with silicon oil)		
Maximum Pump Pressure (bar)	1,2	1,2
Maximum Flow Rate	20 l/min	20 l/min
Pump Performance (with water)		
Maximum Pump Pressure (bar)	1,5	1,5
Maximum Flow Rate	25 l/min	25 l/min
Electrical Performance		
Power Requirements	230 V / 50 Hz - 1 phase	230 V / 50 Hz - 1 phase
Maximum Current	16 A	16 A
Communication	RS232, RS485	RS232, RS485
Display	128 x 64 pixel	128 x 64 pixel

NOTE: Technical data may be changed without notice





НААКЕ Дупатах™



#### new product

## Viskomat XL, rheometer for mortar and fresh concrete

#### construction & building industries

The Schleibinger Viskomat XL, Rheometer for Mortar and Fresh Concrete up to 6 mm grain size.

Based on 20 years experience with rheometers for mortar and fresh concrete, Schleibinger has developed a new instrument called the Viskomat XL. It fills the gap between the Viskomat NT for mortar and paste with a specimen volume of 360 ml and the concrete rheometer BT2 with a sample volume of 201. The operating principle of the Viskomat XL is nearly the same as for the Viskomat NT. A mixer formed probe measures the torque, while the specimen vessel rotates. An additional scraper cleans the wall of the vessel.

#### Dynamic speed range

The speed may be varied from 0.001 to 220 rpm in a clockwise or counter clockwise direction. You may define the speed in several steps, in a linear increase or decrease of speed. As an option an oscillating or a logarithmic mode is available.

#### High Torque Range

The Viskomat XL has a torque range from 0..300 Ncm with a resolution of 0.05 Ncm and accuracy better than 0.2 Ncm. Optionally the rheometer can be supplied with a sensor taking the torque range up to 1000 Ncm and accuracy to 0.8 Ncm.

#### Temperature control

Temperature control is achieved with a double wall specimen container, where a cooling liquid circulates around the outside. The specimen temperature is measured with a RTD mounted inside the shaft of the probe.

#### High Time Resolution

The sampling rate may be set from  $0.005s \ .. \ 10min.$ 

#### Shear Stress Control Mode

As an option, you may run the Viskomat XL in shear stress controlled mode. The torque is pre-set over time, and the speed is automatically controlled to achieve the predefined torque.

#### Software

The Viskomat XL is controlled via a network interface. Simply connect the rheometer to your PC and start Internet Explorer for full control and data transfer. No special software installation is necessary.•

Schleibinger Viskomat XL - Concrete Rheometer

#### Technical Data:

Application	Rheometer for mortar paste & fresh concrete to 6mm
Net. size (h x w x d)	920 x 315 x 410 mm
Specimen Volume	3I, others on request
Motor speed range	0.001220 rpm
Motor torque	1200 Ncm
Motor angle meas. accuracy	better $\pm 0.05^{\circ}$
Meas. torque range	±0300 (1000*) Ncm
Meas. torque resolution	0.05 (0.2*) Ncm
Meas. torque accuracy	0.2 (0.8*) Ncm
Temperature sensors	RTD PT100 1/10 DIN B
Cooling and heating	double wall vessel
Controller	extra cabinet, 110120V, 5060Hz, 800W
Controller interfaces	4 x USB, 1 x network
User interface	Coloured screen with online graphics
Interfaces	Ethernet /TCP/IP for control and data recording
Weight	ca. 90 kg

NOTE: Technical data may be changed without notice





#### industry applications

## On-line optical QC measurements for manufacturing industries

#### all industries

#### Introduction

Production quality and uniformity in manufacturing and processing is closely related to good Quality Control (QC) during the manufacturing process. On-line systems are capable of delivering solutions for QC monitoring.

In particular this technical note deals with optical quality control, for raw materials or final products used in pellet or free flowing powder form, or for sheets and films of various opaque, semitransparent and transparent materials. These online systems are based on optical closed circuit cameras, and are used to observe the powder/pellets or film/sheet. Comprehensive software can analyse the images, recognising and cataloguing user-defined abnormalities, complete with images, sizes and user defined classifications, including tears, scratches, holes, discolourations, as well as dimensional irregularities for pellets and so on. The system can be trained to recognise and catalogue any defect seen by the camera, and the camera can be used in either transmission mode (for transparent films) or reflection mode (for opaque films, or a combination of both).

