ONset NETZSCH
CUSTOMER MAGAZINE
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TG 309 *Libra*®*:* A New Era in Thermogravimetry

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Editorial

Dear Reader:

Let this new issue of our customer magazine, **on**set, take you into tomorrow's world of materials science! We present innovations and application examples that pave the way for new research opportunities, more environmentally friendly technologies, and the use of artificial intelligence in testing laboratories.

Our cover story introduces the next generation of NETZSCH thermobalances. The new TG 309 *Libra*® features an outstanding weighing resolution of up to 10 ng, an Eco Mode function, and an optional magnetic levitation system that eliminates external interferences. Like our DSC 300 *Caliris*® series, the new TGA is also available in three models, tailored to your individual application.

Hydrogen offers significant potential in industry to reduce greenhouse gas emissions and replace fossil fuels. But H_2 also harbors risks. Find out how our new development, *H_2Secure*, can help you achieve maximum safety in thermal measurements under a hydrogen atmosphere.

Animal-based foods such as meat, eggs and milk are also responsible for a large proportion of global CO_2 emissions. In the *CUSTOMERS FOR CUSTOMERS* section, the company ProteinDistillery GmbH reports about the production of vegan proteins based on brewer's yeast, for example, and its characterization by means of thermal analysis.

Excellent! True to our motto "Proven Excellence", the NETZSCH DSC 300 *Caliris*® was honored with the German Design Award 2024 in gold. Read more on page 17.

In this issue, we would also like to take you into the digital future with the AI-supported data management tool, LabV®. Read how the software can also transform your materials testing laboratory.

The *TIPS & TRICKS* section is all about rheological measurements. We reveal how you can easily screen the flow behavior of your samples with the viscosity toolkit in rSpace software.

Last but not least, I would like to draw your attention to our **on**set customer magazine survey: Your feedback is very important to us because we want to tailor our magazine to your needs, wishes and interests. Please take the time to answer five short questions. You can access the survey via the QR code on the right or via the following link: https://netzs.ch/onset-survey.

I hope you enjoy browsing through the current issue and would like to thank you in advance for your feedback.

All best,

Aileen Sammler
Content Marketing and Social Media Manager

TG 309 *Libra*® – A New Era in Thermogravimetric Analysis

Philipp Köppe, Head of Strategic Customer Engagement

Fig. 1. TG 309 *Libra*® *Classic, Select/Supreme*

The Perfect Instrument for Your Requirements

We are thrilled to introduce our latest innovation, the TG 309 *Libra*® family, marking a new era in thermogravimetric analysis. Crafted meticulously to meet the diverse needs of our valued customers, this product range not only boasts unparalleled usability and convenience but also upholds the vertical top-loading design that has stood the test of time.

The TG 309 *Libra*® series (Figure1) offers the greatest versatility for TGA users with an operating temperature range from 10°C to 1100°C. The series also offers outstanding balance resolution for detecting even the smallest of changes in a material's mass. In addition, it offers the ability to couple to gas analysis and to be equipped with a large, 204-position ASC (Automatic Sample Changer).

Our commitment to innovation shines through, as the new TGA series features a state-of-the-art operating concept and adheres to the highest technical standards. The instrument is available in three distinct versions – *Classic*, *Select*, and *Supreme* – each of which is tailored to cater to specific customer requirements, ensuring a seamless fit for every application.

Classic – Quality Control at Its Best

The *Classic* version, renowned for its exceptional price-to-performance ratio, emerges as the go-to instrument for quality control, offering a temperature range from 10°C to 1025°C and a balance resolution of 50 ng. With options for both display and illuminated information panels, coupled with software accurately crafted for quality control applications, the *Classic* version ensures both versatility and reliability.

Select – High Temperature and Top Resolution

For those seeking unparalleled customization, the TG 309 *Libra*® *Select* stands as a beacon of flexibility. From hardware to software, every aspect of this instrument can be precisely configured to meet the unique needs of our customers. With temperature ranges extending up to 1100°C and a balance resolution of 20 ng, it empowers users with unrivaled adaptability.

Supreme – Highest Balance Resolution

The crown of the TG 309 *Libra*® family is the *Supreme* version, meticulously engineered to exceed even the most demanding performance requirements. Whether in industrial R&D settings or academic institutions, its extensive feature set and temperature range of 10°C to 1100°C, along with a groundbreaking 10 ng in balance resolution, make it the instrument of choice for those pursuing excellence.

The TG 309 *Libra*® Family

Choose from among a Variety of Accessories

The cost-effective TG 309 *Libra*® *Classic* can be equipped with an optional automatic sample changer (ASC) for up to 20 samples and references, while the *Select* and *Supreme* versions offer up to 204 positions for your samples and references (Figure 2).

With its low-volume furnace, the TG 309 *Libra*® offers up to 20 times faster measurements, thanks to its high heating and cooling capabilities. It supports rapid heating rates of up to 200 K/min over the entire temperature range and fast ballistic cooling from 1100°C down to room temperature. This allows for faster analysis results, even at high temperatures, with a turnaround time of just a few minutes.

Exploring further into the core of this innovative device, it is its exceptional design that places strong emphasis on user-friendliness. In addition to its interface, which can be accessed both from the integrated touch display and a connected PC, the TG 309 *Libra*® now boasts an LED strip on the device housing. This LED strip provides a visual indicator of the current status of the measurement, offering users a quick and intuitive way to monitor progress.

The TG 309 *Libra*® can be equipped with various sample carriers to meet your requirements for any measurement task (see Figure 3). Furthermore, with the new generation of TGA instruments by NETZSCH, the sample carrier is automatically detected.

Moreover, external influences such as vibration are effectively mitigated through the magnetic levitation shock absorbers by NETZSCH, ensuring the integrity of measurements. The inclusion of features like *AutoVac* for automatic evacuation and gas filling underscores our commitment to efficiency. And, with the addition of the Eco Mode, we have demonstrated our commitment to a sustainable future.

Fig. 2. Automatic sample changer (ASC)

