ONset

NETZSCH
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TWO HOT ACQUISITIONS FOR A SUCCESSFUL FUTURE

Taurus Instruments AG & Rheology Division from Malvern Panalytical

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Editorial

Dear Reader:

I am very happy to present you today with the latest edition of our **on**set. In this edition, we are proud to announce two new M&As. The merger of TAURUS Instruments AG with NETZSCH Analyzing & Testing has resulted in today's NETZSCH TAURUS Instruments GmbH in Weimar, Germany. NETZSCH is thus expanding its product range for determining thermal conductivity and heat transmission and is further adding a comprehensive product line in the field of fire testing which is used in both materials testing and quality control. Along with various guarded hot plate systems, we now also offer large standard-compliant test chambers that can accommodate compact and complex components (e.g., windows, brickwork, etc.). The fire-testing instruments are based on the European classification system for reaction to fire and fire resistance.

We are also particularly pleased to add rheology to our thermal analysis product line, which had been missing from it until now; we have filled this gap via acquisition of the rheometer product line by Malvern Panalytical. The Kinexus rotational rheometer series along with the Rosand capillary rheometer series allow for the investigation of the flow and deformation behavior of a wide range of materials – from asphalt to shampoo. How a rheometer is used for testing in the asphalt and bitumen industry, for example, is described on page 15.

We would also like to introduce our new TMA 402 ***F3*** *Hyperion® Polymer Edition*. Starting on page 9, we report on this robust, reliable and easy-to-operate instrument for quality control, especially on polymers.

We would like to thank Johannes Görl and Dr. Thomas Neumeyer from the Institut für Neue Materialien Bayreuth GmbH for their informative contribution on determining the blowing agent content of expanded polystyrene by means of STA.

In the *Tips&Tricks* section, we will be showing how important it is to choose the correct measuring geometry for rheological investigations of your sample.

Under *PRECISE PRACTICE*, we demonstrate the influence humidity can have on medicines during storage and how to measure the moisture sensitivity of substances.

The first half of the year was marked for all of us by the outbreak of the SARS-COVID 19 virus. In order to continue to be there for you, we are breaking new ground with the "Virtual Lab" concept, which we would like to introduce in this edition and encourage you to utilize. Our Applications Laboratory is looking forward to hearing from you.

Under Events, you will find links to webcasts and webinars that might be of interest to you.

I hope you enjoy your browse through this 21st issue of **on**set!

Philipp Köppe
Head of Marketing

NETZSCH TAURUS INSTRUMENTS GmbH – Now Part of NETZSCH Analyzing & Testing!

Dr. André Lindemann, Managing Director of NETZSCH TAURUS INSTRUMENTS GmbH, Weimar, Germany

GHP 800 S – 90° position

GHP 900 S

GHP 500-1

TLR 1000

Good Things Come to Those Who Wait!

Today's NETZSCH TAURUS INSTRUMENTS GmbH in Weimar, Germany, is the result of the merger of TAURUS Instruments AG with the NETZSCH Analyzing & Testing Business Unit. In the field of thermal conductivity, the two companies had served the same market, but with different devices and specifications. With the additional fire testing from TAURUS, NETZSCH can now enter a completely new market.

In the field of thermal conductivity testing, we offer three instruments with guarded hot plates (GHP); additionally, we also offer pipe testers with a protected heating pipe for measuring pipe insulations as well as hotbox systems for measurement of the U-value of large and complex building parts (windows, doors, facades, etc.).

Our fire-testing product line includes the entire spectrum required for the establishment of a Eurolab for the legally prescribed testing of plastics, building materials, textiles, etc., in accordance with European standards. Fire tests can also be carried out in accordance with comparable standards worldwide. In the automotive, building, cable and plastic manufacturing sectors, there has been strong worldwide growth in demand for fire tests in recent years due to stricter safety regulations.

Guarded Hot Plates and Pipe Testers

The GHP series by NETZSCH TAURUS Instruments comprises instruments for measuring the thermal conductivity of products from the building industry such as insulation materials, building materials (bricks) and insulation glass. In addition to offering the advantages of the GHP method as a direct measurement technique without any need for additional calibration, the instruments in the series were also specially designed to handle a variety of sample geometries in the range from 100 mm x 100 mm to 900 mm x 900 mm. Flat insulation glass panels can be measured as well as vertically perforated bricks with a height of up to 380 mm. The GHP 900 S with tiltable test chamber is particularly well suited for investigating convective influences in cavities in insulating materials. Depending on the installation position (vertically or as a roof window),

for example, different effective thermal conductivities are obtained for the same insulating glass pane.

The TLR 1000 pipe tester for measurement of the thermal conductivity of pipe insulations also operates with a protected heat source. Additional calibration measurements are not necessary here either, as the electrical heating power can be calculated from the applied voltage and current. Under adiabatic conditions, the heating power corresponds exactly to the heat flow through the sample. By counter-heating, heat losses are prevented and the thermal conductivity can be determined directly provided that surface temperatures of the sample are known.

What Is a HotBox?

A hotbox is a test apparatus for determining the heat transfer properties of wall elements, walls, windows and doors. In a hotbox, real-world conditions as they would exist for a building wall are simulated: In practice, the conditions both inside the building and on the outside can be simulated. This means that temperature, humidity, air speed and radiation properties on both sides of the test wall must be detectable and precisely adjustable.

TDW 4240

There are usually two test chambers (warm and cold sections), and between them is positioned a test frame with the sample. Stone walls to be tested are first bricked onto a base and then dried in drying chambers; windows, doors or facade parts are inserted directly into the test frame. The prepared test frame is inserted between the warm and cold sections and can then be measured.

In NETZSCH TAURUS HotBox systems, homogeneity of the flow and temperature profiles is guaranteed to be in accordance with standards. This means that the same temperature and air speed must prevail on the test wall at every point of the warm and cold sections. To this end, numerous sensors near the wall or directly on the test wall surface are used for monitoring and securing.

What Purpose Do HotBox Tests Serve?

The heat transfer through a brick wall or a window part is defined by the U-value (k-value) in [W/(m^2·K)]. The lower the U-value, the lower the loss heat-flow $\dot{Q}$ (see equation 1) and the better the insulation properties of the building part.

The U-value is therefore a measure of the energy efficiency of parts of a building envelope such as brickwork, facades, windows and doors. Manufacturers have to specify the U-value of their products.

Differences Versus Conventional Methods for Measuring Thermal Conductivity

In contrast with conventional analysis methods for determining thermal conductivity (e.g., laser flash, plate method or transient source), the hotbox takes into consideration not only the heat conduction through the test specimen (purely dependent on the material), but also the heat transfer conditions (material property + ambient conditions). The surface structures of walls or transmission properties of window panes, for example, thus have a direct influence on the effective heat transfer.