These on-line systems provide streamlined data, showing changes in important QC criteria for the product during the process. In this way corrective action can be quickly taken, minimising potential for waste and reprocessing.

#### On-line optical QC

The optical properties of many materials are a crucial indication of their quality. Optical properties of raw materials like granules or powders can be important in dictating the quality of the materials they are used to manufacture. Any discolouration, dimensional irregularities or contamination can adversely impact on the quality of the manufactured product. On-line analysis of some, or all, of the pellet or powder stream for these defects can prevent poor quality product entering the manufacturing process, and reduce the wastage therein. On-line CCD camera systems with high performance computers and

software are becoming increasingly important in the recognition, recording and removal of these impurities before they can harm the product or the reputation of the manufacturer.

Once a sheet or film of product has been formed, it is important that it is of high quality with low levels of impurities (eg. insects), discolourations (fish eyes, black spots etc), pinholes, tears, surface defects etc. Poor quality control in these areas can lead to customer returns, dissatisfaction and reduced shelf-life of the product. Using CCD technology, along with sophisticated image processing and analysis software, the quality of a film or sheet can be continuously monitored and impurities detected. The size, colour and shape of the impurities which are important can be defined by the user.

Optical Control Systems gmbh (OCS) manufacture cutting edge optical QC equipment for pellets, powders, films and sheets for manufacturing industries.

A full copy of this paper is available on request by quoting Rheo053TP •





www.rheologysolutions.com

#### industry applications

### Polymer and polymer process characterisation for applications based material and extruder optimisation

#### polymer industries

Polymer manufacture today is one of the most important world-wide industrial sectors and, significantly, is not confined in the centre of manufacture to one or two highly industrialised nations only. In contrast, a large number of diverse nations have resources, which are based on a variety of raw materials, including rubber, crude oil, and recycled materials. Associated with this, many countries also have a large capacity for polymer production. Clearly an industry such as this can not easily be summarised and no two polymers are produced under exactly the same processing conditions.

Often many thousands of tonnes of product can be wasted by poor or inappropriate set-up of process equipment, in particular when novel products are being produced for the first time. Good research and development and quality control requires that the material being manufactured meet often quite stringent criteria, and that processing conditions be optimised for that material. This can be achieved through good materials characterisation techniques and through sensible pilot scale testing before full production commences.

Material characteristics, which play a crucial role in polymer processing are often rheological such as viscoelasticity, viscosity etc and dictate how the molten polymer will flow and deform in response to forces exerted by the screw, at the die, in the mould, during and after cure etc. These properties can be measured using a controlled stress rheometer with high temperature capability. They help the manufacturer predict the propensity for the final product to exhibit surface defects (especially at high throughputs), as well as giving indications of the optimal conditions for Compatibilisation etc. Other rheology-based techniques such as MFI, MVI etc have long been in use, but have often been shown to be weak with respect to the quantity and quality of the data they provide. The power and ease of collection of information provided by a rheometer far increases the capacity of the engineer, either as a tool for QC, R&D or for assessing the possibilities directly during production.

Recent advances in extrusion technologies have allowed ever smaller units to be constructed for



Thermo HAAKE PolyLab OS – Torque Rheometer

small scale production as well as for R&D. These small units allow an engineer to assess the possibilities for production of a new polymer product by altering process variables, like barrel and die temperature profile, or screw speed and collecting the resulting product. Later analysis of these products can tell the engineer whether or not a sample is likely to meet specifications when produced on a larger scale. Torque sensors on the screws of small-scale extruders give an idea of the relative processability of 12 different mixtures. These extruders have sufficient instrumentation (temperature, torque, screw speed) and flexibility (multiple temperature zones, screw speed, segmentation of screws for alteration of different zones within the machine. and alternate die geometries) to allow the user to truly research and evaluate different scenarios. This flexibility, combined with the extensive postex take-off systems also allows these machines to be used for production on a small scale, often for low volume, high value products, or when several small batches of different products are required.