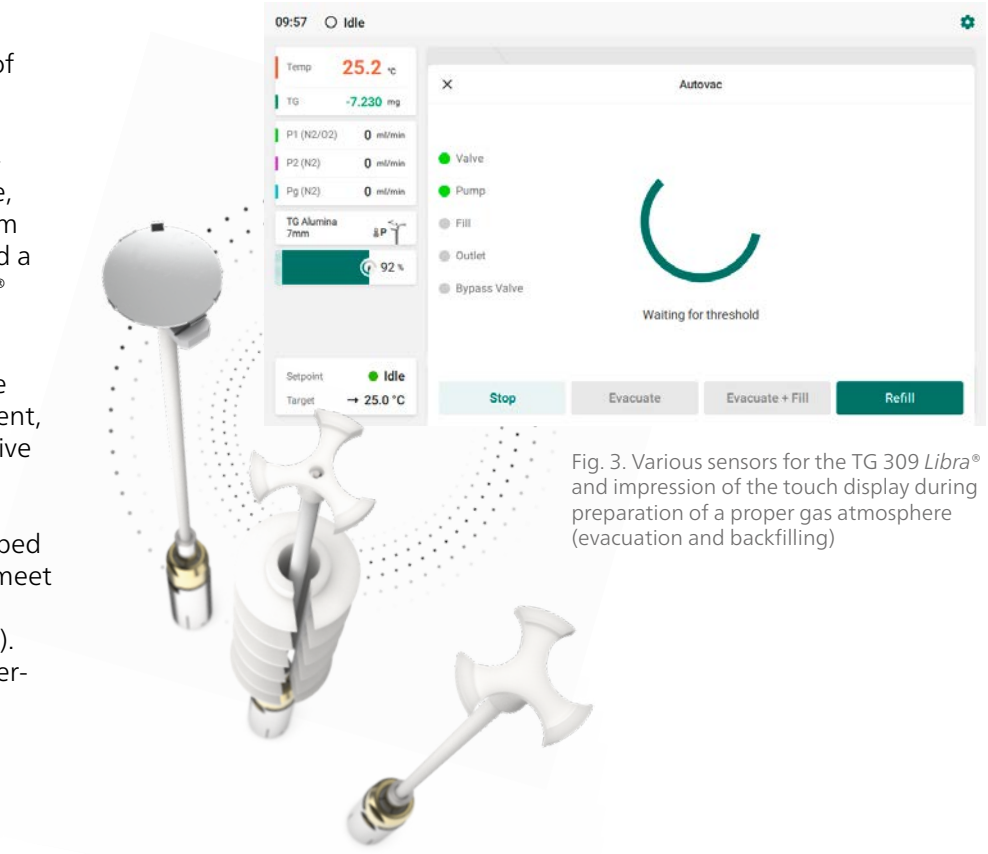

Fig. 3. Various sensors for the TG 309 *Libra*® and impression of the touch display during preparation of a proper gas atmosphere (evacuation and backfilling)

The TG 309 *Libra*® Family

Eco Mode – Maximizing Efficiency and Saving Energy for a Sustainable Lab

In today's drive for efficiency and sustainability, energy-conscious instruments are crucial. Traditional thermostats used in thermogravimetric analysis consume energy and produce waste heat, posing challenges for laboratories striving to reduce their carbon footprint.

The TG 309 *Libra*® series introduces an innovative Eco Mode to address these challenges. This feature allows for automatic chiller shutdown via software, significantly reducing operational costs and energy consumption. With customizable scheduling options, the instrument activates Idle or Eco Mode based on user-defined parameters.

This intelligent feature eliminates downtime associated with complete shutdowns by selectively reactivating essential functions as needed. Transitioning to Eco Mode can save up to 1 kW of electricity per instance, translating to potential annual savings of over 6000 kWh.

Beyond cost savings, embracing Eco Mode aligns with sustainability goals, reducing the laboratory's carbon footprint. The TG 309 *Libra*® not only offers precision and reliability, but also demonstrates a commitment to environmental stewardship, making it an ideal choice for efficient and sustainable laboratories.

Benefit from *BeFlat*® and c-*DTA*®

With the best balance resolution on the market and the ability to characterize sample properties comprehensively, the TG 309 series opens up new avenues for precise experimentation.

In addition to its advanced features, the TG 309 series comes equipped with user-friendly *Proteus*® software that can be upgraded for (almost) any advanced task, ensuring versatility and adaptability for years to come.

The patented c-*DTA*® technology sets new standards in thermogravimetric analysis by enabling more comprehensive and faster characterization of sample properties. By placing the sample thermocouple in direct contact with the crucible, temperature variations in the material are precisely measured. This approach allows for a thorough exploration of endothermic phenomena such as melting and exothermic effects like oxidation during thermogravimetric investigations. Furthermore, c-*DTA*® facilitates temperature calibration using DSC reference materials, enhancing the accuracy of results.

Traditionally, conducting a baseline run has been crucial to ensuring accurate mass change values in thermogravimetric analysis. This involved replicating test conditions such as heating rate, gas type, and flow. It was necessary to take various factors such as crucible type and geometry as well as instrument and buoyancy effects into consideration and subtract them from the sample measurement. However, with the TG 309 *Libra*®'s integrated *BeFlat*® baseline run for typical temperature measurements, the need for a further correction measurement is eliminated. This not only simplifies the testing process but also significantly accelerates the acquisition of results.

Application Example: Bicycle Tire Casing

TGA is widely employed for the compositional analysis of rubber, a practice outlined in standards such as ISO 9924 or ASTM E1131. In this example (see Figure 4), an examination was conducted on a bicycle tire casing. Rubber is a rather complex mixture of different components. The initial mass loss, at 8.7%, indicates the presence of plasticizer.

Following this, the subsequent two stages of rubber degradation can be observed. TGA proves effective in detecting even minute quantities of inorganic fillers like chalk. The mass loss observed at 664.5°C (as indicated by the DTG peak), accounting for only 1.2%, corresponds to the release of CO_2 from chalk decomposition ($CaCO_3$).

Transitioning to an oxidizing atmosphere at 850°C and further heating to 1100°C allows for the observation and quantification of carbon black combustion.

Application Example: Walnut Shell

Biomass serves as a renewable energy source for generating syngas, chemical precursors, and pure

The TG 309 *Libra*® Family

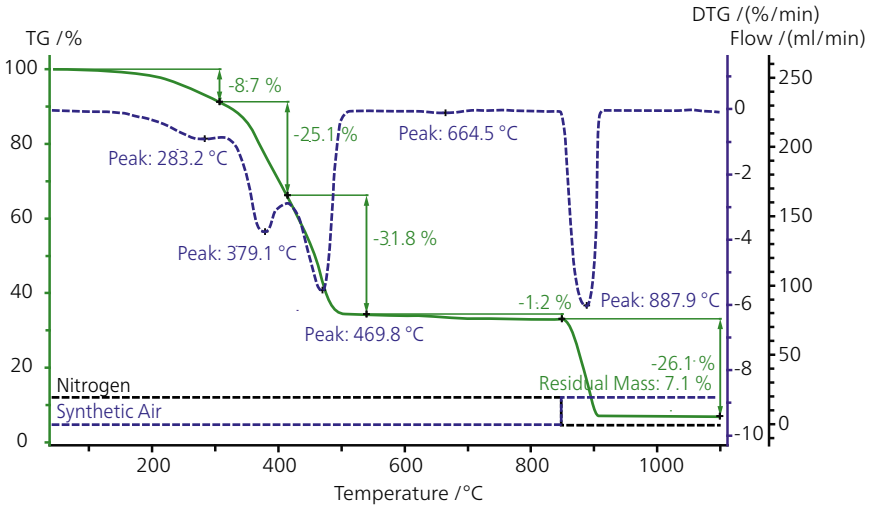

Fig. 4. TGA measurement on bicycle tire casing (9.79 mg, heating rate 10 K/min); TGA curve (green line), DTG curve (blue dashed).