The result of the measurement is either the effective thermal conductivity of the entire test wall (heat-flow method) or the U- or k-value (heat transmission

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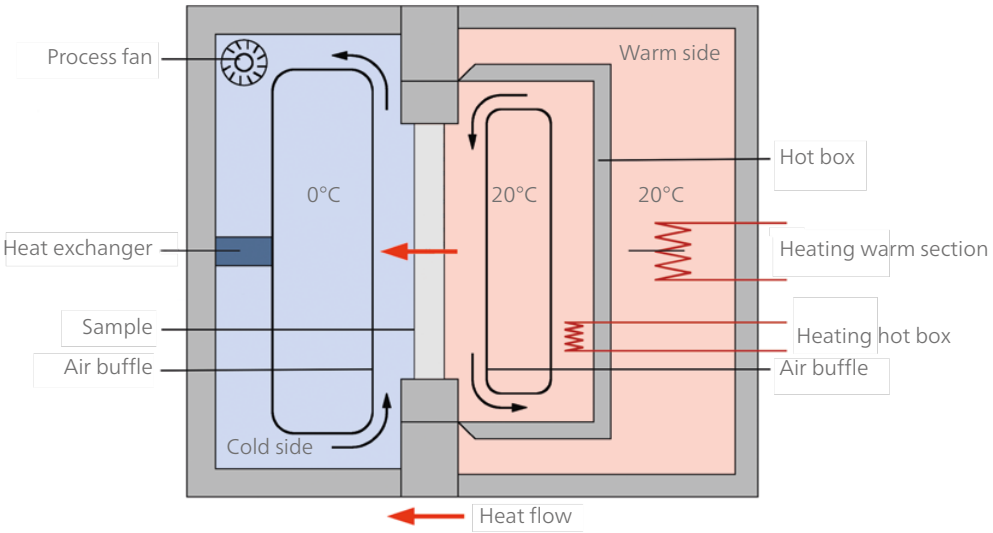

HotBox: Principle illustration of HotBox test chamber with heat flow meter

coefficient) as it relates to the heat flow between the air of the warm and cold sections (method with protected hotbox). The determination is based on the known equation for stationary heat transfer through flat walls:

$$\dot{Q} = k \cdot A \cdot (T_a - T_i) \qquad (1)$$

where
$\dot{Q}$ – Heat flow from the air of the warm section to the air of the cold section
k – Heat transmission coefficient
A – Wall area
T_a – Air temperature, cold section
T_i – Air temperature, warm section

The k-value refers to the thermal resistances between T_a and T_i and is calculated as follows:

$$1/k = 1/\alpha_i + s/\lambda_{Wall} + 1/\alpha_a \qquad (2)$$

with
α_i – Heat transmission coefficient, cold section
α_a – Heat transmisison coefficient, warm section
s – Wall thickness
λ – Thermal conductivity

For a hotbox measurement, all quantities of equation (1) are known and the k- and U-values can be calculated directly. Determination of the heat flow $\dot{Q}$ differs between the two hotbox methods mentioned above. In one case, the heat-flow sensor is placed directly onto the sample surface. Via a calibration measurement, the effective thermal conductivity of the test wall is obtained. In the other method with protected hotbox, there is an additional hotbox on the warm section. The heating power supplied to this hotbox essentially corresponds to the heat flow passing through the specimen to the cold section. The heat losses are comparatively low since the temperature is regulated to be the same outside the hot box.

Fire Testing

Fire testing has been increasing in importance. For reasons of safety, and per the corresponding regulations, it is necessary to classify products and materials into fire classes, for example. Various properties play a role here.

Particularly relevant for the construction industry is the reaction to fire of building materials, including parameters such as flammability, flame spread, flaming droplets, combustibility, heat release, smoke production, and the resistance to fire of building components!

It is possible to test the reaction to fire of all materials and structures used for construction such as facades, building and insulation materials, plastics, gaskets and floor coverings. Similar tests are standardized in the field of the automotive industry and focus mainly

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SBI Single Burning Item

on the flammability and flame spread of materials for the interior of vehicles such as interior lining and trim, seats and floor coverings.

In the field of the electrical and cable products industry, some of the areas investigated include flaming droplets for electronic and electrical parts,

TDW 4240 (open) with test mask for windows

smoke density, insulation integrity and the formation of corrosive gases upon flame impact. Textile products such as upholstery fabrics, curtains and drapes, decorative materials, floor coverings and protective clothing are tested and classified primarily for flammability, reaction to fire and dripping behavior.

The NETZSCH sales team and the colleagues in Weimar will be happy to answer any questions you may have about our products.

Product	Heat release	Smoke development	Flame spread	Flaming droplet	Non-combustibility	Ignitability	Burning time/ speed	Time to ignition	Mass loss	Heat of combustion	Combustion gas	Oxygen index	Euro classification/ Euro fire testing lab	Industry	Standards
KBT	x	x	x	x										Cables	EN 50399, IEC 60332-3
SBI	x	x	x	x			x						x	Building	EN 13823
TBB		x	x										x	Building	EN ISO 9239-1
TNB					x								x	Building	DIN EN ISO 1182
KBK						x							x	Building	DIN EN ISO 11925-2
TCC	x	x						x	x	x	x		x	Building, Automotive, Polymers	ISO 5660-1, ASTM E 1354
UL 94				x			x						x	Building, Electronics, Polymers	UL 94, DIN EN 60695-11, ISO 9773
LOI												x		Polymers	ISO 4589-2, DIN 22117, ASTM D 2863
HBK							x							Automotive	MVSS 302, GB 8410, IS 15061, CMVSS 302, U.T.A.C. 18-502, FAR 25.853
TRDA/ TRDL		x											Mostly integrated	Single/ Stand-alone	DIN 50055

NETZSCH Acquires the Rheometry Product Line from Malvern Panalytical

Dr. Shona Marsh, Application & Product Marketing Manager for Rheology

NETZSCH is delighted to announce that the Kinexus range of rotational rheometers and the Rosand line of capillary rheometers have become a recent addition to the product portfolio of the Analyzing & Testing business unit!

The introduction of rheometers into our portfolio has been a carefully considered acquisition. Due to the natural affiliation of rheometry with the products and application areas which constitute our brand in thermal analysis and materials testing, it has been a long-term desire to integrate rheometry into our company. We hope you agree that this is an exciting opportunity for us. Customers will continue to receive outstanding support and we look forward to establishing new relationships and collaborations over the coming months.

