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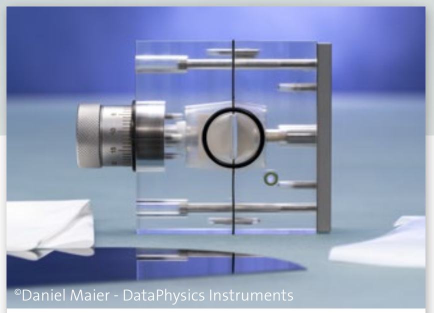
DataPhysics Instruments Company Magazine

Beyond the Surface

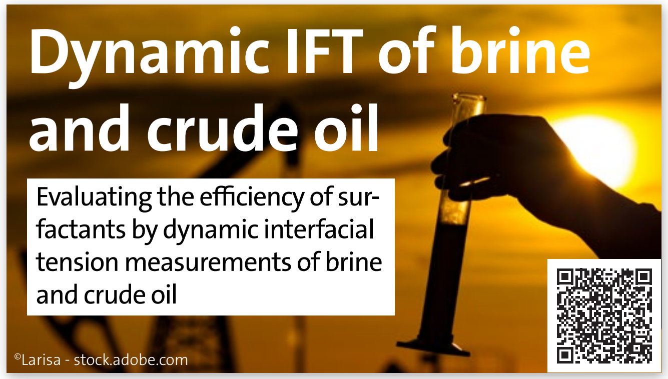
Milestones of our Company's Success Story



IPF Dresden Validates
ZPA 20 Zeta Potential Analyzer

Use Case: Developing Innovative
Tablets Using 3D Printing

How Surface Studies
Enhance Battery Technology



Editorial

A Reason to Celebrate

Nils Langer
CEO, DataPhysics Instruments

Dear readers,

Welcome to the very first issue of our company magazine *Beyond the Surface*. It is being published in what is a very special year for DataPhysics Instruments, because 2022 marks our 25th anniversary. In this quarter of a century, our premises and workforce have grown, and the business has established operations in more than 80 countries. On top of all that, our company has continuously expanded its product portfolio. We believe that this is a good reason to look back on recent years in this issue and share the highlights of our story to date with our clients and partners around the world.

Beyond the Surface will be published at regular intervals in the future to provide information about our product developments and company news. We also regularly release application notes, because studies based in real-life use cases paint the most vivid picture of what our measuring instruments can do.

We have chosen battery research as the focus topic of this first issue, as the development of modern battery systems is clearly on the upswing. What is more, they are a major topic in numerous fields of research, because they play a crucial role in sustainable energy storage solutions.

As we focus on developing our products further, we constantly expand our portfolio. Alongside the MultiScan MS 20 dispersion stability analysis system, which we launched some time ago, we are particularly proud to have achieved market readiness with our patented ZPA 20 zeta potential analyzer in this special anniversary year. The ZPA 20 makes it possible to measure the surface charge of solids, non-woven materials and powders in aqueous solution. It takes less than a minute to run a measurement with the ZPA 20, making it one of the quickest measurement systems on the market today. Both devices are presented in detail in this magazine.

Another highlight of our 25th anniversary year is that our software developers have completed the new dpiMAX software for our contact angle measuring systems of the OCA series. The modern, entirely redeveloped software represents a breakthrough in user-friendliness and intuitive design.

I hope you enjoy reading our magazine and look forward to working with you for many years to come. Stay with us!

Nils Langer
CEO
DataPhysics Instruments

Content

About us

Milestones of our Company's Success Story 6

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A Clear Vision of Things to Come 10


©Delphotostock - stock.adobe.com

The Application Centre: Where Theory Meets Reality 12


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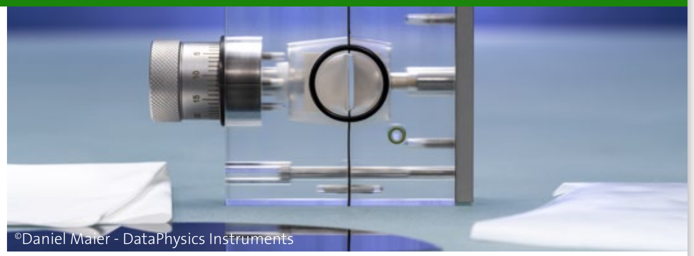
News

IPF Dresden Validates ZPA 20 Zeta Potential Analyzer 16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Changing How You Look at Surfaces 18