Combining laboratory- and pilot-scale instrumented machines and post-ex options with high grade rheological measurements for research and guality control provides a good opportunity for engineers and scientists to take full advantage of the increasing trend for specialised polymeric products. As other technologies mature further and, for example, lighter, stronger more flexible polymers are required to replace current generation materials, the need for good (and relevant) materials characterisation and process optimisation, as well as small scale, niche production of novel or improved polymers will grow. The combination of rheometry and small-scale extrusion equipment is certainly playing, and will certainly play, a big part in these developments.

A full copy of this paper is available on request by quoting Rheo054 •



#### industry applications

## QC measurements for liquid food products and packaging

#### food industries

#### polymer industries

#### Abstract

Production quality and uniformity in food processing and its subsequent packaging is closely related to good Quality Control (QC) during the manufacturing process. In order to achieve high quality merchandise with minimal production losses, good process control and monitoring is critical. This is true for production of both the food product itself, the packaging, and of course for putting the former inside the latter efficiently. Laboratory and on-line systems are capable of delivering solutions for QC monitoring, and in cases where extra information (related sometimes to product development, and not to QC) about the flow properties are unnecessary, an on-line option can be the most useful.

On-line systems often provide streamlined data, showing changes in more limited criteria for the product during the process. In this way corrective action can be quickly taken, minimising potential for waste and reprocessing. Laboratory

instruments, designed for guick data collection (only a few seconds or minutes) and intuitive understanding of the data can also be useful in this respect where no proven on-line alternative exists. On-line monitoring of shear viscosity is widely accepted for many kinds of materials including for liquid foods, and molten polymer packaging. In-pipe or in-tank probes can closely monitor the shear viscosity of the material in-situ, allowing operators and engineers to take appropriate action to maintain the specifications of the food product. These probes can also be mounted at the die end of an extruder, to monitor the viscosity of molten polymer before the moulding of packaging. Post-extrusion, the quality of the packaging material can be monitored optically to detect, catalogue and notify when user defined flaws exist in the packaging material (eg fish eyes, pin-holes, dark/light spots etc). For filling a liquid food into a package (by pouring, squirting, spraying etc), extensional viscosity often dominates the process. Unfortunately, there is no proven on-line method for monitoring the extensional viscosity of foods as they are dispensed from above into their packaging. Fortunately a novel, simple and quick laboratory technique has been developed for exactly this purpose - measuring the relative impact of the extensional properties of a liquid. In this way the

product and the packaging can be monitored online right through the process, until the product has safely been deposited in its packaging.

#### Introduction

Several products exist today for testing in the laboratory, both for QA and for product development. Laboratory testing for product quality in the food industry can include rheometers and viscometers, to measure liquid flow properties such as yield stress, viscosity and elasticity under different shear flow conditions. These instruments can also measure the flow properties of packaging materials, eg molten plastics etc, to ensure that they can be properly moulded etc. Testing of solid-like properties in the laboratory is possible for both foods and their packaging with uniaxial testing. Miniature uniaxial testers - texture analysers - can be used to objectively quantify textural properties of a food, while units with larger capacities can also be used to investigate the tearing, stretching, crushing etc of the finished packaging product. Often, during filling, a liquid is poured, squirted, sprayed or otherwise dispensed into the package. For some materials 'stringy' strands hang from the nozzle and soil the packaging, necessitating an extra process step to clean the packaging. This stranding is related to the extensional viscosity of the material (completely different to the shear viscosity measured by rotational viscometers). Recently developed techniques now enable these extensional properties to be properly measured in the laboratory using extensional rheometers.

Laboratory analyses are good solutions for material characterisation and in many cases can not be otherwise replicated on-line. However, during production it is sometimes preferable to have an on-line system for QC, so that any deficiencies in the product or its packaging can be detected as quickly as possible. This technical note seeks to explore and to explain on-line

Marimex ViscoScope Process Viscometer



viscosity monitoring, on-line quality control for packaging film and ingredients and a novel laboratory technique for monitoring the extensional properties of liquids to diagnose excessive stranding.

