rheotalk
jul-oct 10

• News & Views

Online rheometer (OLR) commercialisation project underway at Rheology Solutions

all industries

Introduction

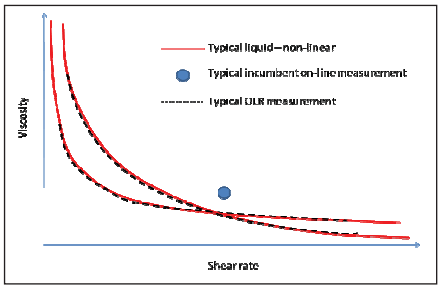
The project is to commercialise novel CSIRO technologies for an online process control instrument that deliver real-time data to achieve and maintain better quality control for production processes. The instrument is the 'Online Rheometer' (OLR), which measures and delivers the flow properties of a liquid in a pipe in real time. The market need for an instrument like this one is acute, and there are currently no direct competitors for the instrument.

Across key sectors e.g. food technology, mining, and petrochemicals, product quality is verified by measuring flow properties (rheology) of liquids. Currently, these measurements are either through laboratory analysis which is highly accurate, or real-time, through in- or online viscometers, where low quality data compromises reliable interpretation. The OLR will deliver a constant flow of high quality data in real time, to maximise the benefits achieved with real-time process control. We will expand our current capabilities to build a high technology manufacturing facility in rural Victoria and export the product from Australia to the world through established national and international distribution channels.

OLR applications field

Except in a few niche applications (notably the polymer industry), incumbent online measurement technologies offer a single data-point from a usually non-linear curve to imply the flow properties of the fluid being measured. These measurements are generally made with a vibrating reed or tuning fork type of probe, and the location of the single datapoint are dictated by the frequency of vibration of the probe. Often the measurement is only an approximation of the actual viscosity and is not equal to the viscosity of the liquid in question.

Usually, the operating frequency of these instruments has been chosen to suit what the instrument manufacturers see as a robust general setting for all liquids. Because most liquids of industrial interest have non-identical, non-linear flow properties, the assumption that a single data point can provide a good quality control is flawed. Until now though, that has been the only option for on-line quality control and industry has had to 'make-do'. The graphical representation following illustrates the point.



The OLR instrument with data collection and processing technologies consists of an axially symmetric short flanged section of pipe, with vertically oscillating plates inside. One plate moves and the other measures the response of the liquid (a force exerted on the lower plate). The photograph and schematic below show the prototype OLR, as developed by the CSIRO.

The OLR offers the user an opportunity to continuously measure, in real-time in the pipe, a substantial portion of the curve, representing the fluids key characteristics – as shown in the graphical representation earlier. We can provide superior quality and quantity of information in the same time (near-instantaneous, real-time) as the current on- or in-line technologies for our target market segments, at a similar cost.

The OLR development team

Development OLR from prototype to production model will have significant challenges from the technical, applications development viewpoint, as well as from the viewpoint of transferring what is essentially an R&D project into a commercial reality. With this in mind, Rheology Solutions have begun to build a team to address the needs of this project. The key staff members are in place, with more to follow as the project expands into preproduction and beyond.

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• News & Views

Missed an issue of RheoTalk?

All issues of RheoTalk are available as PDF downloads on our website **www.rheologysolutions.com/newsletters**

all industries

The Rheology Solutions Newsletter was created in 1999 specifically for Australian customers and interested parties. The concept of this newsletter is to provide an update and overview of news and information covering Australian seminars, industry specific application reports, product launches, new appointments and general product information.

Additionally, we have commenced producing a PDF version of the Biennial RheoTalk covering 2007 - 2008. The concept of the cumulative issue is to provide industries with a complete index that categorises the contents into specific industry related information.

The industry classifications that have been used are:

- Mineral Slurries
- Polymer Industries
- Food Industries
- Surface Coatings Industries
- Chemical & General Manufacturing Industries
- Pharmaceutical, Cosmetics & Allied Industries
- Construction & Building Industries.

• In This Issue

Specific Industry Key

Product News & Industry Applications

Save water & save money

all industries

Many laboratories and small scale pilot-plants use tap water as the cooling liquid for the process or application.

Just think, if your equipment was using tap water based on the average tap water flow rate of 16L/min, for 24 hours, seven days per week, 52 weeks per year, you are potentially sending 8,000,000 L of water per year – per piece of equipment down the drain.

Recirculating chillers and bath recirculators conserve water and save you money by recirculating the water used to cool your equipment. Using a recirculating chiller or bath circulator will conserve our greatest and most precious resource – water.

The ThermoFlex range covers +5ºC to +40ºC and is available from 900W through to 10,000W cooling capacities.

Thermo Scientific NESLAB ThermoFlex - recirculating chillers – models TF 35 (3,500W) & TF50 (5,000W) models shown

Product News

New & exclusive – refrigerated water bath

all industries

Cool Tech Plus refrigerated bath circulator at **$3,990** excluding GST & local delivery.

Max: flow rate	17 L/min
Max: pressure	300 mbar
Temp. range	-28ºC to 100ºC
Dimensions	711 x 273 x 483 (HxWxL) mm
Cooling capacity	320W @ 20ºC
Bath capacity	6 to 10L
Work area	204 x 173 x 129 (DxWxL) mm
Net weight	36kg
Order no	152528101

The Cool Tech Plus is supplied complete with SC100 circulator head (as shown in the picture) and 10L stainless steel bath. Delivery within 48 hours from receipt of purchase order.

Looking for something else?

Rheology Solutions are able to offer you the most flexible, cost-effective temperature control solutions for a multitude of applications across:

- Pharmaceutical
- Food & Beverage
- Research & Development
- Analytical Instruments
- Laboratory Equipment
- Petrochemical
- Chemical
- QA/QC
- BioTech

The range covers:

- Refrigerated Bath Circulators
- Heating Bath Circulators
- Ultra Low Refrigerated Circulators
- Recirculating Chillers
- Water Recirculators
- Open Bath Circulators
- Software options
- Extensive range of accessories.