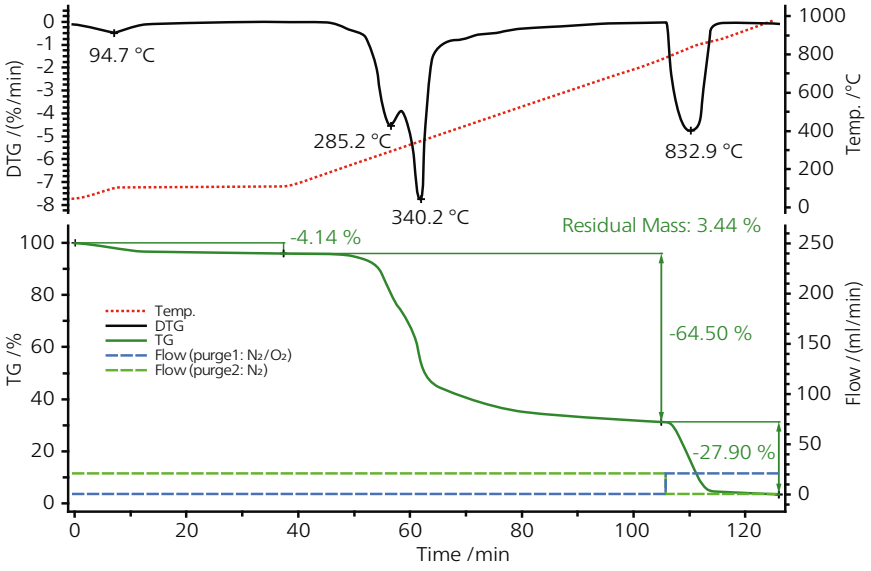

Fig. 5. Proximate analysis of walnut shell: TGA curve (green), DTG curve (black), temperature profile (red dotted).

carbon. Illustrated in this instance (see Figure 5) is the proximate analysis of a walnut shell sample to determine the content of moisture, volatile matter, fixed carbon and ash. Upon initial drying at 110°C, the substances released included 4.1% moisture.

Upon heating to 800°C in an inert atmosphere, two overlapping pyrolysis steps resulted in a total mass loss of 64.5%. These steps involved in the decomposition of organic components. Subsequent transition to an air atmosphere led to the combustion of carbon, which had accounted for 27.9% of the initial sample, thereby yielding CO_2. The residual ash content amounts to 3.4%.

Summary

In conclusion, we are immensely proud to present the TG 309 *Libra*® family, a testimony to our dedication to meeting and exceeding the needs of our valued customers. Join us as we embark on this journey of innovation and discovery together.

Find out more:
https://netzs.ch/libra309

H_2Secure – The All-in-One Solution for Hydrogen Use in Thermal Analysis Instruments

Dr. Michael Schöneich, Product Line Manager STA & EGA

Fig.1. Setup of *H_2Secure*

1 **Hydrogen Gas Supply**
Hydrogen can be supplied from an H_2 generator or H_2 cylinder and is connected to the special H_2 gas inlet on the rear of the STA with integrated safety valves.

2 **Optimized Gas Path**
This provides a precise concentration of gas, e.g., up to 100% hydrogen, while maintaining a protective gas atmosphere at the balance.

3 **Continuous Monitoring of Gas Concentrations**
STA exhaust gas flow is monitored for H_2 and O_2 concentration.

4 ***H_2Secure* Box**
Central communication box to control signals and allow or deny gas flows depending on the H_2 or O_2 limits defined.

Introduction

Hydrogen, the simplest and most abundant element in the universe, has long captured humanity's imagination. From its role in the formation of stars to its presence in the water molecules that sustain life, hydrogen has woven itself into the fabric of our understanding of the cosmos. However, in the face of pressing environmental challenges, it is now taking center stage as a crucial player in the transition to sustainable energy solutions. Its versatility and potential applications in transportation, energy production, and industry are garnering increasing attention, offering a promising pathway towards a cleaner, greener future.

As we explore hydrogen's diverse capabilities, from its role in fuel cells powering vehicles to balancing renewable energy grids and decarbonizing industrial processes, we embark on a transformative journey toward a more sustainable and resilient global energy landscape.

The Setup

The *H_2Secure* Concept (Figure 1) is available for our current STA 509 *Jupiter*® series and can also be retrofitted to existing STA 449 *Jupiter*® devices at any time.

Fig. 2. The four pillars of the *H_2Secure* concept

Defined H_2 Gas Volume
Hydrogen enters at the top of the furnace. H_2 is confined to a defined space above the continuously purged balance chamber.

Monitoring of H_2 and O_2
H_2 and O_2 gas concentrations are continuously measured to ensure safe handling.

H_2Secure Box
The central communication box receives gas concentration information and allows or denies gas flow based on set limits.

Fail-Safe Security
In the case of a power failure, magnetic valves open up and release inert gas, which removes hydrogen from the system.

H_2Secure

The H_2Secure Concept

The H_2Secure concept by NETZSCH is the complete solution for experiments in variable hydrogen environments while maintaining the highest possible safety standards. This innovative concept allows for research in pure hydrogen atmospheres as well as in environments with lower H_2 concentrations – diluted with non-flammable gases such as nitrogen (N_2) or argon (Ar). This flexibility is achieved through a comprehensive safety concept that is based on the four pillars (see Figure 2).

The Reversible Nature of Copper – Copper Oxide Redox Reduction

A key feature of the H_2Secure concept is the ability to seamlessly perform complex oxidation and reduction cycles. Within a single measurement, the system can switch between hydrogen-rich and oxygen-rich environments, allowing precise investigation of reaction kinetics and material behavior under different conditions.

The example presented illustrates a cycle experiment exploring the reversible reaction of copper oxide with hydrogen and air by monitoring the mass changes throughout the process.

Initially, copper oxide undergoes reduction in a hydrogen atmosphere, leading to the formation of metallic copper. This transformation is accompanied by an initial mass loss of 20.07%, which aligns closely with the theoretical expectation for pure copper oxide (20.11%). In the subsequent oxidation/reduction cycles, initiated by alternating access of hydrogen and air, the mass curve exhibits a consistent pattern: an increase during oxidation followed by a subsequent loss during the reduction step. This pattern emphasizes the reversible nature of the Cu/CuO redox reaction.

However, over the course of several cycles, a noticeable change in the oxidation behavior emerges. This change is indicated by alterations in the curve shape and the total mass change between the first and the following oxidation cycles. This change signifies a shift between the initial and subsequent cycles of the process, suggesting a change in the sample's reaction behavior.

This observation holds valuable insights into reaction kinetics, mechanisms, and thermodynamic properties of oxide-based redox reactions. By enhancing our understanding of these processes, it paves the way for optimizing redox systems and advancing various fields like catalysis and decarbonization of industrial processes.

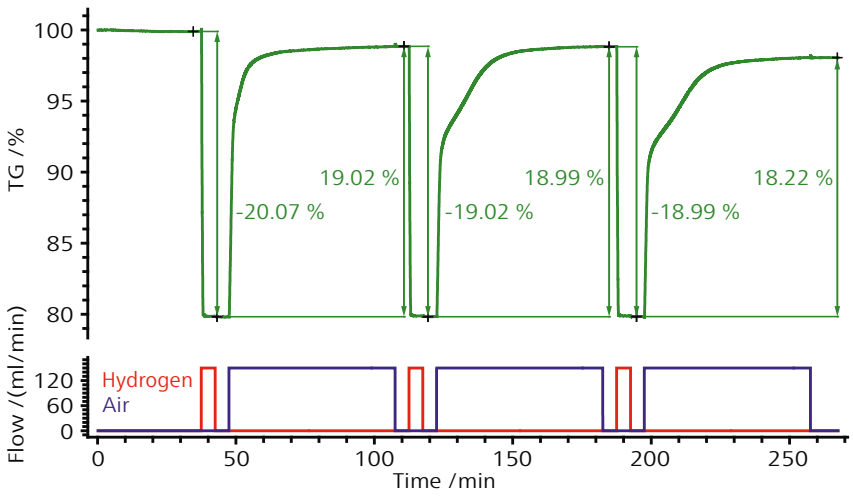

Fig .3. Thermogravimetric reduction-oxidation cycle of 29.975 mg copper (II) oxide powder at 1000°C

Summary

Hydrogen is a crucial element in today's cutting-edge research and developments. Our new H_2-Secure concept enables our existing thermal analyzers to significantly contribute to these advancements by opening up new application possibilities for them, all while providing a safe and easy-to-use experimental environment.