## Process viscosity QC measurement

Using the torsional principle for shear viscosity measurement, bulb-shaped probes can be mounted in-the-pipe or in-the-tank, and arranged so that the data is directly accessible to the operator through a digital display, a PLC interface, or standalone software. The principle of operation of these instruments is that they vibrate at a fixed, known frequency in the flow field. The damping effect of the liquid on the vibration is related to its viscosity and is measured. The relative flow rate of the material around the probe does not usually effect the reading, except indirectly because high shear rates (analogous to high flow rates in these cases) can have an effect on the fundamental structure of the pumped material. The probe sensor can have its dimensions and materials of construction designed to suit the process in which it is used. For example, small surface area probes allow highly viscous materials, like polymer melts, to be measured, or larger surface area probes can be utilised for lower viscosity materials, like liquid foods and beverages.

The probes measure at a single shear rate only (usually only a few s-1), based on the single frequency of vibration, but this need not be a limitation for QC applications since a change in the material is often all that needs to be detected. The probes have to be rugged, reliable, and low maintenance because of their continuous use and the nature of the environments in which they are used.

#### On-line optical QC

Once a polymer product has been formed, it is important, especially for food applications, that the packaging is of high quality with low levels of impurities (eg insects), discolourations (fish eyes, black spots etc) and pinholes. Poor quality control in these areas can lead to customer returns. dissatisfaction and reduced shelf-life of the product. Using CCD technology, along with sophisticated image processing and analysis software, the quality of a film can be continuously monitored and impurities detected. The size, colour and shape of the impurities which are important can be defined by the user. Similar CCD techniques can be used to monitor raw materials in granular form for their size, shape and any impurities. This kind of analysis can be used both for granular polymeric feedstock to an extruder during the manufacture of packaging, and also for granular free flowing food materials.



Optical Control systems - FSA100 Film Test

## Extensional viscosity measurement for QC to predict stranding

Often during the filling process, a liquid product will be 'stringy', causing difficulties with filling it into packaging. These difficulties include product losses due to strand formation, soiling of the packaging, necessitating cleaning, and sometimes incorrect dosage of the product into the packaging. For most filling, spraying and atomisation applications the extensional viscosity is extremely important, and dominates the occurrence of phenomena like strand formation. Extensional viscosity is not usually related to shear viscosity, and can not be measured by a rotational instrument. Extensional properties must be measured in extension. There now exists a



Thermo HAAKE CaBER 1 – Extensional Rheometer

relatively guick and intuitive extensional viscometer to quickly measure extensional properties. These measurements can take fractions of a second to perform if the material has low extensional viscosity, and the total time for the measurements can be as little as a minute or so. The principle off measurement is that the changing diameter of a strand of liquid until it breaks is related to surface tension and to extensional viscosity. A small sample of the material is placed between two plates, stretched so that a strand remains and the changing diameter with time taken until the strand breaks is measured. A simple measure of the time to break of the strand can immediately tell the user if the product meets its QC criteria. More detailed analysis, calculation of the extensional viscosity etc can also be achieved relatively simply using software if necessary.

#### Summary

This note outlines some of the less known (compared with laboratory analyses using rotational viscometers and rheometers) techniques for QC in the food industry. The torsional principal for on-line viscosity measurement is not a new one, but has been overshadowed by rotational techniques because of their flexibility in the lab. Rotational techniques are extremely difficult to implement on-line, and so often single-point torsional measurements can suffice for on-line QC applications.

Similarly, optical techniques have been outlined for QC. These types of tests can not be performed in any other way. The availability of powerful computers with purpose designed software, have enabled off-the-shelf lighting and high-end camera components to be combined to provide a powerful new tool. Understanding the data from these tests is intuitive, since the results are actual pictures, collected in real time, of impurities in pellets or film. Efficient software can also allow these defects to be displayed statistically.

Finally, the first commercially available easy-touse extensional rheometer has been outlined, along with some applications and techniques for measurement. In common with optical techniques, extensional properties can not be measured in any way other than in extension. Rotational instruments can not measure this property.

It is outside the scope of this note to discuss ubiquitous laboratory based rheological measurements for liquid and solid materials, such as rotational viscometry, or uniaxial testing. Rather, three more unusual techniques have been outlined for the information of the interested reader.

A full copy of this paper is available on request by quoting Rheo051 •



## industry applications Measurements at higher pressures

chemical & allied industries

food industries

#### polymer industries

This application package – HAAKE RheoStress 6000 "Measurements at higher pressures" has been created for the determination of the pressure-dependent rheological characteristics of a sample.