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Applications

Use Case: Developing Innovative Tablets Using 3D Printing 22


©Bogdan - stock.adobe.com

Restoring a Perfect Smile with Imaginary Contact Angles 26


©Fizkes - stock.adobe.com

Turning it off and on again: Reversible Hydrogels 30


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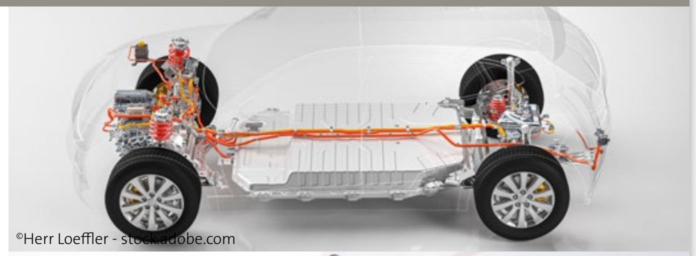
Focus on: Energy Storage Solutions

How Surface Studies Enhance Battery Technology 34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Charged up for the Future 38

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Extras

Book Review: The Spaces in Between 42


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Celebrating the 25th Anniversary of DataPhysics Instruments

Milestones of our Company's Success Story

DataPhysics Instruments began with a team of only seven visionaries. From its headquarters in Filderstadt, Germany, the small company rapidly established itself as a competent partner for science and industry, providing tailored measurement solutions for surface and interfacial phenomena. Today, DataPhysics Instruments has approximately 50 team members and over 3,500 satisfied customers worldwide. To celebrate its 25th anniversary, we review the milestones that were part of this success story.

August 1997

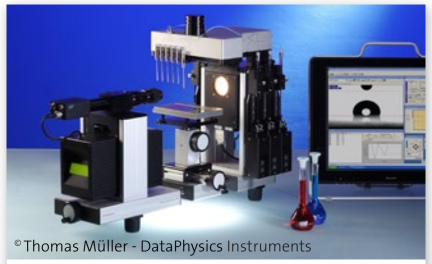
DataPhysics Instruments GmbH is founded in Filderstadt, Germany. The small team of seven visionaries, including its three shareholders, Dr. Ulrich Busch, Torsten Holz, and Horst Rau, begins its journey to becoming a successful and reputable company producing high-quality measuring systems for surface and interfacial phenomena. The small company has its own website from the beginning, which is unusual for the time. To this day, DataPhysics Instruments follows its philosophy of developing and assembling its products locally in Filderstadt, Germany, and distributes its systems worldwide.

©DataPhysics Instruments

The red and grey colour scheme for the DataPhysics Instruments logo is decided early on, but its final design takes several months.

April 1998

During the Analytica expo in Munich, Germany, DataPhysics Instruments presents the OCA 20, its first optical contact angle measuring and contour analysis system of the OCA series, to the world. With its unique modular design and the option to assemble the right solution for every customer, the OCA series is highly valued by partners in research and industry alike.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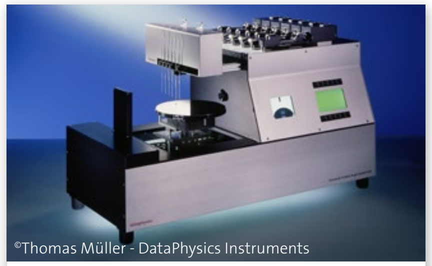
The OCA 20 is a major success for the young company. It sells over 500 units until it is replaced by its successor, the OCA 25, in late 2014.

August 1999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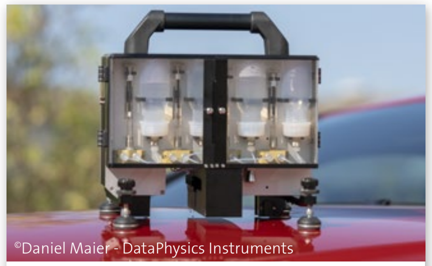
With the ACA 50, the surface energy of, for example, a silicon wafer can be determined fully automatically within a compact instrument design.

March 2000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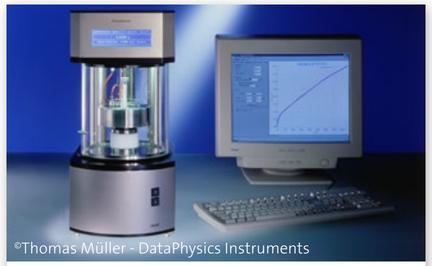
The PCA 100 portable contact angle measuring system is groundbreaking in its design. It enables "on-the-go" contact angle measurements, even on large surface areas.