We have a variety of webinars, application notes and white papers at your disposal!

The Kinexus Rotational Rheometer Series

The rotational rheometer range consists of both the standard Kinexus platform and the Kinexus DSR. These rheometers possess an ultra-low friction air bearing which is what makes them so incredibly sensitive. In comparison to a simple viscometer, the performance of a rheometer allows far greater characterization of flow, deformation and even tackiness of a material (for Newtonian and non-Newtonian materials).

Kinexus Rotational Rheometer

The Dynamic Shear Rheometer Kinexus DSR is specifically tailored to test asphalt and bitumen and has a long-standing reputation within this market. It features a great variety of the rheology industry standards in the software. Its capabilities have been proven worldwide; our user list includes acclaimed universities, major asphalt producers and government installations all over the world.

The Rosand Capillary Rheometers

Capillary rheometers are designed to operate at much higher shear rates than rotational rheometers allowing the rheological behavior under processes such as extrusion or injection molding to be investigated. A capillary rheometer has two barrels which are filled with sample that is driven through a small diameter die by pistons at different speeds (shear rates). This instrument not only provides information about the material's shear viscosity (resistance to flow) but also the extensional viscosity (resistance to stretch). This means we can detect how different polymers / grades will perform in processes such as blow molding. However, there are many other application areas for which samples can be measured by a capillary rheometer such as inks, foods and personal care products.

Rosand Capillary Rheometer

If you would like to learn more about these products, please visit our website.
https://www.netzsch-thermal-analysis.com/en/products-solutions/rheology/

Thermal Expansion – A Major Reason Why Products Fail

Gabriele Stock, Marketing

Introduction

When developing electronics, polymer films or rubber seals, it is important to determine the glass transition – the point at which materials start to soften – in order to be able to specify safe operating temperatures. The dimensional compatibility of two or more materials is also important information for deciding on the correct material mix for a product.

Thermomechanical analysis (TMA) is a perfect tool for studying the expansion behavior and softening temperature of various materials such as thermoplastics, elastomers and composites. It provides fundamental information about the coefficient of thermal expansion (CTE), the glass transition temperature (T_g), and visco-elastic properties. It is a sensitive method and can be used to determine weak physical transitions that are associated with changes in modulus, curing, or delamination, which sometimes Differential Scanning Calorimetry (DSC) can't detect.

Fig. 1. TMA 402 ***F3*** *Hyperion® Polymer Edition*

TMA 402 ***F3*** *Hyperion® Polymer Edition* – Cost-Effective and Tailor-Made for Polymers

The new TMA 402 ***F3*** *Hyperion® Polymer Edition* (figure 1) is a robust instrument suitable for use in quality control, for example, and is offered as a ready-to-use bundle. The *Polymer Edition* comprises a compact highly reactive furnace which is connected to a mechanical cooling device. In this way, temperatures down to -70°C can be reached without the need for LN_2. The *Polymer Edition* includes a software- managed mass flow controller for gas rates from 0 to 250 ml/min.

The force exerted on the sample is generated electromagnetically. This ensures a quick response time for experiments with changing loads. In expansion, penetration and tension mode, the sample length is detected automatically. The newly added displacement control feature allows for stress relaxation experiments, in which the specimen is stretched and the required force is measured.

Another advantage of using the TMA 402 ***F3*** *Hyperion® Polymer Edition* is its smart software features.

The first of these is the *AutoEvaluation* one-click support system – an intelligent software functionality offered exclusively by NETZSCH which automatically and autonomously evaluates thermoanalytical measurement curves , thus serving as an immense help and time saver. Secondly, there is the optional *Identify* feature. The *Identify* database offers a means of verifying materials. It allows for the comparison of a measured curve with other individual curves available in the database – which is particularly valuable for quality control. It also makes comparisons with literature data from selected libraries. Any library and class created by the user can be edited or expanded within *Identify*.

Avoiding Product Failure in Electronics with Thermomechanical Analysis

Thermal expansion of material is a major factor when electronic products fail. This is why electronics manufacturing require the measurement of thermal expansion, glass transition and softening points under IPC standards [see IPC-TM-650 2.4.24.1 Time to Delamination (TMA Method)]. After the changeover to lead-free soldering processes, using soldering materials

with higher melting temperatures, manufacturers of printed circuit boards and assemblies realized that their printed circuit boards where delaminating due to the higher thermal loads. One of the reasons is that, despite higher requirements, most FR4 substrates are ordered even today with general material specifications and can vary in their material properties. At the glass transition event, the expansion rate of the epoxy matrix increases, which can lead to delamination between the fibers and matrix and consequently to product failure.

In the following measurement on a FR4 composite (figure 2), the time to delamination was recorded. Two measurements were conducted: one with an isothermal temperature of 260°C (according to IPC standard) and a second one with an isothermal temperature of 300°C. In the first measurement, no delamination effect was visible. In the second measurement, the time to delamination was recorded at 18.1 minutes after being held at an isothermal temperature of 300°C.

Figure 3 shows the typical color change as a sign of the beginning degradation. Neither of the measured samples show any visible delamination, whereas the TMA method is sensitive enough to detect it at 300°C.

Using the TMA 402 ***F3*** *Hyperion® Polymer Edition* for materials in the design and production process

Fig. 3. Typical color change as a sign of beginning degradation: left before measurement, in the middle after being measured at 260°C and right hand side at 300°C.

allows for the detection of material changes which are sometimes invisible, thus helping endure the functionality of a product.

Characterizing Anisotropic Behavior in Composite Systems

TMA can be used to characterize multiple properties of composite systems. It can be used to determine the glass transition temperature (T_g) in thermoset or thermoplastic matrix composites. At the glass transition of a thermoset, a decrease in matrix stiffness occurs. Due to the low strength of these polymer matrices in the rubbery state, the matrix can no longer function effectively to transfer load to the fibers or suppress fiber buckling above the glass transition. Determining