Cool Tech Plus - the cost effective refrigerated bath circulator

Call Rheology Solutions or email sales@rheologysolutions.com to arrange for a formal quotation

Solutions for pharmaceutical, cosmetic & allied industries

pharmaceutical, cosmetic & allied industries

Rheological parameters are reliable pointers for the pharmaceutical, cosmetic and allied industries product performance and are critical factors for processability and product quality.

Measuring these parameters will improve process and product performance within the pharmaceutical, cosmetic and allied industries.

There are a range of instruments and measurements that can be used to quantify rheology related challenges some of which may be described by the following terminology:

mouthfeel is the experience from all the senses of the skin in the mouth during and after swallowing. The viscoelastic properties of a product that is swallowed such as a cough medicine, impact the length of time that flavour and sensation of the product remains in the mouth and assists in coating the lining of the mouth and throat. Measuring the viscoelastic properties of products requires a controlled stress rheometer. The HAAKE RheoStress 1 is a controlled stress rheometer and will measure the viscoelastic properties of liquids and assist in developing and maintaining the right product mouthfeel.

(Left) Thermo Scientific HAAKE RheoStress 1 with cone heater

(Below) Thermo Scientific PolyLab OS with RheoMix pilot scale torque rheometer with instrumented mixer

processability is typically related to the amount of energy required to carry out the relevant unit operation. Determining the processability of a cream, gel, liquid or powder can be achieved using a HAAKE PolyLab and RheoMix. It provides information on torque, temperature and rpm inputs which relate directly to the processability of the materials being mixed or transported.

consistency is the combination of all the senses from stimulation of the mechanical and tactile receptors in the region of the mouth and skin and varies with the texture of the product. For liquid products that are swallowed, the impression of consistency is dictated in part by viscoelastic properties; also this is the case for semi-solid products that are applied to the skin. A HAAKE MARS rheometer can measure viscoelasticity for characterisation of the consistency of a product.

(Above) *Thermo Scientific HAAKE MARS controlled stress rheometer*

shelf life is the maximum time interval that a material may be kept in a usable condition under correct storage conditions. Elements that impact shelf life include settling or phase separation and are governed by rheological factors.

Measurement of yield point will indicate if settling has occurred and measuring shear viscosity at different shear rates will provide an indication of what conditions under which the phase separation may occur. The HAAKE RotoVisco is a controlled rate viscometer and is suitable for measuring the key rheological pointers for shelf life on both liquid and solid samples.

(Left) Thermo Scientific HAAKE RotoVisco controlled rate viscometer

Do you need to characterise your product? The terms usually referred to when you assess a product include Consistency, Mouthfeel Processability, Shelf Life, Tack & Yield Stress.

tack is the property of cohesion between particles (stickiness), and the separation force needed for the transfer of the coating from the substrate as in the case of a coated tablet. Measuring the extensional viscosity will provide data to identify stickiness during coating applications. Extensional properties can be measured with the HAAKE CaBER 1 extensional rheometer and provide valuable information on the suitability of the cream or other liquid to be filled into bottles, sachets or tubes. Understanding the extensional properties of a product is also important to eliminate 'strings' during the filling process to maximise filling line production speed.

(Right) Thermo Scientific HAAKE CaBER 1 extensional rheometer

yield stress is the minimum force required to initiate movement in a fluid. Measuring the yield point in suspension such as in a liquid, cream or gel using the HAAKE Viscotester 550 with a vane sensor will provide valuable information about the force required to initiate movement of the suspension etc. for initiating the filling process and so on.

(Left) HAAKE Viscotester 550 controlled rate viscometer with vane sensor

temperature control Process and testing requires constant and accurate temperature control to ensure consistent test conditions are maintained throughout. Temperature deviations and fluctuations will influence the process and/or the test results. Choosing the correct temperature control product is as critical the test itself. A range of recirculating

heating and cooling bath circulators, process chillers, immersion coolers, recirculating chillers, and Ultra Low Temperature (ULT) bath circulators are available to meet most chemical industry applications across QA, QC, R&D and production from HAAKE and NESLAB.

Thermo Scientific refrigerated & heated bath circulators – configure the most flexible, cost-effective temperature control solution

Micro-conical twin-screw compounders

polymer industries

pharmaceutical industries

surface coatings industries

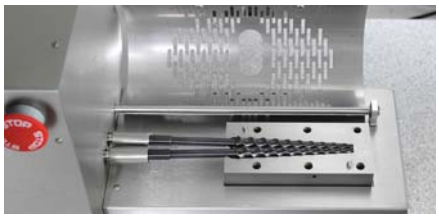
The Thermo Scientific HAAKE MiniCTW is the latest model in the suite of micro-conical twin-screw compounders.

These machines feature several models that serve a variety of markets, including polymer, bioscience, nanotechnology and pharmaceutical.

All models use as little as 5 grams or 7 millilitres (ml) of material for compounding, which is especially beneficial for industries that compound expensive or small-scale materials such as nano-composites, bio-polymers or pharmaceuticals. By using the optional force feeder, customers can continuously extrude very small material volumes. The instrument is based on proven, conical twin-screw technology with co-rotating and counter-rotating screws, and works as a standalone unit with data export. The residence time is well-defined due to the channel and a bypass valve.

The HAAKE MiniCTW is ideal for the precisely controlled reactive extrusion of high viscous melts. By running the instrument in circulation mode, the required reaction time for the reactive mixture can be easily controlled. At the end of the test, the bypass valve can be opened and the sample extruded as a strand. By measuring the torque of the drive motor, the reaction process can be monitored effectively.