Artificial Intelligence: Precision and Efficiency in Modern Laboratories

Dr. Marc Egelhofer, Senior Marketing Manager, LabV®

Fig. 1. Only with complete connection of the test devices and the IT infrastructure can the database for AI use be formed.

Artificial Intelligence (AI) has rapidly evolved from a mere buzzword to an integral part of the technology landscape. Tools such as ChatGPT are representative of this progress. In testing laboratories, tailored AI applications have the potential to significantly increase efficiency and enable more precise control and optimization of laboratory processes through the use of advanced data analysis and automation. But how does this work in everyday laboratory practice?

Prerequisites for Using AI

The successful implementation of artificial intelligence in the test laboratory starts with structured and high-quality data. One tool that plays a central role in this is the digital twin – a digital copy of a physical object or process. By being continuously updated with real-time data, the digital twin enables comprehensive monitoring, analysis and optimization in the laboratory. The added value of AI is particularly well utilized when the digital twin integrates all four data levels: Formulation data, technical specifications, sample data and measurement results (Table 1). This comprehensive integration provides a holistic view of the entire process, increasing the benefits of the AI application.

In order to map the digital twin, it is therefore necessary to integrate all relevant data sources – from appropriate testing equipment to other sources of company information such as MES (Manufacturing Execution System) and ERP (Enterprise Resource Planning). This enables not only more comprehensive data collection, but also the integration of contextual information, which is essential for decision-making and process optimization by AI. With such a comprehensive database, intelligent systems can recognize patterns, make more accurate predictions and continuously refine their learning processes.

However, connecting all the devices and the IT infrastructure is a complex and often expensive task for the lab. The heterogeneity of data formats and standards, the complexity of integration, the continuous evolution of technology and the lack of generally accepted interoperability standards make this process considerably more difficult.

To solve this problem, LabV® has developed a patented mapper that enables the seamless and fast integration of any device – including all analyzers by NETZSCH-Gerätebau GmbH (Figure 1). LabV® allows a laboratory to combine data from different laboratory instruments, often originating in different formats, into a standardized database.

AI in Quality Assurance and Product Development

In the laboratory, it is obvious that even without the use of AI, holistic data management already offers added value. The systematic recording of all relevant sample information enables effective analysis and evaluation of laboratory results, which, in turn, supports quality assurance and product development. For example, the clear reports generated allow for the immediate evaluation of results in a product development project, facilitating selections for formulations based on the test data and ensuring that all customer requirements are met.

Table 1. The digital twin in the test laboratory

Data Level	
Sample data	Meta data, manufacturer's information, processing parameters
Measurement results	Meta data, measurement values and statistical evaluation across all measurement techniques
Formulation	Mixtures and ratios of all raw materials and chemicals used
Technical specifications	Physical and chemical properties, tolerances and dimensions, performance requirements, standards and other material information (e.g., procurement costs, MSDS)

LabV®

Once data are collected in a database like LabV®, the application of AI opens a range of possibilities for improving quality assurance and product development. In quality assurance, AI analyzes both historical and current data, identifies trends and provides early warning about possible quality problems. Intelligent algorithms facilitate complex analyses and offer immediate visual interpretations of data patterns, whereby decision-making is improved and the time spent on checking results is minimized. In product development, AI uses historical data to identify correlations between parameters that play an important role in the formulation and optimization of products. It helps identify potentially successful formulations even before physical testing, thus reducing development time and costs. AI can also help with the targeted adaptation of formulations in order to improve certain properties and minimize unwanted reactions, thereby speeding up the time to market launch for new products.

AI thus significantly contributes to increasing efficiency by integrating data from various sources and thereby providing an overall view of the development and quality assurance process.

At LabV®, an AI-supported Digital Assistant is integrated (Figure 2). The AI transforms voice commands into visualizations or complex analyses and is fully embedded in the data management platform. The assistant makes optimal use of your data, provides insights and helps increase productivity by focusing on the really important tasks and reducing the workload. With command input by voice or text, similar to ChatGPT, the Assistant enables time-saving data analysis and the transformation of data into comprehensive insights.

LabV® – The AI-Based Data Platform for Test Laboratories

Those who wish to use AI in the development lab or in quality assurance should first ensure the availability of an appropriately organized database. The performance of AI systems depends directly on the quality and structure of the underlying data. Thorough preparation of the data and the integration of IT systems therefore constitute the first step before implementing AI. This is because the strategic organization and management of data in a comprehensive database such as LabV® is crucial to reaping the full benefits of AI and making the laboratory future-proof.

For further information, go to https://netzs.ch/labV_en

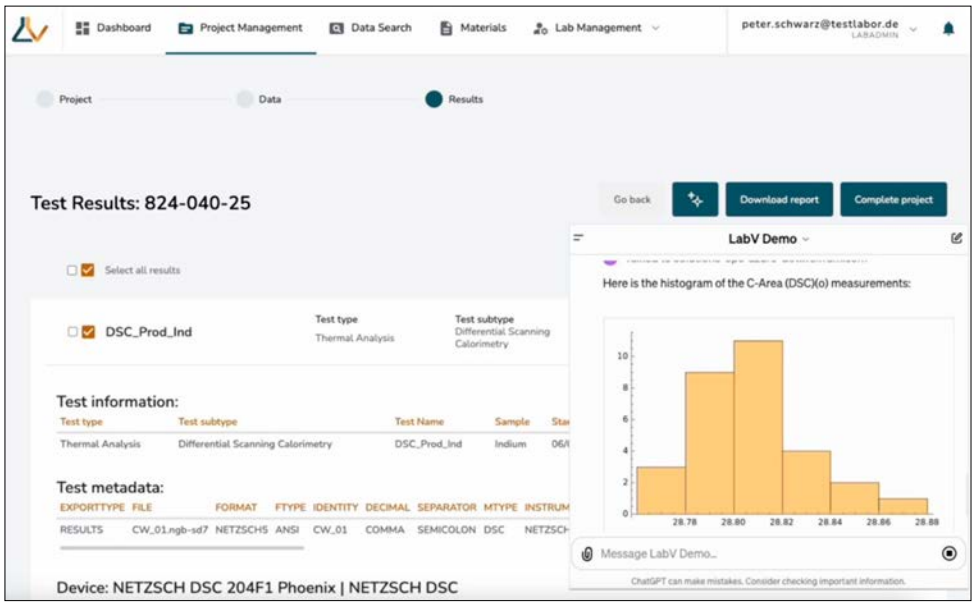

Fig. 2. The Digital Assistant of LabV® allows for complex analyses and visualizations at the touch of a button.