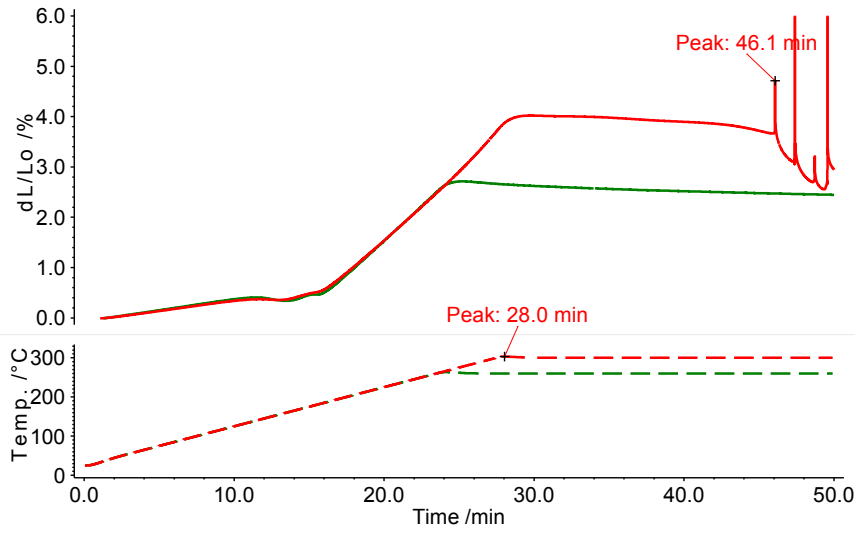

Fig. 2. Determination of time to delamination on an FR4 circuit board
Measurement conditions: sample width 6.35 mm (IPC standard), 2 h drying process at 105°C, heating rate 10 K/min, isothermal segment at 260°C and 300°C, respectively, N_2 atmosphere, sample holder for expansion (fused silica).

the start of the glass transition is therefore a good method for determining the upper temperature limit of these materials. For thermoplastic matrix composites such as PP-GF, T_g signifies the temperature at which the material starts to exhibit a more viscous behavior. Fibers and other fillers significantly reduce thermal expansion. The degree of anisotropy of the filler and the filler orientation pose great impact on the linear coefficient of thermal expansion (CTE). For example, uni-directional composites exhibit the CTE of the fibers in the fiber direction. They also exhibit a mixture of the matrix and fiber CTE as a function of fiber volume content perpendicular to the fiber direction. In the example below, the red curve depicts the measurement on a uni-directional PP-GF material in the fiber direction where the CTE is dominated by the low thermal expansion of the glass fiber. The sample 90° to the fiber direction (black curve) is dominated by the polypropylene matrix and shows a much higher CTE. Therefore, the T_g of PP is only observable in this direction (figure 4).

Conclusion

The TMA 402 ***F3*** *Hyperion® Polymer Edition* is now available and comprises a robust, reliable and easy-to-use instrument for quality control tests on polymers. Of course, other materials such as pharmaceuticals or food can be measured as well. If a wider temperature range is necessary – for example, for investigating metals or ceramics – the TMA 402 ***F3*** *Hyperion®* and TMA 402 ***F1*** *Hyperion®* are perfect for measurements in the temperature range from -150°C to 1550°C.

TMA 402 ***F3*** *Hyperion® Polymer Edition* – At a Glance

- New compact, fast-reacting IC furnace for temperatures down to -70°C without the need for liquid nitrogen. Existing customers can also update their TMA 402 ***F1/F3*** systems with this furnace.
- Smart displacement control for stress relaxation tests
- Automatic detection of the sample length in expansion, penetration and tension mode
- *AutoEvaluation*
- *Identify* (option)

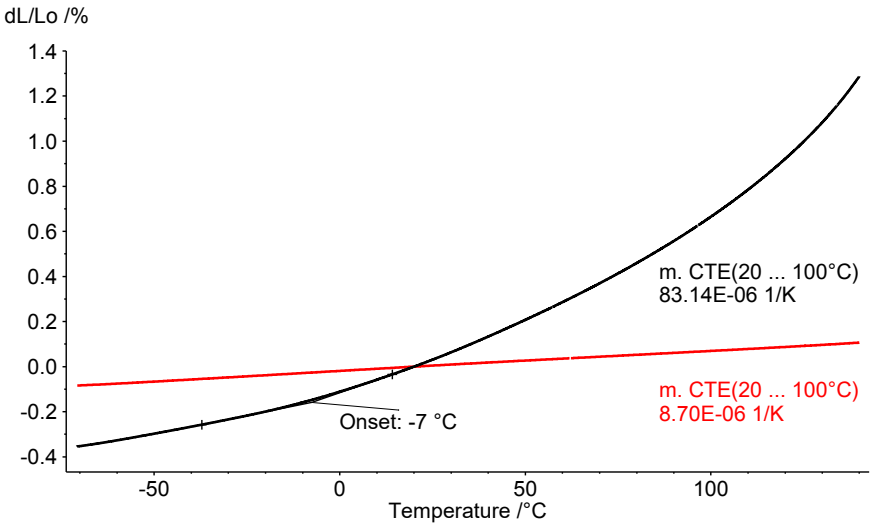

Fig. 4. TMA measurements on two PP-GF UD composite samples prepared in fiber direction (red curve) and 90° to fiber direction (black curve). Heating rate 5 K/min, N_2 atmosphere, sample holder for expansion (fused silica), sample length 25 mm, sample thickness 1 mm.

How to Achieve Consistent Product Quality in Expanded Polystyrene (EPS) by Means of STA

Johannes Görl and Dr.-Ing. Thomas Neumeyer, Neue Materialien Bayreuth GmbH, Germany

Microgranules | Pre-foamed material | Final product

Fig. 1. Blowing-agent-loaded polystyrene microgranules, pre-foamed beads and the final EPS product

Before diving into the topic of how thermal analysis can help in the quality control of expanded polystyrene (EPS), it is warranted to show how the material is produced.

Expanded Polystyrene Is Known to Everybody!

EPS is used as packaging material, e.g., for consumer electronics such as TVs or computers. It is also widespread in building and housing insulations due to its outstanding thermal insulation properties as compared to other materials. A major benefit of EPS is its low price compared to other materials for the application fields mentioned.

The starting basis for EPS parts are polystyrene microgranules that are homogenously enriched with a blowing agent – commonly pentane. The conversion process from blowing-agent-loaded polystyrene microgranules to foamed polystyrene products (see figure 2) is conducted in three stages:

- The first stage – the so-called pre-foaming – involves steam to heat up the blowing-agent-loaded polystyrene microgranules. The polymer softens and the pentane evaporates, hence the beads expand to about 40 times their original diameter.
- In the second step, the expanded polystyrene beads are stabilized for about 12 to 48 hours. During that storage time, remaining pentane diffuses out of the beads.
- In the third stage, the beads are fused together in a steam-chest molding machine to shape the final product.

An Important Material Component: The Blowing Agent

On delivery, the polystyrene microgranules contain around 5 to 7 wt-% of the blowing agent pentane. During pre-foaming, the blowing agent diffuses out of the material, leaving only 3 to 5 wt-% of blowing agent in the material. In the final product, less than 2 wt-% blowing agent remains by the time the part leaves the production machine.