November 2000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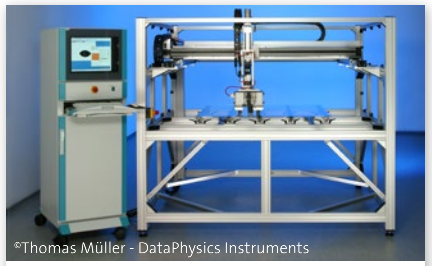
The force-based tensiometers of the DCAT series is the first major enhancement to the portfolio of DataPhysics Instruments. With standard test methods such as the Du Noüy ring method and advanced evaluation capabilities, such as dynamic contact angle measurements, the new devices further strengthen the company's position as a competent and valuable partner for surface and interfacial science applications.

April 2001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Using the portable contact angle measuring system, fully integrated robotic systems are specially designed for companies producing displays.

September 2002


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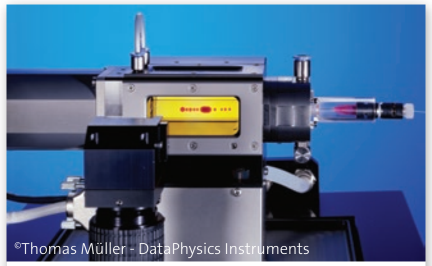
Based on the DCAT series, a special galvanisation simulator is built for a famous German steel producer.

October 2002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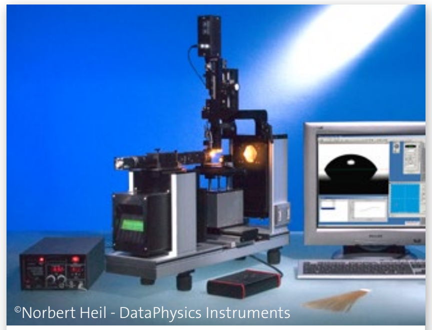
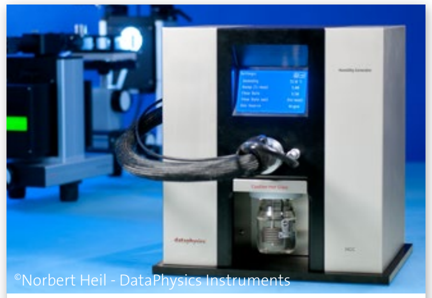
The SVT 20 surpasses what is deemed possible with a spinning drop tensiometer. Due to its high throughput rates, ease of use, and vast automation capabilities, it becomes a major success.

July 2004


©Norbert Heil - DataPhysics Instruments

The OCA 40Micro with a nanolitre or picolitre dosing system enables contact angle measurements on the smallest structures.

March 2012



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The humidity generators and controllers of the HGC series are originally intended to extend the capabilities of DataPhysics Instruments' own devices. However, during development, it becomes evident that the system is easily adaptable to the environmental chambers of other laboratory equipment, such as AFM, DMA, IMC, NIR, TGA, and TMA.

In a joint venture with GÖTTFERT, the Goettfert-Dataphysics Instruments India Pvt. Ltd. is founded as a representative office in India.

January 2014

An additional 500 m² of office space is rented to expand the development department.

March 2014

Dr. Busch retires and sells his shares to DataPhysics Instruments GmbH.

June 2015

©Daniel Maier - DataPhysics Instruments

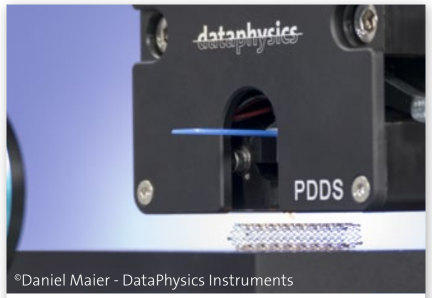
DataPhysics Instruments introduces the MultiScan MS 20 dispersion stability analysis system. The device enables measurements in related but previously unexplored fields of applications.

July 2015

©Norbert Heil - DataPhysics Instruments

A newly equipped training centre with 250 m² of additional floor space opens its doors in Filderstadt, Germany. Talks and product presentations can be hosted in the large seminar room. Additionally, the training centre features a recreation room, where lunch and snacks are available.

February 2016



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The revolutionary picolitre dosing system, PDDS, makes measuring contact angles on tiny surface areas easier and more accessible than ever before.

February 2017

Founder Torsten Holz retires and sells his shares to two new shareholders. The long-term employees Nils Langer and Jens Ole Wund become new managing directors, joining founder Horst Rau.

September 2017

©Daniel Maier - DataPhysics Instruments

A new Application Centre features an additional 250 m² of laboratory and office space to facilitate contract measurements and customer training.