Creation of Clean Label Protein Ingredients with Superior Functionality and Nutritional Properties

Prof. Dr. Tomas Kurz, ProteinDistillery GmbH, Ostfildern, Germany

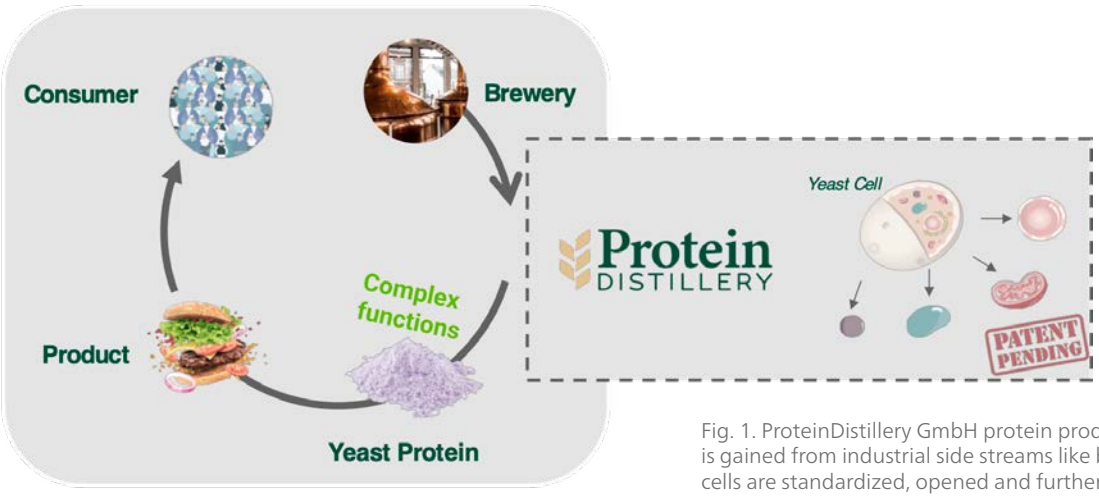

Fig. 1. ProteinDistillery GmbH protein production. Yeast biomass is gained from industrial side streams like breweries. The yeast cells are standardized, opened and further processed into specific components with specific properties.

About ProteinDistillery GmbH

ProteinDistillery GmbH is a Stuttgart-based start-up that is revolutionizing the alternative protein industry with its sustainable processing method. The company produces high-quality vegan protein through a unique refinement process. The production of the protein is based on one of the oldest human cultural techniques – fermentation.

ProteinDistillery GmbH uses a byproduct of the brewing industry and breaks down the beer yeast into functional building blocks to extract the valuable natural protein (Figure 1). The resulting protein exhibits remarkable techno-functional properties that are comparable to those of egg protein, making it a viable option for use in the food industry.

Animal-based food like meat, eggs and milk is responsible for a big part of the global CO_2 emissions and land use. Therefore, it is necessary to change our consumption behavior toward alternatives.

In this regard, the alternative protein market is forecasted to rise from a global volume of around 30 billion USD to 300 billion USD in 2035*. The main part of the alternative protein market is plant-based. However, when we have a look at the available products, we often are disappointed, as the properties of animal-based proteins in food regarding texture formation, taste and nutrition are much better than the properties of plant-based proteins like pea and soy. A compensation for the lack in taste and functionality must be made by using food additives like methyl-cellulose or aroma components.

Products of ProteinDistillery GmbH

ProteinDistillery GmbH is producing proteins from micro-organisms like yeast, especially brewer's yeast. With this approach, we can replicate the functional properties of animal-based proteins like egg white protein in the most sustainable way. Our protein principally behaves like an egg, which is the gold standard in the food industry. Therefore, our protein preparations can be used in a widespread field of food applications like meat replacer systems, egg replacers like scrambled eggs, or pastries and cheese.

Our product adds to our customer's final product through its properties like emulsion capacity, gelling, and thickening. Also, we must provide consistent physical properties to ensure the processability of our products. Therefore, it is of highest importance to know everything about the powder structure, as well as the rheological and denaturation properties of our product.

For each food application, there is a combination of necessary techno-functional properties. For the

* Blue Horizon & BCG analysis 2021, Food for Thought: The Protein Transformation | BCG

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Fig. 2. Different application fields of PD's yeast protein

production of plant-based egg analogues, solubility, gelling behavior and emulsifying properties are important, while foaming and emulsifying properties are more crucial for egg replacement in bakery products (Figure 2).

Determination of the Denaturation Temperature

Denaturation of a protein describes a structural change. Denaturation of yeast proteins can be measured by means of DSC (Figure 3), illustrated by endothermal effects in the temperature range between 40°C and 80°C within the first heating as well as by the characterization of the rheological behavior of the protein solution (Figure 4). At the onset temperature of denaturation (DSC), the intrinsic viscosity (rheometer) increases significantly. In the second heating step, no denaturation and a constant high viscosity level can be seen.

In addition, it is possible to create kinetic models for the denaturation rate of the protein at different heating temperatures on the basis of DSC experiments. These models are used to define heating profiles (temperature-time combinations) that will deactivate microorganisms without gelling the protein and thus enable pasteurization with the lowest possible impact on the proteins. The kinetic models can also be used for optimization of the gel formation in gelled products.

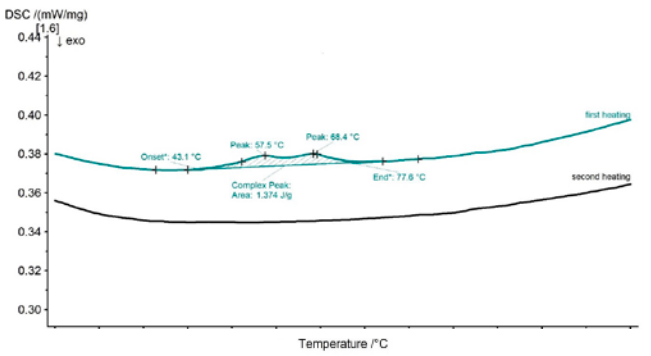

Fig. 3. DSC curve of yeast protein denaturation as a function of temperature. The onset, peak and end temperatures of denaturation are evaluated.

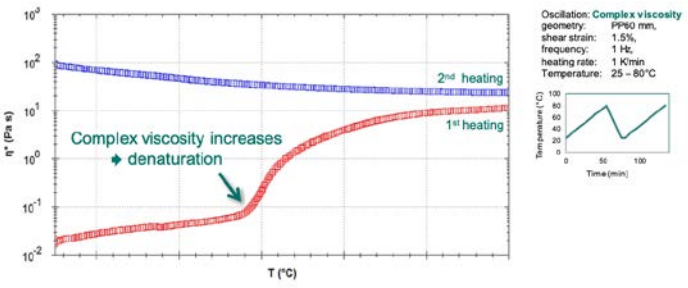

Fig. 4. Left: Rheological measurement of protein denaturation. Right: Temperature profile and parameters of rheological measurement.

CUSTOMERS FOR CUSTOMERS

Model-Based Simulation and Optimization of Protein Conversion in a Pasteurization Process Using Kinetics Neo

The general objective of pasteurization is to prolong the product shelf-life by deactivating all non-spore-forming pathogenic bacteria and the majority of vegetative spoilage microorganisms, as well as inhibiting or stopping microbial and enzyme activity. However, during heat treatment, proteins lose parts of their techno-functional properties like gelling or emulsification capacity. Therefore, especially for the functional protein product of ProteinDistillery GmbH, it is of the highest importance to gain knowledge about the denaturation/conversion behavior during heat treatment in order to find processing regimes which allow industrial users of the protein to pasteurize their product (e.g., alternative cheese products) and conserve the functional properties of the proteins as much as possible.