The more blowing agent a microgranule contains, the easier it is to expand it in the pre-foaming process step. If the polystyrene microgranules have only a low pentane content, more energy is required to pre-foam the material. In order to precisely determine

Fig. 2. Steam-chest molding machine (on the left) and pre-foaming unit (on the right) for production of the EPS parts

Customers for Customers

the required processing parameters, it is important to know the blowing agent content. Furthermore, the residual blowing agent content at the end of the processing chain influences the flammability and dimensional stability of the final part. Currently, producers in the industry mostly rely on experience to adjust their processing parameters. However, such approximations make it challenging to ensure a consistent and reproducible product quality.

The most reliable method of determining the blowing agent content is gas chromatography. However, this laboratory method requires considerable effort and is not easy to implement in a production environment. Thus, Neue Materialien Bayreuth developed an experimental procedure to detect volatile contents along the EPS processing chain using thermogravimetric analysis. This way, homogenous temperature distribution inside the sample was achieved.

Thermogravimetric Analysis Is the Answer!

The NETZSCH STA 449 ***F3*** *Jupiter*® was used to measure the blowing agent content in the material in order to achieve a consistent product quality for expanded polystyrene.

STA allows for diverse application areas (TGA, TGA-DTA and TGA-DSC measurements). Furthermore, the instrument has quite a large furnace and crucible, which is important for the measurement of the EPS beads, as a certain volume of material is required in order to have critical mass inside the crucible. In this example, a 3.4-ml crucible was employed; the sample mass amounted to 76.34 mg.

Temperature Program

Starting at ambient temperature, the sample was heated to 120°C at a heating rate of 10 K/min in a nitrogen atmosphere. The heating rate was chosen because the beads feature good insulation properties. After heating to 120°C, the temperature remained constant over 25 minutes (figure 3).

At the beginning of the measurement, pentane (5 wt-% of the material) is equally dispersed in the microgranules. At about 40°C, pentane starts to evaporate and the material starts to foam. Pentane slowly diffuses outside the bead. When the pentane is fully diffused out, an equilibrium is reached. The mass loss remains constant.

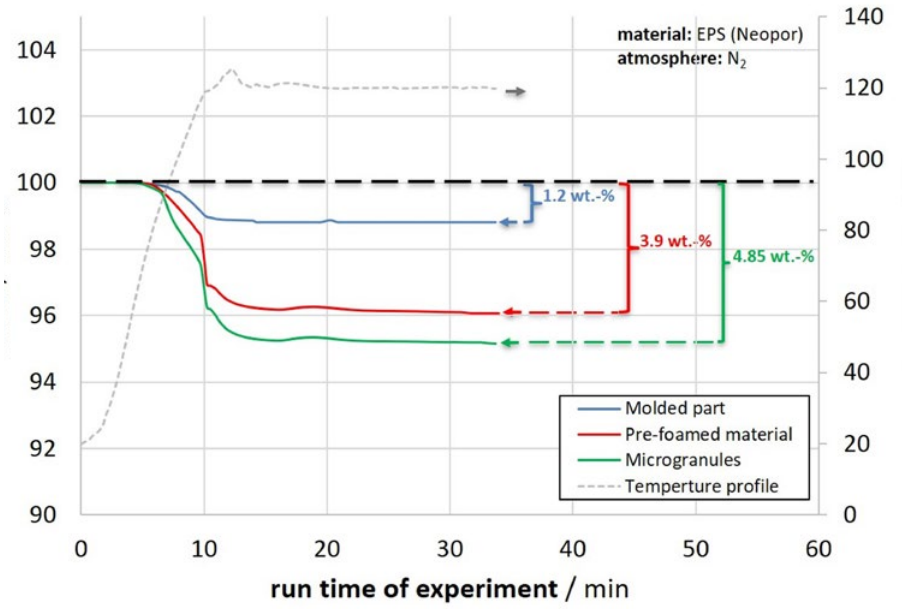

Fig. 3. Change in volatile content from microgranules to pre-foamed material and molded part

Customers for Customers

In the pre-foamed beads, there is a volatile substance (pentane) content of about 3.9 wt-%. And in the final product, the content decreases to only 1.2 wt-%.

Is It Only Pentane That Diffuses Out of the Material?

In order to answer this question, the gas emerging from the material was analyzed by means of gas chromatography. This analysis confirmed that the main substance being released by the material is pentane. 90% corresponds to the pentane derivatives and only 10% have been characterized as other substances. Thus, thermogravimetric analysis enables quite a good estimation of the pentane content in all steps along the EPS processing chain.

The simplicity of the method makes it suitable for use in the production environment of any EPS molder.

The Authors

Johannes Görl

Johannes Görl studied materials science and engineering at the University of Bayreuth. His main topics were polymer materials and metallic lightweight structures.

Since 2016, he has been working as a scientific assistant at the research institution Neue Materialien Bayreuth GmbH in the “Polymers” division. He focuses on the processing of established and novel bead foams, among them EPS, EPP and E-PET.

In his scientific work, he is investigating a steamless process for expandable polystyrene (EPS).

Dr. Thomas Neumeyer

Dr. Neumeyer has been leading the division “Polymers” at Neue Materialien Bayreuth GmbH since 2015.

Prior to that, he was a scientific staff member at the Department of Polymer Engineering at the University of Bayreuth (Department Head: Professor Dr. Volker Altstädt), where he also headed the group "Thermosets and Composites". In 2015, he finalized his PhD thesis, entitled "Structure-Property Relationships of Novel, Flame-Retarded Prepreg Resins for Applications in Aircraft Interiors”.

Since July 2016 he has also been the managing director of an industrial network for expanded polypropylene foams (EPP) called “EPP-Forum e.V.”. In addition, Dr. Thomas Neumeyer is currently a lecturer at the University of Bayreuth, teaching in the field of polymer composites.

Customized All-in-One Solutions for Asphalt and Bitumen Testing

Torsten Remmler, Sales & Application Rheology

Following our acquisition of the Malvern Panalytical rheometer line, our customers are now able – in addition to conducting thermal analysis – to also investigate the flow and deformation behavior of materials. In the road construction sector, our Kinexus rotational rheometer is well known as a "DSR" dynamic shear rheometer. It serves for the analysis of binder in asphalt – bitumen – with regard to its mechanical properties at different temperatures, load durations and acting forces. Binders are often modified to optimize stiffness, elasticity and viscous damping at both high and low road temperatures. With the DSR, it is possible to detect the impact of additives in bitumen with great precision.