The universal rheometer Thermo Scientific HAAKE RheoStress 6000 is designed to perform rheological tests in CR (controlled rate) mode as well as CS (controlled stress) and CD (controlled deformation). Thanks to a liquid or electric temperature control unit, precise and fast temperature control of the sample can be achieved. Different versions of pressure cells are available: a version for standard applications made of stainless steel as well as a Hastelloy version to test chemically aggressive samples. The standard version can be pressurised up to 400 bar using a suitable pump whereas the upper pressure limit of the Hastelloy version is 70 bar. Both pressure cells are designed for temperatures up to 300 °C. A variety of measuring geometries is available for different applications: cylindrical rotors with different diameters for homogeneous samples and vane rotors for suspensions with larger particles [1].

With the configurations described above, the pressure and/ or temperature dependency of the sample can be easily monitored, e.g. the rheological behaviour of crude oil and drilling fluids as well as food products can be monitored directly under real-life conditions to improve performance in the respective application.

Advantages at a glance

- Universal rheometer Thermo Scientific HAAKE RheoStress 6000 for standard applications in QC as well as R&D
- Easy-to-use, multilingual HAAKE RheoWin software for beginners and experts
- Solid material measuring geometries with high resistance
- Low torque measurements thanks to stateof-the-art sapphire-bedded measuring geometries and software controlled torque correction



 High torque measurements thanks to a strong magnetic coupling between motor and measuring geometry

A copy of this application package note is available by quoting reference no: D-002

#### Literature

[1] Thermo Scientific Product information P21 "Vane Rotors for pressure cells for HAAKE MARS and HAAKE RheoStress 6000", Cornelia Küchenmeister, Klaus Oldorp

A copy of this literature is available by quoting reference no: P21 •

## industry applications Rheological characterisation of paints and inks

#### surface coatings industries

This package has been created for the rheological characterisation of paints and inks. The universal rheometer Thermo Scientific HAAKE RheoStress 6000 is designed to perform measurements in CR (controlled rate), CS (controlled stress) and CD (controlled deformation). The integrated normal force sensor allows the measurement of positive and negative normal forces. Thanks to the liquid temperature control unit using a separate circulator, a precise and constant temperature control of the sample can be achieved. A cone with a diameter of 20 mm suitable for measuring higher viscosities has been selected.

With this configuration measurements such as thixotropy and structure recovery tests can be carried out. The sensitive and precise normal force sensor can be used for reproducible gap setting and for keeping the gap constant during the measurement. The ''tackiness'' of a sample can be determined by measuring the negative normal force which is needed to lift a measuring geometry resting on the sample upwards.

For measurements on UV-curing paints a UV curing cell as optional accessory is recommended [1].

Advantage at a glance

- Universal rheometer Thermo Scientific HAAKE RheoStress 6000 for standard application in quality control as well as research and development
- Normal force sensor for measurements of positive and negative normal forces
- Easy-to-use, multilingual HAAKE RheoWin software for beginners and experts
- Lower measuring plate for an optimal gap filling
- Measuring geometry made of titanium with low inertia

A copy of this application package note is available by quoting reference no: D-001



#### Literature

[1] Thermo Scientific Product information P13 "UV curing cell for HAAKE Rheometers", Cornelia Küchenmeister, Fritz Soergel

A copy of this literature is available by quoting reference no: P13 •



## application notes Rheology of lubricating greases

#### mining industries

#### surface coatings industries

#### pharmaceutical, cosmetic & allied industries

The primary objective of lubrication is the separation of the rubbing surfaces by a layer which is more easily deformable than the material of the rubbing bodies (Bondi, 1960). Consequently, the rheological characterisation of the materials used for lubrication is extremely important.

These materials have been traditionally divided, from a rheological point of view, into fluid and gelled lubricants. The lubricating greases belong to the latter group, and they have been differentiated from the fluid lubricants because of the characteristics finite yield stress. That is a stress value below which the material behaves as an elastic solid. Its magnitude has been considered sufficient to prevent loss of lubricant under operating conditions, but not such as to offer significant resistance to the motion of the rubbing surfaces (Bondi, 1960). However, there is growing evidence that concentrated systems, such as lubricating greases, flow in the limit of very low stresses (Barnes and Walters, 1985; Barnes et al., 1989). Nevertheless, the concept of yield stress, from an engineering point of view, is still interesting and widely used (Astarita, 1990).