Filling the injection moulder test sample die from the HAAKE MiniCTW



Features and benefits of the new suite of micro-conical twin-screw compounders include:

- Requires only 7 ml of material for compounding
- Removable top barrel for easy and quick cleaning
- New software for user-friendly process monitoring
- Various models serving a breadth of applications (from polymers to pharmaceuticals).

Technical Specifications

Drive	
Motor power	400 W
Speed range	1...360 min^{-1}
Max. torque	5 Nm per screw
Control mode	Constant Speed Constant Torque
Power supply	230 V ± 10 %, 50/60 Hz

HAAKE Force Feeder	
Max. speed	30 min^{-1}
Material	Stainless steel 1.4122 Cr coated
Power supply	230 V ± 10 %, 50/60 Hz

Extruder	
Design	Conical co-/counter-rotating
Temperature	300°C
Heating time	< 10 Minutes (80°C - 240°C)
Barrel & screws	High performance plastic mold steel (M340)
Cooling	Forced Convection
Pressure	up to 200 bar
Volume	7 ml
Bypass	Manual valve
Feeding	Manual feeding Force feeder (optional)

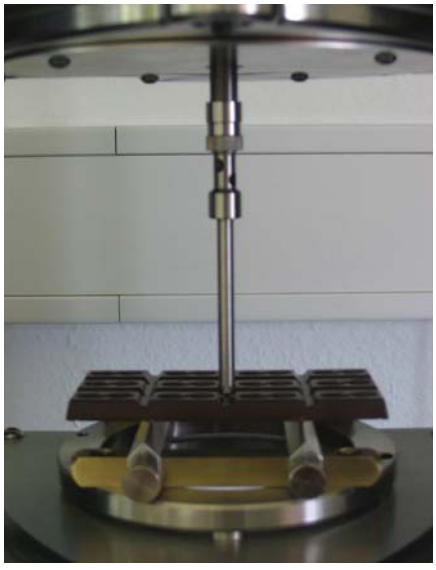
Viscometers & rheometers for chocolate manufacture

food industries

Rheological measurements are essential in optimising the production and properties of confectionery.

The HAAKE Viscotester 550 is used to determine the flow behaviour and yield point of chocolate melts according to the guidelines of the expert commission of the Office International du Cacao, du Chocolat et de la Confiserie (OICCC), therefore influencing product characteristics such as melt and setting behaviour.

The HAAKE Viscotester 550 assists in the measurement of data and different parameters that influence the quality of the chocolate. When used in conjunction with the use of the HAAKE RheoWin software, you can graphically visualise the measured data.



HAAKE MARS with bending and breaking tool for measuring the breaking behaviour of chocolate

An extensive range of accessories for the range of viscometers and rheometers are available to cover the unique aspects of a wide variety of applications. For example, a bending instrument is available for the rheometer line which can be used to place bars of chocolate and perform bending, breakage and penetration tests.

With the RheoScope module for the HAAKE MARS rheometer platform, structural examinations

HAAKE MARS with RheoScope module - modular rheometer with optical microscopy

can be performed using optical microscopy at the same time as the rheological measurements.

In this way, the melting behaviour of fat samples and crystal formation can be studied. A universal holder makes it possible to take measurements in original containers, such as jars of chocolate sauce. This eliminates the time-consuming process of pouring out samples and cleaning cups, and also preserves the material structures.

The role of rheology in pharmaceutical & cosmetic industries

pharmaceutical, cosmetic & allied industries

The paper "Rheology for Cosmetics and Pharmaceutical Manufacture" includes a balance of theory and applied examples across the life cycle of a product from raw materials through to consumer use.

These included:

Mixing, pouring & filling

The section covering mixing and pumping introduced the concepts of viscosity and elasticity. Ease of pouring/filling introduced concepts of yield stress and extensional properties (extensional properties influence the 'stringiness' of a material).

Commonly encountered rheology terms were defined and explained in the context of typical shear rates and test data for some cosmetic and pharmaceutical products such as: soap, toothpaste and gel toothpaste, gums and oils, and shampoos.

Storage stability

Storage stability is often a function of storage time which affects the amount of settling and/or phase separation and is determined by viscosity, density and yield stress. Temperature also has an impact on the storage stability of a product and samples can be tested to predict the stability and effect of a temperature on the sample. An example of a freeze-thaw test to determine the behaviour of a stable and unstable sample were also presented and discussed.

Quality control

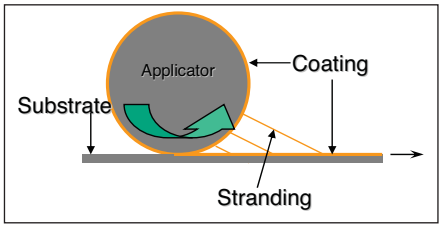
QC criteria include tests such as flow curve, yield stress, amplitude sweep and frequency sweep. Results for each test were presented using real data from hair gel.

Dispensing/application

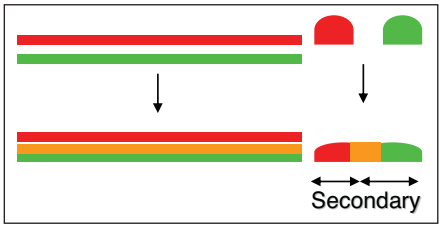
Dispensing with a roller may involve problems with stranding, misting and spatter which are determined by extensional properties and elastic properties of the sample. When a product is squeezed from a tube, problems can occur with edge and line definition and spreading and are governed by thixotropy, elasticity, yield stress and extensional properties.

Squeezing the product from a tube is determined by viscosity and viscoelastic properties, and surface finish (ribbing etc) is measured by extensional properties and viscoelasticity.