Comprehensive Test Methods at infraTest

Currently, there are four field manuals issued by the German "FGSV" (Road and Transportation Research Association) for determining the deformation behavior of binders; the requirements set by these are met by the various Kinexus DSR base units. Moreover, there are many additional standardized testing methods for bitumen. These include, among others, the ring and ball method for the softening point, the needle penetration method, the ductility method, the Fraaß brittle point method and tests for bending stiffness using the BBR Bending Beam Rheometer.

infraTest, located in Brackenheim, Germany, and specializing in these test methods, is an experienced resource for any questions related to the testing of asphalt and bitumen. Their product portfolio ranges from innovative asphalt analyzers to asphalt and bitumen extraction equipment to numerous bitumen-testing instruments. At this year's "Asphalt Days" in Berchtesgaden, Germany, NETZSCH Analyzing & Testing and infraTest signed a collaborative agreement which allows us to now offer our customers tailored all-in-one solutions by means of the Kinexus DSC and various infraTest testing and analysis methods.

Aging Tests

These packages are not limited to testing equipment only, but cover all of the requirements in the field of bitumen analysis. For example, the short-term aging of bitumen – as it occurs during the production of asphalt in the mixing plant and during the paving of roads – can now be simulated by means of the infraTest RTFOT furnace. Over time, the binder in the asphalt continues to age, leading to changes in stiffness and elasticity. This long-term aging can be simulated using an infraTest PAV pressure aging vessel. The binders aged over the long- or short-term can then in turn be characterized by our Kinexus DSR to determine changes in mechanical properties as compared to fresh bitumen.

Two Partners – One Complete Solution for Our Customers!

The collaboration between NETZSCH Analyzing & Testing and infraTest offers our customers comprehensive know-how in the field of road construction and the entire range of different test methods from one source. We would be happy to introduce you to our measuring instruments by carrying out some test measurements together with you.

For further information, please visit
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Rheology – How to Select the Appropriate Measuring Geometry

Dr. Shona Marsh, Application and Product Marketing Manager for Rheology

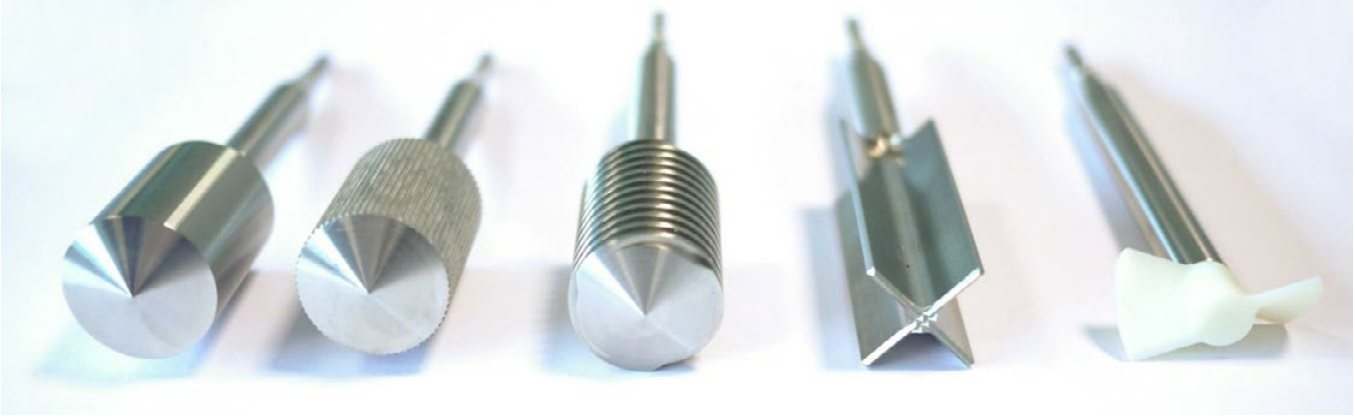

Fig. 1. Selection of upper bob geometries. From left to right: smooth, splined, spiralled, vane, paddle

Rheometers can measure the viscosity and viscoelasticity of a material by applying a range of shear deformations. In simple terms, the viscosity of a material is its resistance to flow and viscoelasticity can explain whether a material behaves more like a liquid ('viscous') or solid ('elastic'). This information can help scientists in R&D, for example, determine whether an intravenous drug can be injected or an oral dose can be swallowed, and even if it is likely to be a stable dispersion over time to prevent overdosing. It is also used in QC environments to assess whether a material passes or fails important performance criteria.

Kinexus Series

The Kinexus series of rheometers are class-leading rotational rheometers. These rheometers possess a custom air bearing making them incredibly sensitive to small material differences. Their torque sensitivity capabilities are even better than the equivalent of dropping an eyelash onto the instrument! What does that mean in practice? It allows you to easily measure materials under 'at rest' conditions. Therefore, we can determine whether products are going to be stable after having sat in the bottle on the shelf, i.e., their shelf life.

Geometry Choice

The measuring geometry selection is deliberately extensive. This is to ensure you have an appropriate measuring tool for both the type of test you wish to perform and the nature of your sample. The categories of standard geometries are: plate systems (parallel plates, cone & plates) and cylinder systems (cup & bobs).

Parallel Plates

These simple sets of flat upper and lower plates come in various materials, diameters and surface finishes, and are incredibly versatile.

- ***Size*** – ranging from 4 mm to 60 mm in diameter as standard. This wide range of sizes is available to accommodate for different viscosities. The smaller geometries (<25 mm) are suited to highly viscous (> 10 Pa·s) samples and the larger geometries (>50 mm) are for low viscosity (<0.1 Pa·s) materials.
- ***Surface finish*** – can be smooth, roughened (sandblasted) or serrated. Different surface finishes are available to accommodate those stubborn samples! Emulsions and slurries, for example, may be prone to slippage. This manifests itself as a lowering/drop in viscosity during a shear rate measurement. If you see a sudden drop in your viscosity and suspect slip, swap to using a roughened surface finish (see figure 2) for those samples. In order to encourage the material to flow, provide an extra grip using a modified surface interface.
- ***Measuring gap*** – can be changed with parallel plates. This flexible feature means that gaps can be tailored to match the samples' viscosity (i.e., smaller gaps for lower viscosity samples) and to achieve different shear rates. Smaller gaps subject samples to higher shear rates (for the same angular velocity),

Tips & Tricks

whilst larger gaps will only achieve lower shear rates. As a compromise for the modifiable gap with these measuring systems, an average shear rate is applied to the sample and therefore, results are not absolute (as with cones and plates). In addition, as a general rule of thumb, if particles are present, select a measuring gap of at least 10 times larger than the largest particles. This is to prevent particles from jamming during the measurement, which will cause artefacts in the results.