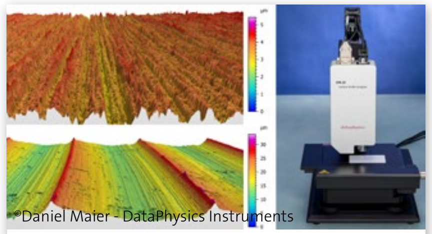
June 2018

DataPhysics Instruments founds a new United States subsidiary company in Charlotte, North Carolina. Bob Fidler, Executive Vice President of DataPhysics Instruments USA Corp., rapidly establishes the new company as a competent local contact for the surface and interfacial measurement needs of customers in the United States and Canada.

August 2018

A completely redesigned website goes online, providing visitors a modern and fast information platform. By moving from a *.de* to a *.com* domain, the site reaches significantly more international visitors than previously.

November 2019



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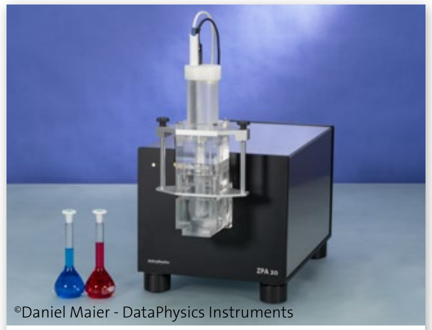
The SPA 25 surface profile analyzer can measure surface topography and roughness parameters with exceptional speed and resolution.

November 2020



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The ZPA 20 zeta potential analyzer again expands the measurement insights DataPhysics Instruments can provide. Its patented measurement technology enables the ZPA 20 to analyse the zeta potential exceptionally quickly.

December 2020

The joint venture with GÖTTFERT ends, and DataPhysics Instruments founds DataPhysics Instruments India Pvt. Ltd. in Faridabad with local partners.

February 2021

Dr. Sebastian Schaubach, Innovation Manager, buys company shares and becomes a fourth shareholder and managing director for DataPhysics Instruments GmbH.

May 2022

DataPhysics Instruments opens a technical office in France, enabling new local colleagues to support French customers more effectively.

June 2022

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DataPhysics Instruments releases dpiMAX, a new and innovative software for the optical contact angle measuring and contour analysis systems of the OCA series.

August 2022

©Barbara Sommer

DataPhysics Instruments GmbH has established itself, after 25 years, as a valued partner for science and industry in all measurement requirements for surface and interfacial phenomena. The small team of seven visionaries has since grown into a respectable small company of approximately 50 team members. The high-quality measuring systems are used by over 3,500 customers worldwide.

Daniel Maier
M.Sc. Physics
Media Designer & Content Manager
DataPhysics Instruments

DataPhysics Instruments' Corporate Vision and its Pillars of Success

A Clear Vision of Things to Come

Founded in 1997, DataPhysics Instruments can already reflect on a long success story. Rather than resting on its laurels, the company will continue taking bold steps into the future. Recently, it has committed its corporate vision to paper. It explicitly states what the company strives to achieve in the future. Additionally, it has spelled out their four pillars of success. With these two strategic tools in its toolbox, DataPhysics Instruments can confidently tackle the challenges of the next 25 years.

Companies generally aim to run their businesses smoothly and sustainably. Long-term business development is often neglected as people aim only for next week's deadline. Moreover, if the long-term strategy is unclear, it is difficult for employees to identify with the company. Additionally, a company's managers could lack guidelines for their decision-making. "If you don't know where you are going, you might wind up someplace else," said Yogi Berra, a famous baseball-catcher and manager. This quotation highlights the importance of identifying a long-term, overarching vision. Such a vision helps a company to focus on its business success.

A strategic, long-term vision can strengthen the team spirit and offers the management team guidelines in their daily decision-making-process.

A corporate vision can also strengthen the team spirit and motivate employees to give their best, even beyond their daily to-do-lists.

A Vision Based on the Company's Unique Selling Points

For a company to be effective, a corporate vision must have a solid basis in the company's unique characteristics. DataPhysics Instruments' vision has been developed based on the management team's close inspection of the company. To develop this vision, the team worked on questions such as, "What is the object and purpose of our enterprise?" and "What kind of company do we strive to be?". Consequently, the team distilled DataPhysics Instruments' vision into five sentences. They express the corporate identity the company strives to achieve in the long term:

"We are a team of future-oriented experts working in a supportive and friendly atmosphere."

The first vision statement shows that DataPhysics Instruments is not a huge, anonymous enterprise. Conversely, due to the manageable size of the company, employees can work together within flat hierarchies. Additionally, the four company shareholders are involved in daily business as managing directors.

"We help our customers to understand interfaces and, on this basis, advance their research and develop the products of tomorrow."