Table. 1. Simulation temperatures and times for batch pasteurization, High-Temperature Short-Time pasteurization (HTST), Ultra Pasteurization and Ultra-High-Temperature treatment (UHT).

Method	Temperature (°C)	Time
Batch (vat)	65	30 min
HTST	72	15 s
Ultra pasteurization	89 - 100	1 s
UHT	138	2 s

Food Safety Management, Chapter 17 - Thermal Treatment, Tibor Deak, Academic Press, 2014, pages 423-442, ISBN 9780123815040

Here, we used Kinetics Neo, a simulation and optimization software solution developed by NETZSCH, to describe kinetic reactions.

Standard parameters used in the food industry were chosen as a base for the thermal treatment of products or the protein solution. Table 1 gives an overview of these standard parameters. Pasteurization regimes can occur at low temperatures, like 65°C for 30 minutes, or for only 1 to 2 seconds at higher temperatures of 100°C or even 138°C.

Figure 5 shows an example of an applied temperature profile for analysis and prediction of DSC signals and the related occurring conversion of the protein fraction. In the left diagram, the temperature profile of a measurement at a heating rate of 5 K/min is displayed as an example. The right diagram illustrates response signals in the DSC for heating rates of 5, 20 and 50 K/min, which represent conversion processes in the protein solution.

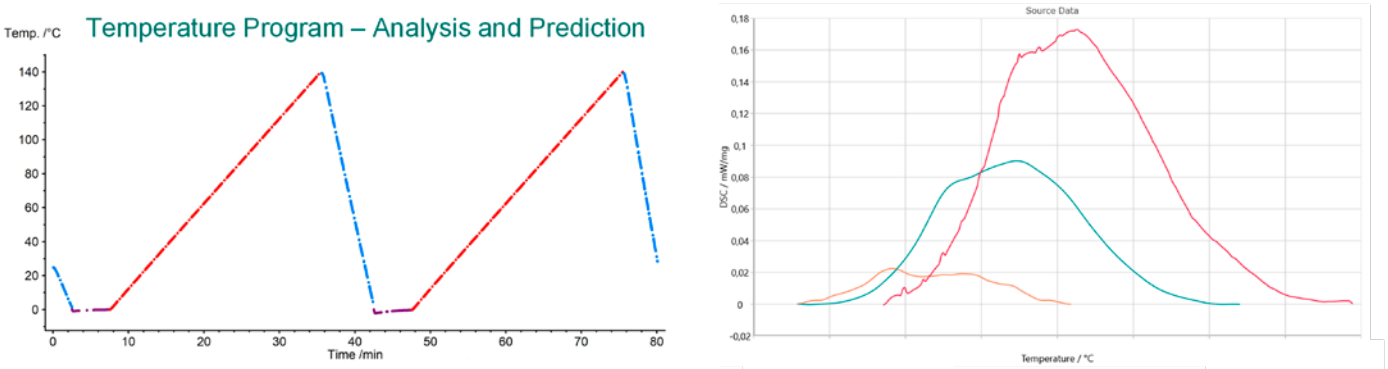

Fig. 5. Left: Temperature profile used for DSC analysis and prediction at a heating rate of 5 K/min; Right: DSC curves for yeast protein denaturation at different heating rates between 5 and 50 K/min as a function of temperature.

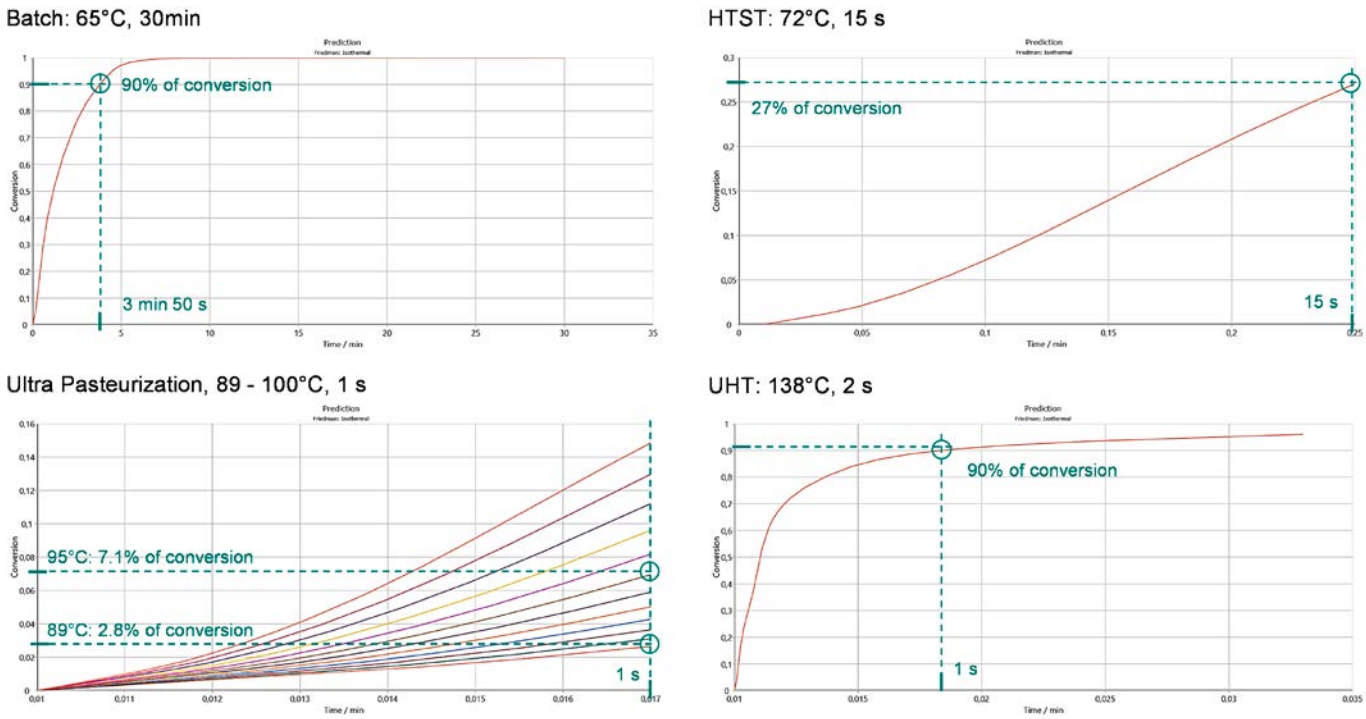

Fig. 6. Different simulation runs based on the pasteurization parameters in Table 1 (Batch 65°C, HTST, Ultra Pasteurization and UHT). Shown are the conversion rates as a function of treatment time with the different pasteurization regimes.

Heating rates of 50 K/min result in a significantly larger DSC signal than the regarded lower heating rates. Based on these DSC signals, it was possible to establish a time- and temperature-dependent model for the conversion rate; this is the basis for the model-based simulation runs shown in Figure 6.

Here, the pasteurization regimes of Table 1 are simulated. A batch-pasteurization at 65°C yielded a conversion rate of ca. 90% after 3 min and 50 s, which was only a small portion of the necessary 30 min. A High-Temperature Short-Time pasteurization (HTST) at 72°C resulted in a conversion of 27% of the protein after the targeted 15 s of treatment. Also, an Ultra-High-Temperature (UHT) treatment at 138°C resulted in an excessively high conversion rate of 90% after 1 s of pasteurization.