The lubricating greases may be considered as colloidal suspensions. They are obtained from the dispersion of a thickener solid (metal-soap molecules) in a lubricant liquid, such as mineral or synthetic oil (Sacchettini et al., 1985). The rheological behaviour of these materials is related to their microstructure, a three-dimensional network created by the interactions among fibres, spheres or rods resulting from the combination of metal-soap molecular chains. The formation of this three-dimensional network explains the viscoelasticity and the non-Newtonian flow behaviour of these materials.

The overall objective of this paper is to summarise the viscous and viscoelastic behaviours of lubricating greases, as well as to describe the experimental techniques employed and to discuss the problems that may arise from the different tests used to study its complex rheological response.



Thermo HAAKE RheoStress 6000 – Universal Rheometer

A copy of this application note (an oldie but a goody) is available on request by quoting reference no: HL-036E •

#### application notes

## How to relate test results of a torque rheometer to problems in elastomer processing

#### polymer industries

High quality standards of elastomers, elastomer compounds and their final products have to be realized at the lowest possible price, because the competitive situation and the requirements have increased extremely during the past eight to ten years. This leads to the necessity of optimizing the development process from the compound design to the presentation of the new product. All activities surrounding compounding (design, mixing and extrusion) are very costly and time consuming.

#### E.g. "How often do you have to run a compound on a production scale machine until it suits your specifications and processing capabilities?"

As a result, more and more manufacturers have started to bring compound- development and -processing closer together and are actively looking for methods of linking laboratory- scaled test results with production experience.

To meet these requirements test methods and development tools have to be meaningful and

process-related. The documentation of test results and a comparison to accepted standards and tolerance levels is a must in order to meet the requirements stipulated by different quality standards like e.g. SPC, ISO 9000 ... In the complete technical report an overview of different test methods gives you an idea how torque rheometer test results can solve your production problems.

A copy of this laboratory report is available on request by quoting reference no: LR-29 •



#### application notes

## Instrument application note: Current possibilities for extrusion and mixing

#### all industries

Applications for extrusion are not confined to any single industry sector or scale of operation. They range from mega- to pilot-scale operations in industries as diverse as master-batch and compound manufacture in the polymer processing sectors, melt mixing of resins for powder coatings, continuous mixing in the food and pharmaceutical industries to fabrication, for example of rubber seals or plastic rods. To successfully carry out these operations efficiently. extruders must be capable of imposing a variety of conditions along the length of their barrel, depending on the application. Product specific requirements can include distinct heating and/or cooling zones, the facility to add metered quantities of extra ingredients (solid or liquid) at any point along the barrel, gas take-off or vacuum ports, and mixing, transport, hold-up etc zones as required. Extrudate may be in the form of pellets rods, sheet, film, etc and ancillary operations include premixing, blown films, cooling and pelletising.

Not only are the applications and requirements of extrusion diverse, so also are the physical quantities of product required. This depends on a number of factors, the foremost of which are the nature of the product, the nature of the business, and the differences in requirements between full production and product/process development or R&D.

High-end small- or pilot-scale equipment is invaluable for both small-scale production needs and for testing new ideas and the processability of, or conditions necessary for, new or improved production techniques. Current technology boasts small- and pilot-scale twin screw extruders (TSE's) with segmented barrels and screws allowing flexibility through configuration changes in both. The scales of operation include 16mm. 24mm and 36mm TSE's and the materials of construction are variable depending upon the application. Food and pharmaceutical applications often require specialised barrel and/or screw construction (from high grade stainless steel for example). These extruders may be bench-or skidmounted depending on their size, and screw L/D can range from as low as 14:1 to as high as 40:1, depending on user requirements.

For both R&D and production, it is important to measure and control process variables at the



Figure 2: PRISM EuroLab featuring sheet line take-off system

motor and along the shaft. Today's technology allows precise control over motor torque and speed (and so power consumption and energy input), temperature profiles throughout the barrel, as well as the addition of extra ingredients or venting of waste gasses during the process. This is most often done through touch-screen controls, or by PLC control. In this way the effects of altering process conditions may be easily and cost effectively assessed, using small quantities of material, prior to larger-scale production.