Application was broken into two parts, spread by rubbing and application by atomised spray or drops. Each of these two parts detailed the dominant rheological properties and the relevant measurements required to determine a products dispensing and application profile.



Extensional properties dictate stranding, misting & spatter



Thixotropy, elasticity, yield stress & extensional properties dictate edge & line definition & spreading

Texture

The measurement criteria to determine texture perception for fluids differs from that of semi-solids. For fluids, skin feel includes tack, greasiness, stiffness, stickiness etc and are governed by viscosity, elasticity, extensional properties, and yield stress. With semi-solids, skin feel relates to cohesiveness, adhesiveness and hardness. These relative properties are governed by viscosity, elasticity, yield stress; solid mechanical properties under extensional or compressive loading rheological parameters are commonly used to scientifically measure the texture of solid materials.

A copy of the presentation "Rheology for Cosmetics and Pharmaceutical Manufacture" is available as a PowerPoint interactive file on request.

Additionally, a solutions kit titled "Rheology Solutions for Pharmaceutical, Cosmetic & Allied Industries" is available free on request.•

Online rheometer (OLR) commercialisation project underway at Rheology Solutions *from page 1*

The new team so far are Dr Tim Kealy and Dr Pradipto Bhattachargee. Dr. Kealy is the project director for the OLR commercialisation project and has a strong background in rheology and rheological measurement techniques, as well as extensive recent experience in project management and product commercialisation.

Tim has worked in academia locally at Curtin and Monash Universities, and outside academia at Rheology Solutions as our technical manager for six years to 2007, followed by three years in the product development team at Plantic Technologies in Victoria.

Dr. Bhattachargee (PK) has been employed as senior scientist for the OLR commercialisation project, and has a strong background in rheology theory and measurement, novel measurement techniques and applied rheology. PK has worked with highly regarded rheologists locally and internationally within research positions at Monash University in Melbourne, and in Massachusetts, USA.

The commitment of experienced and well regarded rheologists such as Dr. Kealy and Dr. Bhattachargee, is further evidence of the perceived potential for success of the OLR in the scientific, manufacturing and process communities. •

Advances in small scale processing equipment for polymers and additives

polymer industries

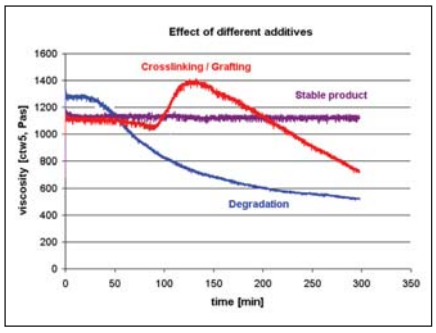
Bernd Jakob
Thermo Fisher Scientific, Karlsruhe, (Germany)
Presented at the ECNP in Madrid

Introduction

The use of a conical twin-screw extruder with backflow channel combines the aspects of mixing and extrusion in a batch process. With a small force feeder and a conveyor belt the micro-compounder operates continuously producing a strand or a small sheet. With a total secondary flow filling volume of 7 ml and a built in slit capillary die the applications focus is on compounding and reactions of small amounts of polymers in the molten stage. Two great areas of application: mixing and rheological recording of the melt characteristics are combined in the micro compounder. For further tests the polymer melt can be transferred into a micro injection moulding machine to shape different kind of test specimens for further analyses.

Compounding

To analyse small batches - typically 5 to 50 grams - different test setups are possible. Either continuous or batch processes with defined mixing times can be selected. Co- or counter-rotating screws are used to melt and mix the polymer pellets or powders. The influence of additives is show in graph 1.



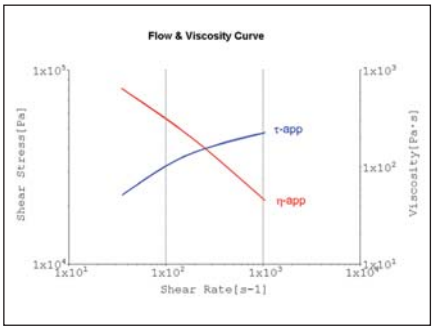
Graph 1: The Effect of different additives

The effect of different additives is obvious. One compound is stable for 5 hours and no change in the viscosity is observed. Where as the other compound degrades after about 30 minutes visible by a decreasing the viscosity. A third compound shows a viscosity increase after about 120 minutes due to a reaction of the product followed by degradation, the viscosity decreases.

Viscosity

The backflow channel of the micro-compounder is designed as a slit capillary. Two pressure transducers in the backflow channel measure the pressure drop. From this pressure drop a shear stress is calculated. To determine the shear rate, the mass flow in the flow is correlated with the screw speed of the Thermo Scientific HAAKE MiniLab. To record a flow and viscosity curve, different screw speeds are set and the pressure drop (shear stress) at those different screw speeds is measured.

The correlation between the screw speed and the mass flow is for each polymer slightly different. So the calculated shear rate and therefore also the calculated viscosity can only be a relative value which is accurate enough to compare different batches. A typical flow and viscosity curve of a polymer is shown in graph 2.



Graph 2: Flow and viscosity curve

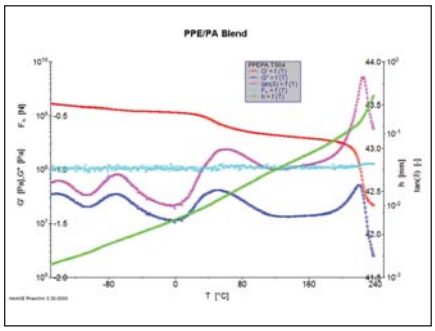
Test specimens

With the compound produced in the MiniLab various test specimens can be produced very efficiently with the Thermo Scientific HAAKE MiniJet, a micro injection moulding machine. The instrument is build as a piston injection machine with a removable heated cylinder with injection piston to collect the melt from the MiniLab. Also pellets or powders can be molten direct in the heated cylinder. The amount of required sample material is minimised to less than 5 g. Depending on the shape of the test specimens up to 5 pieces can be produced with one filling. Almost the whole amount of polymer melt is completely trans-formed into test specimens. This is a major advantage as the waste material of conventional screw driven injection moulding machines can be avoided by this principle. With the exchangeable moulds disks, plates, DMA and tensile bars (Picture 1) can be prepared for hardness tests, breaking strength, Dynamical Mechanical Analyses (DMA) and oscillatory melt rheology.