However, Ultra Pasteurization regimes in a temperature range of 89°C to 100°C showed promising results. After a treatment time of 1 s, for example, conversions of 2.8% and 7.1% occurred at 89°C and 96°C, respectively.

In order to verify the simulations, a calculated DSC signal based on the temperature profile given in Figure 7 was compared with a real measurement curve.

Summary

Based on these results, it was possible to find a practicable processing window for a customer processing plant and to apply the yeast protein from ProteinDistillery GmbH at the respective plant, including the heat treatment step.

It was also possible to validate the model with experimental data. As an example, Figure 7 shows a temperature profile (top), model-based simulation data (middle) and experimental DSC measurements. The model-based simulation describes the experimental data well. Therefore, this model can be considered valid for the application field in question.

CUSTOMERS FOR CUSTOMERS

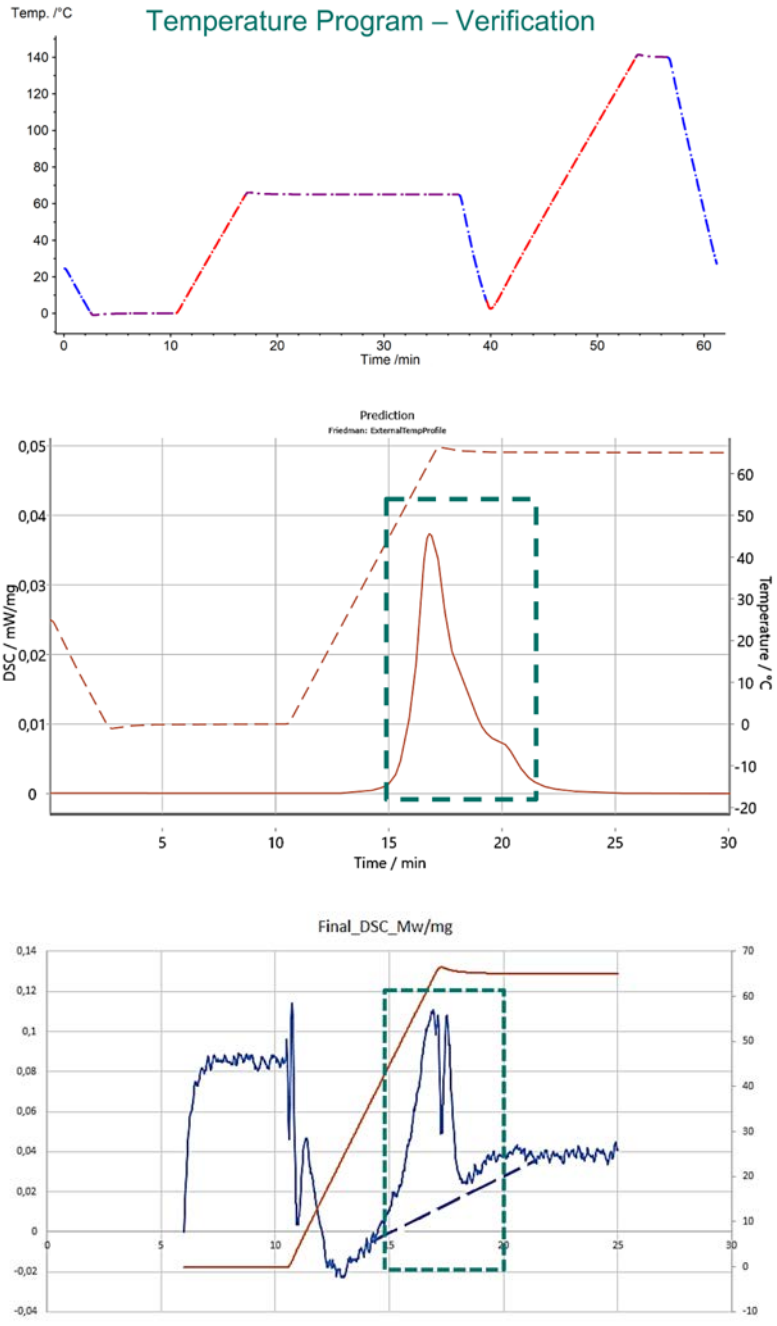

Fig. 7. Verification of the Kinetics Neo model for the yeast pasteurization process. Top: Pasteurization temperature program. Middle: Model-based prediction with Kinetics Neo. Bottom: DSC measurement data for protein denaturation process. Given is the DSC signal as a function of process time while applying the chosen temperature regime.

The Author

Prof. Dr. Tomas Kurz holds a degree in Brewing and Beverage Technology from the Technical University of Munich. After completing his PhD in bioprocess engineering, he was appointed as a junior professor of food process engineering at the Technical University of Berlin. He has extensive industrial experience as a research and development manager at various companies, specializing in alternative proteins, fermentation process development, hydrocolloids, and vegan food systems.

As technical director of a hydrocolloid production facility, he was responsible for equipment planning, maintenance and repair, personnel management, and production with over 100 employees under his direction. As head of product and operations, he is now responsible for the application technology of the manufactured products as well as the transfer of processes from laboratory to pilot and industrial scale.

Acknowledgement

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NETZSCH Is German Design Award Winner 2024 for Excellent Product Design

Aileen Sammler, Content Marketing & Social Media

Fig. 1. Ceremony during the Awards Show in Frankfurt in January.

The German Design Award is presented annually by the German Design Council, Germany's trademark and design authority, which is also the highest authority for new developments in the German design industry.

Fig. 2. Our two winners:
DSC 300 *Caliris*® and DMA 303 *Eplexor*®

True to our maxim *Proven Excellence*, our DSC 300 *Caliris*® *Supreme* & *Select* received their "Gold" Award. This is the highest honor awarded for excellent, holistic and innovative design achievements!

Our DMA 303 *Eplexor*® also received their the coveted "Winner" award for outstanding and exemplary design achievements!

Established in 1953 as a foundation on the initiative of the German Parliament, the German Design Council supports the industry in all matters consistently aimed at generating added brand value through design. This makes the German Design Council one of the world's leading competence centers for communication and brand management in the field of design.

The winners of the German Design Awards are determined by an independent international jury of experts comprising representatives from industry, university and design. The Awards set international standards for innovative design developments and competitiveness on the global market.

Together with the product design team of hoch^{E} – Designing Emotional Identity, we are proud of actually winning two awards!

(Source: German Design Award | The award for excellent design (german-design-award.com))

How the NETZSCH Toolkits Help Easily Understand Your Material

Claire Strasser, Applications Specialist Thermal Analysis and Rheology

Introduction

During testing with a rotational rheometer, a material is typically placed between two parallel plates or between a plate (lower geometry) and a cone (upper geometry). Typical measurements are split into:

- Rotational measurements, during which the upper geometry rotates at a defined speed. The typical result of such a test is the shear viscosity, i.e., the material's resistance to flow.
- Oscillation measurements, during which the upper geometry oscillates at a defined frequency and shear strain. Thus, the viscoelastic properties of the sample, i.e., its behavior before it begins to flow, can be quantified.

Along with having many selectable parameters (geometry type, measurement gap, temperature, shear rate, shear strain, etc.), the measurements may be very dependent on the sample structure and sample preparation. Stability, thixotropic behavior and the existence of a yield stress are all factors that must be considered for proper understanding of the resulting rheological curves.