For small-scale operations, the process may be precisely controlled and modified to take account of new conditions or changes in the quality or quantity of the raw materials.

Cleaning and maintenance are often important issues, especially when more than one product is processed using a single extruder. In these cases, horizontally split barrels, as illustrated by the example in figure 1, with easy opening allow quick access and inspection, as well as ease of cleaning.

Because compounded products are often converted into rods, sheet or film, extruders can be fitted with rod, sheet, or film dies for direct



Figure 1: PRISM 36:1 extruder showing open, horizontally split, barrel for easy cleaning and inspection.

conversion of the product. This saves time and energy by removing extra processing steps. Figure 2 illustrates this concept, showing a typical take-off arrangement, in this case, a sheet takeoff system, in combination with the Thermo PRISM EuroLab twin screw extruder.

Different take-off systems, like cast sheet or blown film lines, roll away belts, chill rolls, air or liquid cooling of the product, and strand or face cut pelletising systems are required for the broad range of applications seen in extrusion.



These systems are now available to suit the enduser requirements as necessary.



Figure 3: PRISM EuroLab TSE, showing screen changer for Delta P pressure test

In addition to the dies and take off systems mentioned previously, a simple Delta P pressure filter test can be carried out directly on the end of the compounder, to give an instantaneous measure of dispersion. This uses a simple manual screen changer, as seen in Figure 3, and the data logging package with the extruder. Premixing of raw materials is often necessary, and with precisely controlled mixers, including segmented blades of different configurations and adjustable baffles this is now easily achievable.

Motor speed is variable, up to 6000rpm for small units, and the mixing bowl may be jacketed for temperature control and so mixing conditions can be optimised.



Figure 4: PRISM PharmaLab change bowl mixer

To increase repeatability, it is also possible to record process time, motor current and product temperature. For pilot scale change-bowl mixers (see Figure 4), bowl volumes are 3, 5 and 15 litres, giving effective maximum working volumes of 2, 3.5 and 10 litres respectively. Larger mixers with static bowls and discharge valves are also available from 10 to 200 litres for production scale mixing. Sensitive, high precision, versatile equipment is available for repeatable and reliable measurements in the laboratory and for production. These widely used, accepted and proven units are the PRISM line of bench top or pilot scale twin screw extruders, compounders and bench top mixers and optional extras from the Thermo Electron Corporation. They have been shown to be of value for both QC and product development in diverse sectors, from polymer processing through to food and pharmaceuticals.

PRISM twin screw extruders are being used in a wide range of continuous mixing applications in plastics, paint, food, pharmaceutical and chemical industries, world-wide. They can measure and control process parameters, and can reproducibly quantify the effects of temperature, additional ingredients, changes in process geometry etc for new and existing production lines. They can remove, the need for subjectivity in relation to estimating the quality or processability of a product, and help with estimating the potential of new products or changes in raw materials. They have an array of mixing, feeding and post extrusion take-off and product handling options. Cleaning, inspection etc are facilitated by userfriendly design - "You know it's clean, because you can see it's clean."

A copy of this application note is available on request by quoting reference no: Rheo026TP •

#### • application notes

# Improvement of the pharmaceutical coating process by rotational rheological characterisation

#### pharmaceutical, cosmetic & allied industries

#### Introduction

Film coating of solid oral dosage forms is a well established process in the pharmaceutical industry. Although, the process itself is based on the laws of thermodynamics, the result is strongly dependant on the rheological characteristics of the film coating dispersion applied.

Issues such as logo bridging, orange peel and spray drying are rooted in the viscous and elastic properties of the coating formulation. Determining and appraising the rheological characteristics is therefore of huge importance in order to prevent problems during the film coating process.

Moreover, in the field of instant release film coating, the process time and, as a result, the manufacturing costs are directly linked to the solid matter content of the dispersion. As viscosity is a specific characteristic of the film forming polymer, rheological investigation is very useful and important in order to select the most economical formulation.