Picture 1: Mould and test specimens for tensile test

Dynamical Mechanical Analyses (DMA) and oscillatory melt rheology

For testing of the solid polymer according to DIN/ISO 6721-1 a DMA bar is mounted with solid clamps in the Thermo Scientific HAAKE MARS monitoring the effect of additives over a wide temperature range. The effect of the additives is seen in the shift of the glass transition temperature. In graph 3 the result of a DMA test is shown. For further tests only the sample holders are changed to parallel plates. Between those plates a round disk of polymer is molten for oscillatory measurements to determine the visco-elastic properties. With another measuring unit a microscopic visualisation of melting or crystallisation simultaneous to the oscillatory test is possible.



Graph 3: DMA test of a PPE/PA Blend

Conclusion

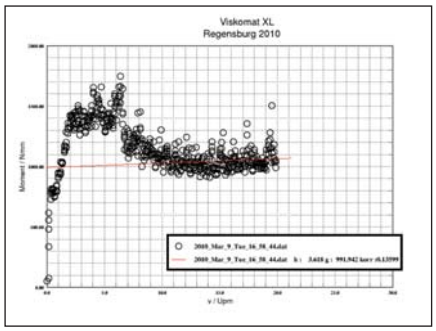
The micro-compounder HAAKE MiniLab is a useful tool to process small amounts of polymers and additives. Especially in the stage of development of new products and the screening process for promising candidates of new compounds reproducible results are obtained in a shorter time. For further tests the compound can be shaped to a variety of different test specimens with the micro injection moulding machine HAAKE MiniJet.

A copy of this technical paper is available on request by quoting HA049.•

Measuring the rheological properties of fresh concrete

construction & building industries

The flow of a floor screed with a maximum aggregate size of 8mm was measured.



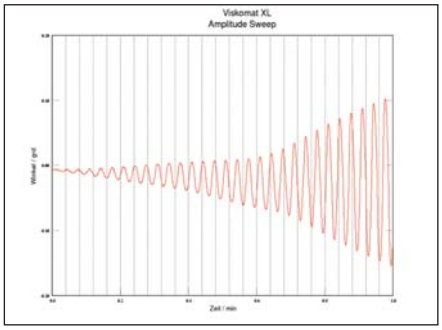
Oscillatory measurements have been widely used in the last 20 years for polymers and similar materials. Rheometers, offering this measurement mode, usually have a sample volume less then 400ml and are from the Searle type. We have now developed a Couette rheometer, that can handle a specimen volume of 3 litres and a specimen weight of more the 6 kg.

The maximum frequency is 5 Hz, depending on the maximum amplitude. Frequency and amplitude sweeps are possible.

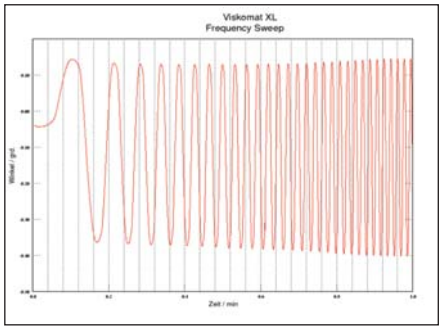
Based on 20 years experience with rheometers for mortar and fresh concrete, Schleibinger has developed a new instrument called Viskomat XL. There is a technology gap between the Viskomat NT filled with mortar and paste with a specimen volume of 360 ml and the concrete rheometer BT2 with a sample volume of 20 litres.

The operation principle of the Viskomat XL is near the same as for the Viskomat NT. A mixer shaped probe measures the torque, and the specimen vessel rotates. Also standard probes, like cylinder/cylinder or cone/plate are available. An additional, optional scraper is cleans the wall of the vessel. The speed may be 0.001 to 180 rpm in both directions, clockwise or counter clockwise.

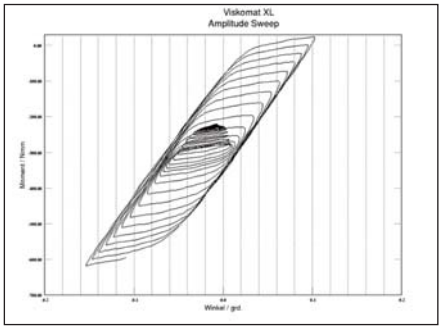
You may define the speed in several steps, in a linear increase or decrease of speed. Also a shear stress controlled mode or an oscillating mode is available. As far as we know this is the first concrete rheometer offering this feature.



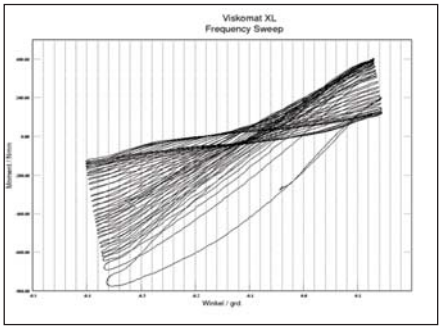
Amplitude-sweep, the vessel is oscillating, angle (0..±0.2°) over time (0..1min) material floor-screed, max. aggregate size 8mm



Frequency-sweep, the vessel is oscillating, angle (0..±0.25°) over time (0..1min) material floor-screed, max. aggregate size 8mm



The resulting Lissajous figure of the amplitude sweep, torque (0..±700 Nmm) over the angle (0..0.2°), material floor-screed



The resulting Lissajous figure of the frequency sweep, torque (0..±700 Nmm) over the angle (0..0.25°), material floor-screed

The Viskomat XL has a torque range from 0..300 Ncm with a resolution of 0.05 Ncm and accuracy better then 0.2 Ncm. Optional we can install a sensor with a torque range up to 1000 Ncm and a accuracy of 0.8 Ncm.