- ***Materials*** – the standard geometries on offer are made of stainless steel (SS316L) which is perfect for most laboratory environments as they are compatible with a wide range of sample types and can easily be cleaned with solvents. However, in some circumstances when working with acidic samples, a polymeric geometry may be more suitable. For example, PEEK and acrylic geometries (see figure 3) can be selected. The added advantage is that they are lighter and hence useful for high-frequency oscillation measurements on low-viscosity samples. In addition, titanium, aluminum and hastelloy steel geometries are also available.

Cones and Plates

Cone-and-plate combinations consist of a flat lower plate with an upper cone-shaped geometry and come in a variety of materials and surface finishes, e.g., roughened to prevent sample slippage. The tip of the cone is truncated and any measurements with these geometries are performed at a set gap (automatically controlled by the software). This is to allow for absolute viscosity measurements, so that wherever the sample is on the surface of this cone, it will be subjected to the same shear rate – a significant advantage over parallel plate geometries.

- ***Cone angles*** – the upper geometry angle can vary from typically 0.5° to 4°. The selection allows you to select your cone choice to achieve different shear rates. The smaller the cone angle, the higher the achievable shear rate. However, the presence of particles (and size) still needs to be considered. Cone and plates have a fixed (nominal) measuring gap; for a 1° cone, the gap is 30 microns; 70 microns for 2° cones and 150 microns for 4°. Particles still need to be at least 10 times smaller than these gaps to prevent them from jamming at the apex of the geometry. This can be a particular limitation for the use of cones with particulate dispersions considering the small truncation gap, and plate geometries are more suitable for highly filled samples as the measuring gap can be changed to accommodate for this. If no particles (or very small particles) are present, then no worries!

Cups and Bobs

Cup-and-bob geometries are simply a lower cup to accommodate the sample and an upper bob to measure it. Just like the other measuring systems, there are options for surface finishes and different materials. They are useful for lower viscosity samples because there is extra surface area which makes them more sensitive. The relatively large gap between the upper bob and the wall of the lower cup is advantageous if samples possess larger particles because they will not jam. However, for low- viscosity materials being measured with any larger gap, one needs to be cautious of the onset of Taylor (non-shear) flow affecting the results. This can be detected by a false increase in viscosity at higher shear rates. Cups can be selected with fill-up marks for ease of sample loading and with removable bottoms to allow for easier cleaning between measurements, although this is not as straightforward as cleaning a lower flat plate, so consideration should be given to how easy your samples are to clean.

Fig. 2. Lower pedestal plate to match a 20-mm upper geometry. Roughened surface finish.

Fig. 3. Alternative material upper plate geometries: PEEK and acrylic

Tips & Tricks

- ***Surface finish*** – for those slippery samples one can also use a roughened (sandblasted) or splined (~1 mm square pyramid "teeth") cup and bob. If there are particles present in the sample and sedimentation occurs, a spiralled bob may help slow/prevent the dispersion from settling during the measurement. If the dispersion is very unstable, then using a paddle will be more effective (see figure 1).

- ***Vane tools*** – are useful for measuring samples with very delicate structures such as foams or soft solids with a yield stress like yoghurt. The shape of the vane (see figure 1) lends itself to slicing into the sample without disturbing/destroying too much of the structure prior to measurement (in comparison to a solid bob).

- ***Double gap*** – for extremely low viscosity samples, these geometries are a good option. As can be seen (figure 4), the upper bob is hollow, providing an extra measuring surface area and consequently, improved sensitivity. The use of these geomteries is recommended for more volatile samples at elevated temperatures due to the relatively large volume requirements (for relatively volatile samples at elevated temperature, the double gap must be used with a solvent trap).

Questions to Ask Yourself

There is no hard-and-fast rule for selecting a geometry as this article highlights a number of factors which could come into play. But when considering a new sample and geometry selection, ask yourself:

What is the general viscosity of my sample?

- If you have a water-like low viscosity, select a large diameter cone/plate or plate/plate geometry (>50 mm).

Fig. 4. Double gap upper bob and lower bob

- If you have a free-flowing liquid (e.g., shower gel), a medium-sized geometry will work well (40 mm.)

- If you have a very stiff, thick sample (treacle), a small geometry should be selected (<40 mm).

- If you have a very low-viscosity or volatile sample, consider using a cup and bob or double gap. For evaporating samples, a solvent trap should be used.

Do I have particles in my samples?

- If the answer is yes, what size? The measuring gap should be at least 10 times larger than the largest particle size which can be changed for parallel plates.

- Cup and bob systems should also be considered, especially for settling samples where circulating grooved bobs are advantageous.

What is the composition of my sample?

- **Is my sample prone to slippage?** Emulsions or concentrated dispersions can slip on the smooth geometries. Consider using a roughened or serrated surface finish (for plates) and roughened or splined (for bobs).

- **Does my sample have a delicate structure?** A vane tool can be used on samples such as foams or soft solids for yield stress measurements.

- **Is my sample aggressive?** Acidic samples can be measured with polymeric PEEK materials instead.

Start with these simple questions and review your results. The Kinexus is very forgiving and provides extra information to give users confidence that they have selected the correct geometry. Its clever feature of being able to easily swap to a different geometry and automatic recognition will make testing new samples fun and effortless!

The Impact of Water on the Storage Conditions of Medicines and How to Detect It

Dr. Gabriele Kaiser, Business Field for Pharmacy, Cosmetics & Food

Many people store their medicine in the bathroom. But in the bathroom as well as in the kitchen, both the temperature and the concentration of water in the air are often high. Some active pharmaceutical ingredients (APIs) and excipients will interact with moisture from the environment. They may adsorb water molecules onto their surface, absorb water into their bulk structure or exhibit chemical interactions; in other words, they are hygroscopic.

But high hygroscopicity is not desired. It may lead to handling or manufacturing problems – for example, with respect to the powder flow – or it can influence the physical and chemical stability of the substance(s). Water can, for example, induce swelling, the formation of hydrates, hydrolysis or degradation reactions (even resulting in toxic degradation products), or it can affect a material's glass transition or degree of crystallinity. It is even possible for substances to start to dissolve in their own sorbed water; this process is called deliquescence [1, 2].

Because of this, it is crucial to learn within the development phase of a drug product how the API, the excipient or the formulation at hand will behave in contact with moisture.

Classification of Hygroscopicity

According to the extent of water uptake, pharmaceutical solids can be categorized into different groups. Two commonly used classification systems are juxtaposed in table 1.

Tab. 1. Comparison of the classification systems of hygroscopicity according to source: European Pharmacopeia vs. Callahan et al.