A copy of this application note is available on request by quoting reference no: V-234 •

Pictured: Thermo HAAKE RotoVisco 1 -Rotational Rheometer





## application notes Rheological characterisation of C-S-H

#### mining industries

#### building & construction industries

Calcium silicate hydrate has an important position in the building-material industry. The characterisation of the variety of different phases which can be crystalline, semicrystalline or amorphous are of interest in the basic research in addition to its great practical relevance.

The investigations are performed with diluted suspensions of the commonly used raw materials especially microsilica and calcium hydroxide. Microsilica is an industrial deposit product with outstanding properties and many different applications. It is separated from the waste air of the silicon metal or silicon alloy production in the form of dust and has been used for a few decades. A large amount of microsilica is used in the concrete construction section. Special concretes with outstanding properties are revolutionising the building and construction industry, Besides this, amorphous  $SiO_2$  raw material can also be used in other sectors like the refractory industry or the production of thermal insulation. A slight change in the amount of the microslica can change the properties of the product drastically.

At any rate the great reactivity of the microsilica is primarily used at the conversion of calcium hydroxide in the pozzolanic reaction. This is due to its high content of amorphous  $SiO_2$  and large surface area.

The recording or modelling of the kinetic of this reaction is difficult because of the overlapping of different partial reactions. The progress of the reaction is generally determined using various methods, for example the concentration of the calcium hydroxide or the amount of chemically bound water in dependence of the reaction time. These methods are also suitable to characterise the reaction of pozzolana after longer periods. Immediately after mixing the reaction of microsilica with calcium hydroxide starts and shows a noticeable progress of the reaction under definite circumstances within a short time.

These changes shall be recorded in our experiments by using suitable rheological measurements. A broader motivation to investigate the rheology CSH suspensions is that these suspensions are stirred, pumped and formed in the industrial production of thermal insulation materials. Here the rheological properties have a great significance.

A full copy of the application note is available on request by quoting reference no: RF-210802-DE-01 •

## application notes Rheometry of dispersions

#### surface coatings industry

#### pharmaceutical, cosmetic & allied industries

#### Abstract

This paper shows the strengths and limitations of using rheometer as a tool to carry out product development with clear outcomes and measurements. Individual tests derived can be installed as quality control tests. The general rheological behaviour of dispersions is presented and put into relation with the application of sprayability or levelling. Measurement procedures for sagging, levelling, sedimentation, brushability and the yield point determination are discussed and demonstrated on selected samples.

#### Introduction

Rheometry characterises the sum of all components of a fluid and its interactions in respect of its mechanical properties. If one part of a formulation changes then the rheology of the product changes as well. Therefore rheometry is often used as the preferred tool to perform the final inspection of materials before leaving production. This advantage is opposed by the problem that rheometry cannot tell us which part is causing a different behaviour from the expected one unless there is very good practical experience available. Rheology does not replace chemistry or personal experience but it is an objective tool to judge overall product quality.

## Rheology of dispersions (paint like fluids)

Many paints behave like dispersions or suspensions as seen in other products and substances. Their mechanical behaviour expressed as viscosity and elasticity depends on:

- temperature
- deformation, deformation rate
- time

To fully characterise a fluid the influence of these parameters has to be know in order to predict the response under different applications.

A full copy of the application note is available on request by quoting reference no: HA-016 •



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

**Telephone:** 03 5367 7477 **Facsimile:** 03 5367 6477 **Email:** info@rheologysolutions.com **Web:** www.rheologysolutions.com

### • rheotalk jul-aug 2009

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- Concrete Rheometer Schleibinger Viskomat NT
- Extensional Rheometer HAAKE CaBER 1
- Closed Circulator HAAKE DynaMax
- Recirculating Chiller NESLAB ThermoFlex
- Twin Screw Extruder PRISM TSE
- Modular Twin Screw Extruder PRISM EuroLab
- Sheet line take-off system PRISM Sheet Take-Off
- High Speed Pellet Scan System OCS PS200C
- PharmaLab PRISM PharmaLab
- Change Bowl Mixer -PRISM Change Bowl Mixer
- Process Viscometer Marimex ViscoScope
- Extrusion & Material Characterisation for Drug Development & Production
- Comprehensive Materials Characterisation for Polymer

#### Services

Calibration & Service. Please provide details on model of product and serial number: .....

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