A rheometer for coarse suspensions like fresh concrete with a maximum grain size up to 16mm was developed working also at very low speeds < 0.001 rpm.

- It may also run with a shear stress controlled mode.
- The Viskomat XL is the first oscillating fresh concrete rheometer on the market.

A reprint of this paper is available on request by quoting Schl030.•

Technical Papers

Equipment and measuring systems for testing the quality of concrete, mortar and additives

construction & building industries

Introduction

The quality of cement-based building materials such as mortar and concrete can be divided into three fields: durability, dimensional stability and workability.

While the testing methods for testing the strength by compression testing machines has been standardised both nationally and all over the world for decades, tests and testing methods for the durability, dimensional stability and workability are not standardised at all, or the procedures differs from nation to nation. A lot of testing methods are very controversially discussed among experts and many arguments are influenced by an industrial, political and financial background. In this article testing equipment and methods are presented which are partially described in rules and standards. However, none of these testing methods has been established in a valid EN or ISO standard up to now.

A full copy of this paper is available by quoting Schl031.

Industry Applications

Solutions for pipeline transport

mining industries

The process of moving slurries or pastes from one location to another by pumping the material through a pipe.

Measuring on-line viscosity using a Marimex ViscoScope system will provide constant data monitoring in the pipe or tank so adjustments can be made during the process to maintain the best conditions for pumping.

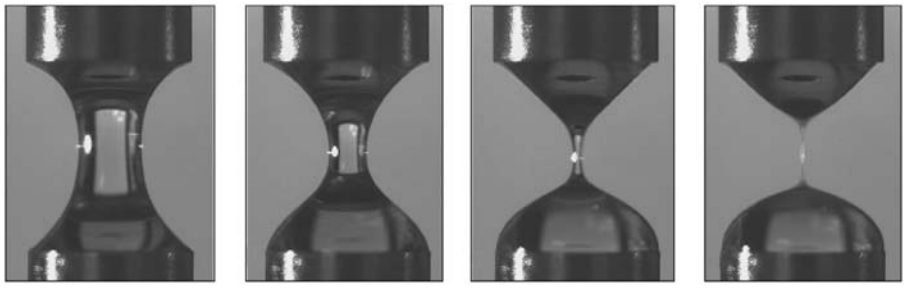
Marimex ViscoScope – in-line viscometer

Industry Applications

Solutions for misting & spatter

food industries

Measuring your products extensional properties with the HAAKE CaBER 1 extensional rheometer can eliminate excessive paint removal from rollers and improve your process and products performance.



Knowledge of the extensional behaviour of fluids is important for both industrial and research purposes. Complex flows that contain strong extensional components occur in many industrial processes and applications. Some examples are extrusion flows, coating flows, contraction flows, and fibre spinning flows. Most materials exhibit very different responses in an extensional flow compared to a shearing flow. The thinning and break-up of a fluid filament that is analysed with the HAAKE CaBER 1 instrument provides valuable information about the material's physical properties that rotational rheometers simply cannot provide!

New Product Brochure

Chemical & physical property analysers

polymer industries

Optical Control Systems offer an extensive range of products specifically for R&D, QC, Lab and on-line applications to measure and test the chemical and physical properties of polymers.

This brochure provides detailed information on the following products.

On-Line FT-infrared spectroscopy system APLAIRS®

APLAIRS® (Analysis of Plastics by InfraRed Spectroscopy) is a spectroscopic technology, applied in the modern plastic industry, to measure real time additives, co-monomer composition as well as some chemical properties during the production of the base resin. For state of the art process as well as quality control, the APLAIRS system, is an absolute requirement in modern plastic production facilities. With a better precision and a faster analysis time, this single technology replaces most of the conventional off-line quality control (QC) methods. For process control, in addition to melt index and in homogeneity, the APLAIRS system generates real time very useful complementary analytical data. For example, for products with a similar melt index and a different additive package, APLAIRS will be able to accurately monitor type changes. In addition, the technology offers tools for monitoring real time co-monomer incorporation. Physical property such as density in polyethylene can be determined real time, with a better precision than the offline column gradient or Archimedes methods. The technology provides early warning in erroneous master batch preparation, which in many cases avoids huge customer claims. As this technology replaces many conventional QC methods, savings in labour cost are considerable. The APLAIRS system is fully automated and no people are needed to run the system.

APLAIRS on-line FT-infrared spectroscopy system

Measuring principle

A continuous flow of cast or blown film runs through a special infrared sampling section of the APLAIRS® system, which is equipped with an FTIR spectrometer and controlled by dedicated software. APLAIRS® focuses on film measurements, although infrared spectroscopy can also be applied to melt samples. Measuring in the film has more advantages than measuring in the melt.

Films are closer to the end product and for that reason a direct link is made with standard QC Analysis. Physical properties not only depend on chemical components but also on the morphology of the resin.

The morphology and chemical information concealed in the spectra can be abstracted by APLAIRS® and linked with physical test data.

Thickness of films can be accurately determined. In addition, it is possible to determine the composition and thickness of different layers in co-laminates.

Other analytical techniques such as Gloss and Haze measurements can be far easier linked to a film line, making the assembled configuration inexpensive.