Source	European Pharmacopeia	Handbook of Pharmaceutical Excipients / Callahan et al. [3, 4]
Conditions	24 hour storage at 25°C/80% RH**(w/w)***	Storage at different RH ** values for one week
Classification		**Water uptake***
Non-hygroscopic		Class I: No water sorption below 90% RH, and <20% (w/w) at 90% RH
Slightly hygroscopic	0.2 to 2% (w/w)	Class II: No water sorption below 80% RH, and <40% (w/w) at 80% RH
(Moderately) hygroscopic	2 to 15% (w/w)	Class III: <5% (w/w) below 60% RH, and <50% (w/w) at 80% RH
Very hygroscopic	≥15% (w/w)	Class IV: >5% (w/w) below 60% RH
Deliquescent	Sufficient water is absorbed to liquify	

* corresponds to the mass increase over the specified time
** RH = relative humidity
*** (w/w) = mass fraction, equal to wt.%

PRECISE PRACTICE

Investigating the Impact of Temperature and Relative Humidity (RH) ...

One way of measuring the mass change of solids during water uptake or water loss is thermogravimetric analysis. This technique is also mentioned in the US Pharmacopeia, [5], or the European Pharmacopeia [6]. When not working under dry gas conditions but under a controlled humid atmosphere, it is possible to study the sorption/desorption capability of a substance. The corresponding method is called Dynamic Vapor Sorption, or DVS. There are instruments specifically for DVS available on the market. However, it is also feasible to carry out such measurements by using a Simultaneous Thermal Analyzer (STA) in combination with a humidity generator (see figure 1).

... Also Works with STA

The test run described in the following was performed with an STA 449 ***F3*** *Nevio* system in conjunction with a humidity generator, the latter of which is capable of generating relative humidity values between 5% and 90% within a temperature range of 30°C to 80°C. Typical carrier gases are nitrogen or synthetic air. In the present case, nitrogen was selected.

Fig. 1. STA 449 ***F3*** *Nevio* and humidity generator

Fig. 2. TGA sample holder with Al_2O_3 plate

41.2 mg of microcrystalline cellulose (MCC), commonly employed as filler or binder during tablet formulation, was placed on a plate made of Al_2O_3 (17 mm in diameter), which in turn was mounted onto a TGA sample holder (see Fig. 2). For such experiments, it is important to have a large contact area between the sample and the surrounding atmosphere. The sample mass, however, can also be smaller than that of the current experiment.

At a constant temperature of 44°C, the relative humidity was increased stepwise (5 steps in total) from 0% to 80% and subsequently decreased again (also in 5 steps). The result is shown in figure 3.

The first mass-gain step of 4% results from an increase in humidity level from 0% to 20% (blue curve). Subsequently, the higher humidity rises, the more the mass increases. With a humidity level of ultimately 80%, a mass increase of 12% occurs. As soon as the humidity decreases, mass losses take place. This happens until dry conditions (i.e., 0% humidity) are reached again. The final mass value at the end of the experiment is 100% and thus identical to the starting point, indicating that the water adsorbed/absorbed during the increase in moisture level has been completely released.

With regard to adsorption, the sorbate (in this case, water) is located on the surface; with regard to absorption, it penetrates the bulk. The term ´sorption´ applies to either of these phenomena.

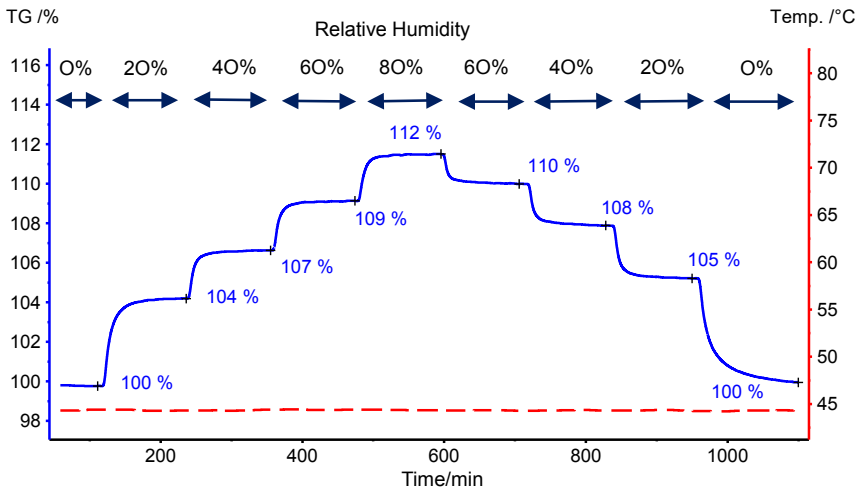

Fig. 3. Mass increase and decrease of microcrystalline cellulose due to variation in the relative humidity level between 0% and 80% at 44°C (red dashed curve)

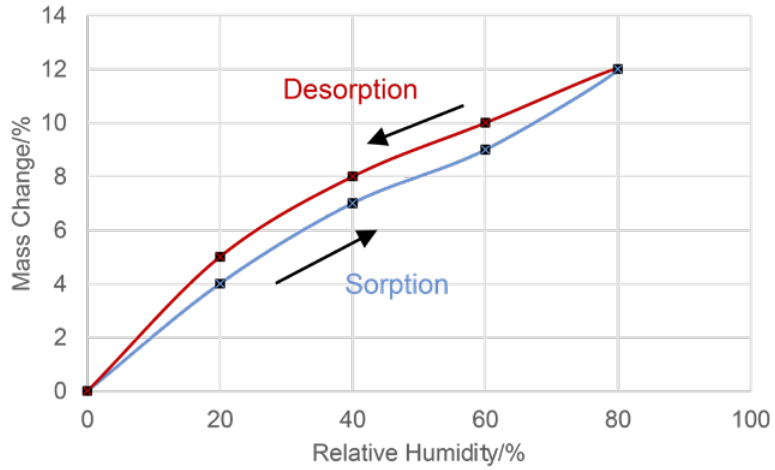

Fig. 4. Sorption hysteresis; plot of the applied relative humidity versus the resulting mass change

The mass-gain and mass-loss values after achieving equilibrium during increasing and decreasing the humidity level differ slightly. This is illustrated by the sorption/desorption isotherm plot in figure 4.

Desorption isotherms (red curve in figure 4) are of practical significance for drying processes.

Conclusion

There are several factors which may influence the shelf life of drug products; amongst them are exposure to heat, light (sunlight) and moisture. As a consequence, information about proper storage conditions – for example, the storage temperature – can be found on the outer packaging of a medicine and in its package insert. These notes are based on stability data for the drug collected by the manufacturer and/or by means of sorption/desorption studies.

Generally, it is recommendable to store pharmaceuticals at home in a cool (but not too cool) and dark place.

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