The rSpace software for measurement and evaluation includes toolkits used for typical viscometry and oscillation tests. These toolkits are methods containing short explanations, appropriate default parameters and easy-to-follow steps, illustrated with pictures, so that even beginners in the field of rheology are able to perform their first measurements without difficulty.

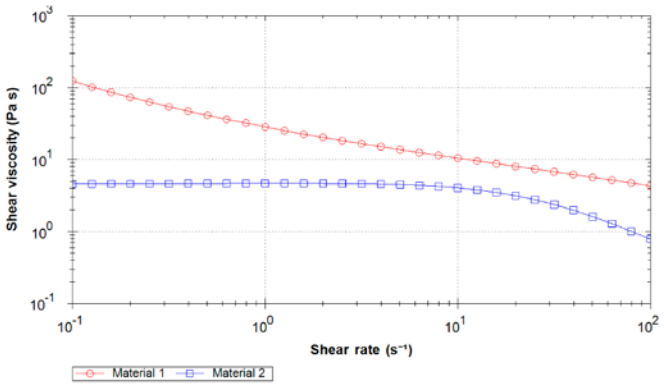

Fig. 1. Time-independent shear viscosity curves of material 1 (red, circles) and 2 (blue, squares) obtained with Toolkit_V001.

In the following, we show how to use the viscometry toolkits to get an initial screening of the sample's flow behavior in the easiest way.

Toolkit_V001: Shear Viscosity Curve

The shear viscosity curve of a material, i.e., its viscosity dependence on the shear rate at a defined temperature, provides the first insights into a sample's behavior. The rSpace Toolkit_V001 (Shear Rate Table) describes the method for this type of test. Used with the default parameters, it begins with an equilibration step of 5 minutes at the defined test temperature. During this time, the sample goes to the requested temperature and relaxes, i.e., the structure is recovering from the stresses imposed during sample preparation (application on the lower plate; squeezing while the upper geometry is lowering to reach the measurement gap). After equilibration, the test starts.

Figure 1 depicts one example for the resulting shear viscosity curves of two shower gels. Here, both products are shear thinning for shear rates higher than 10 s^{-1}; i.e., the shear viscosity decreases with increasing shear rates.

Their behavior differs at low shear rates. While the shear viscosity of material 1 goes to infinite with decreasing shear rates (red curve), it reaches a constant value (zero shear viscosity plateau) for material 2 (blue curve). This indicates a different behavior near rest. Material 1 is a viscoelastic solid with yield stress and good long-time stability. The second one is a viscoelastic liquid with good leveling properties.

Toolkit_V002: Thixotropy Test

The thixotropy of a shear-thinning material is related to the time the structure needs to recover after the breakdown due to the process. The more time the sample structure needs to recover, the more thixotropic it is. A typical daily application that illustrates this is the non-thixotropic behavior of toothpaste: As soon as it has been squeezed out of the tube, the structure recovers immediately, so it does not flow on the toothbrush.

TIPS & TRICKS

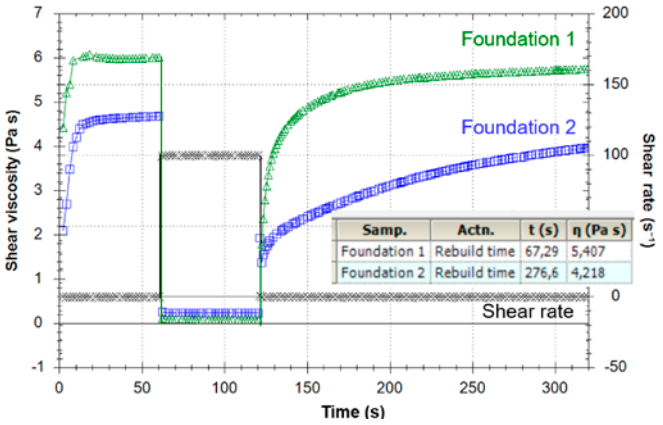

Fig. 2. Thixotropy test on two different foundations.

By means of the thixotropy test (Toolkit_V002), the thixotropic behavior of materials can be tested. It was used for the measurement shown in Figure 2 that compared the thixotropic behavior of two foundations. In each test, a 3-step sequence was applied consisting of:

- Low shear-rate step (0.1 s^{-1}) to mimic the sample's behavior near rest. Here, foundation 1 has a higher shear viscosity than foundation 2 and will most probably show better "stand-up" properties.
- High shear-rate step (100 s^{-1}) to mimic the break-down of the sample's structure during processing. Both products are shear-thinning ,so the shear viscosity decreases.
- Low shear-rate step (same shear rate as step 1), during which the recovery of the material is compared, i.e., the time required to reach initial viscosity. Foundation 2 is more thixotropic than Foundation 1 and will need more time to recover (277 s to 67 s). The rebuild time (here: time to reach 90% of the initial viscosity) is automatically determined and displayed after the measurement.

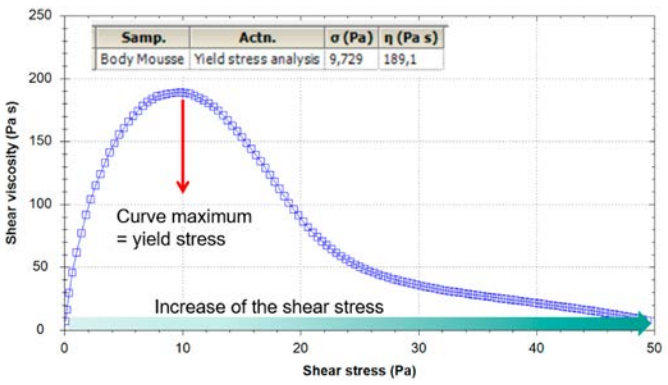

Fig. 3. Yield stress analysis of a body mousse; geometry: Vane tool: 25 mm; gap: 1 mm; temperature: 25°C; shear stress: 1 to 50 Pa.

Toolkit_V003: Yield Stress Test

Yield stress is the shear stress above which a material begins to flow. A linear shear stress ramp is specified during the measurement.

Figure 3 displays a typical resulting curve of a yield stress analysis. At the beginning of the test, the material deforms elastically and the viscosity seems to increase. It should be noted that the viscosity measured during this test is not time-independent, in contrast to the values measured in Toolkit_V001. At the peak maximum, the structure breaks down and the sample begins to flow. This is the yield stress. The test results are automatically determined at the end of the measurement.

Summary

With its Toolkits, rSpace measurement and evaluation software accompanies junior rheologists during sample loading while also suggesting parameters for typical tests. Of course, the Toolkits can be modified as desired so experienced users will be able to write their own methods, including, for example, automatic analysis and evaluation routines.

Please scan our QR-code to get an overview of the typical viscometry tests and possible results.

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Imprint

Editor
NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42
95100 Selb
Germany
Phone: +49 9287 881-0
Fax: +49 9287 881-505
at@netzsch.com
www.netzsch.com

Editorial Staff
Dr. Gabriele Kaiser, Doris Forst, Dr. Ekkehard Füglein, Dr. Elisabeth Kapsch, Philipp Köppe, Aileen Sammler, Dr. Ligia Elena de Souza

Layout
Doris Forst

Translations
Doris Forst, Nicole Unnasch

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