On-line thickness measurement system – TM9

OCS on- line thickness measurement system - TM9

The on-line Thickness Measuring System TM-9 was designed for the continuous thickness measurement of running strips of film.

The measuring system uses an eddy current sensor. The film runs between two free running measuring wheels, one of which is stationary.

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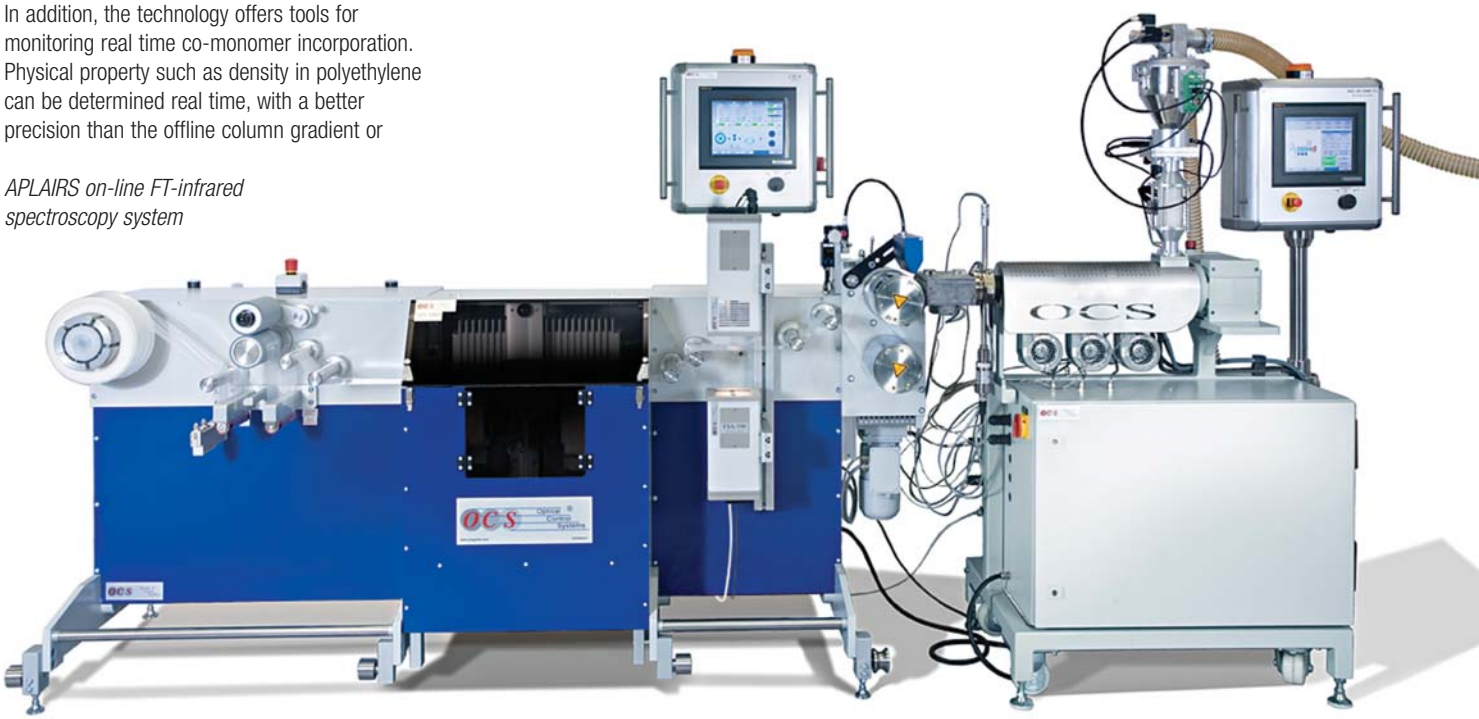

Chemical & physical property analysers *from page 12*

The position of the other and also the force it exerts on the film is adjustable using a spring and an electromagnet. This wheel is connected to the sensor which allows a direct measurement of the distance between the wheels and therefore the film thickness.

The thickness is displayed in µgm. A certain amount of measurements occurs each second and the result is averaged over a certain length of film. Both the amount and the length can be adjusted.

The electronics for evaluation and the regulation of the wheel force and the LED display are located in a separate housing.

On-line Thickness Measuring System TM-9 used for continuous measurement of the thickness of running strips of film for laboratory and production line use.

The TM-9 was constructed to accompany the production of film strips. It can be optimally used with the OCS Measuring Extruder ME 20 / 26 - Chill Roll and Winder Unit CR-9 which additionally has integrated infrared spectroscopy).

On-line haze measurement system – Gamma 12

The functional principle of Gamma12 complies with the standard ASTM 1003. The measurement sample is passed across the opening in an integrating sphere so that parallel light can pass through the sample into the sphere. The intensity of the dispersed light can be measured using a high-precision sensor by means of the dispersion of the transmitted light beam. Light beams with dispersion angles exceeding a standardised value cannot leave the sphere through the exit and are reflected by the sphere surface. The intensity of the scattered light is measured after multiple reflections within the integrating sphere.

The intensity of the total light transmitted can be determined by measuring with the sphere exit closed. The haze value is calculated from the ratio of the scattered light and the total transmitted light.

In addition to the haze, the transmittance of the sample is measured. This is derived from the ratio of the total transmitted light and the intensity of the illumination. The measurements are shown on-line on a display as percentages and therefore provide an objective indication of quality instead of a visual subjective assessment. The measurement results can be documented and analysed with high-performance software tools owing to the link via the interface RS232.

On-line Gloss Measurement system – GM5

The Gloss Measurement System – GM5 serves the determination of the gloss characteristics of the manufactured film. The measurements are effected at the current process by analysis of the reflected light. The ethernet interface permits the evaluation and documentation of the measuring data.

OCS on-line haze measurement system

OCS gloss measurement system GM5

The data exchange is realised by Modbus/TCP protocol or optional OPC server. The measurement process is automatically controlled (back round measuring and calibration).
The measured sensor signals are digitised after analogue processing. By using an RS232-interface, the data can be passed onto a PC or onto the film quality testing system, FSA100. If the data is transmitted to the FSA100 system, it is possible to display details concerning film quality as well as the haze.

New Product Brochure

Rheological & physical property analysers

polymer industries

Optical Control Systems offer an extensive range of products specifically for R&D, QC, Lab and on-line applications to measure and test the rheological and physical properties of polymers.

This brochure provides detailed information on the following products.

Process melt rheometer – OP5

A system for plant optimisation and improved product quality. The function of the OP5 is to make certified measurements of the melt index and/or Polydispersity of small solid polymer samples. The primary duty of these measurements is overall control of many types of polymerisation processes. This ensures that the product can be made to specific formulations.

OCS process melt rheometer - OP5

OCS process melt rheometer - OP5

The secondary duty is quality control in final product selling specification and in batch control. The OP5 is logically situated in the plant analysis laboratory, which ensures best reliability and maintainability for these calibrated, precision measurements. Representative samples are therefore transported from various locations of the polymer manufacturing plant at the call of each analyser. Process Control and QC are full time activities, which require, as a minimum, one sampling point for each analyser.

Representative samples are extracted from each reactor stage and from the finished product. In simple plant configurations, such as LDPE, PS, PET and Nylon this usually means one OP5 but in complex plants such as PP, HDPE and LLDPE two or more OP5 analysers would be required. The complex plants have powder samplers, which have degassing and catalyst deactivation stages close the analyser. At the plant end, these samplers can extract at the main discharge valve(s) of the reactor or at the primary degassing vessel, etc. according to the plant configuration. In the case of the pellet samples, these would normally be supplied from central (OCS) source, and in such cases the supply to the rheometers carries a priority to minimise the sampling delay time.

Melt flow measurements are performed after the solid sample is melted and conditioned to the appropriate test temperature. In the OP5 the melting process minimises any changes to the structure of the polymer but making a very rapid

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Rheological& physical property analysers *from page 14*

transition from solid to liquid, which substantially obviates shear damage, cross-linking, thermal degradation and other degradation processes. This sample preparation removes the last traces of any trapped air or gas and overall makes an important contribution to making the sample at the point of measurement fully representative of the process. In this unique way the OP5 makes control through rheology as reality.

Full Notch Creep Test – FNCT

The Full Notch Creep Test FNCT is a widely used method to classify polyethylene materials in regards to their slow crack growth behaviour under accelerated conditions: ESCR (Environmental Stress Cracking resistance). In this test, a typically square sample is submerged into a surface active agent. This agent accelerates crack growth. Depending on the chosen test conditions, the agent is held at a certain temperature (up to 95ºC) throughout the test. A steady tensile load is applied to the sample which has a defined circumferential notch to the initiate the crack, followed by crazing, crack growth and finally brittle failure. The time to failure is measured and used for the classification of the material.

Pellet Transport System – PTS

The Pellet Transport System PTS is a continuous and automatic transportation of pellets between the production lines and measuring systems. Samples of pellets from the production line are affected by means of pneumatic sample takers. Samples are sent through aluminium or stainless steel pipes (shot peened option). The PTS consists of hopper loaders (cyclone) with shutter valves for extruder with low and high level sensors for sampling.

Furthermore a stand by tank for purge and calibration material and a 3-way switch for a starvation system is available. The PTS is controlled with a PLC which is driven with a TFT touch panel for visualisation and control of the sampling system. The system is equipped with a digital I/O interface to the DCS for transferring status and alarms.

All pipes and bends (elbows) are specified to avoid dust, angel hairs and streamers.Totally gap-free flange connections (recommended: slip-on collars and loose flanges with projection and recess).

Overhead photo of OCS pellet transport system

A de-dusting device for removing dust and streamers etc. is an option. Sample taker, 3 way switch and special hopper for extruder consist of:

- Hopper loader (cyclone) with shutter valve for the analyser with low / high level sensors for sampling.

OCS pellet transport system

Industry Applications

Solutions for coatings & cure

Controlled stress rheometers such as the HAAKE RheoStress provide the data to determine flow, viscosity and yield point as well as elasticity dominated properties such as die swell, ribbing and gel strength

food industries

Coating, dip coating and roller coating are critical where predictable flow properties at an efficient energy cost to minimise waste and optimise.

Build, cover, spread, ribbing, tails and feet are rheology-driven coatings related phenomena which can be quantatively measured.

Quantifying these properties is achieved using controlled rate viscometers or controlled stress rheometers.

Cure within food processing relates mainly to the drying of a coating either applied onto or over a product or the cooling of an extrudate. The terms referred to within the cure process include spread and tails, that is; if the applied coating has spread, covers evenly with no secondary flow or 'stringiness', or drips after dip coating. The governing properties determining cure include elasticity, viscosity and yield point.

Quantifying these parameters is achieved using either a controlled rate viscometer or controlled stress rheometer for both spread and tails and in the case of processes like dip coating or filling an extensional rheometer is used.

Extensional rheometers such as the HAAKE CaBER 1 provide the information necessary to determine the existence and extent of tails within the dip coating process

Industry Applications

Solutions for blending & mixing

chemical industries

Combining materials to give the desired physical or chemical properties.

Blending efficiency relies upon the viscosity of the materials at high shear. Matching viscosities often allows better blending. Viscoelastic properties also play a significant role in blending efficiency. The Thermo Scientific range of Twin-Screw Extruders is an excellent way to assess the potential for blending different materials for extrusion based processes.

Thermo Fisher EuroLab 16 mm twin-screw extruder

Rheology Solutions

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

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