The second vision emphasises that the team has an important mission: with their daily work, they contribute to research and development in the versatile field of surface science. Surface science is faced with exciting challenges which will shape our future. New, innovative materials are designed, and green solutions are explored – to name two use cases in which DataPhysics Instruments' measuring instruments are used.

Corporate Vision of DataPhysics Instruments

We are team of future-oriented experts working in a supportive and friendly atmosphere.

We help our customers to understand interfaces and, on this basis, advance their research and develop the products of tomorrow.

For this purpose, we develop, manufacture, and market innovative solutions for all measuring tasks in the field of surface and interfacial chemistry.

We inspire our customers with our top-quality, advanced measuring systems, which are a pleasure to work with.

Globally, we are recognised as a professional partner in surface and interfacial measuring technology.

"For this purpose, we develop, manufacture, and market innovative solutions for all measuring tasks in the field of surface and interfacial chemistry."

The company strives to provide a full range of appropriate measuring equipment for surface science use cases. Both hardware and software are developed in-house. The devices are manufactured on-site in Filderstadt, Germany, before being sold to customers worldwide.

"We inspire our customers with our top-quality, advanced measuring systems, which are a pleasure to work with."

DataPhysics Instruments' vision also highlights the extraordinarily high standards the company has set itself for the measuring systems it provides. The systems are not only of premium quality, but are valued by their operators for their good performance and intuitive operability.

"Globally, we are recognised as a professional partner in surface and interfacial measuring technology."

This last vision statement shows how customers are regarded as partners, who give important input for new developments. Additionally, customers benefit from the broad experience of DataPhysics Instruments' surface science experts.

DataPhysics Instruments: A Company Built on Firm Ground

Alongside the corporate vision, the management team has identified four pillars of success. These form the basis of DataPhysics Instruments' future developments and are grounded in the company's long-standing core values. The company is proud of these four pillars and committed to continuously strengthening them.

Primarily, DataPhysics Instruments is committed to its excellent personnel. Among other things, this entails a standing invitation for all staff members to participate in training courses and further their education.

Secondly, professional partnerships are important to the company. These partnerships include partners in research institutes and universities around the world, often highly-trained experts in their respective fields as well as industry professionals in many industries such as automotive, coatings and paints, petroleum, and printing.

"If you don't know where you are going, you might wind up someplace else," says Yogi Berra. This is also true for companies in today's fast-paced business world.

Thirdly, DataPhysics Instruments highly values the usability of its products. At the beginning of 2022, the company released dpiMAX, a state-of-the-art software for its OCA contact angle measuring systems. In its development, a user-friendly interface and layout were some of the main goals.

Last but not least, long-term, sustainable corporate development is key to lasting success. At DataPhysics Instruments, this means that all business decisions are made with its corporate vision in mind.

With both its corporate vision and its pillars of success, DataPhysics Instruments is eager move forward together with its customers and will continue to be their **partner in understanding interfaces**.

Dr. Michaela Laupheimer
Dipl. Chemistry
Knowledge Transfer & PR Manager
DataPhysics Instruments

DataPhysics Instruments' Pillars of Success

Excellent personnel: We highly appreciate every single one of our staff members and are proud to be their valued place of work.

Professional partnerships: We engage in professional and cooperative partnerships with our customers and research facilities, as well as with our worldwide sales partners and suppliers.

User-friendly products of highest quality: We provide our customers all over the world with user-friendly products of highest quality standards.

Sustainable corporate development: We continuously develop and improve our company further, always ensuring sustainability in this development.

Services Include Contract Measurements, Trainings, and Development Support

The Application Centre: Where Theory Meets Reality

DataPhysics Instruments' Application Centre is where the manufacturer's devices meet real-life use cases. The scientists working there have four major tasks. They help potential customers understand which devices and methods fit their needs. Additionally, they train customers to make the best use of their devices. They also conduct contract measurements, including measurements under challenging conditions. Finally, they test new product developments under real laboratory conditions to ensure their practicality.

At DataPhysics Instruments it is no secret that every device is only as good as its performance in real-life use cases. Hence, the company owns a fully equipped laboratory – the Application Centre. Dr. Sebastian Schaubach, Chief Innovation Officer and Head of the Application Centre at DataPhysics Instruments, explains why the company values its laboratory so highly, "We are scientists at heart and want to make sure our devices not only look good on paper, but also perform well during their practical application in research labs and development departments."

The scientists working at DataPhysics Instruments Application Centre are highly trained and experienced problem solvers for all challenging use cases arising in real life. They give potential customers insights into the fascinating world of surface science.

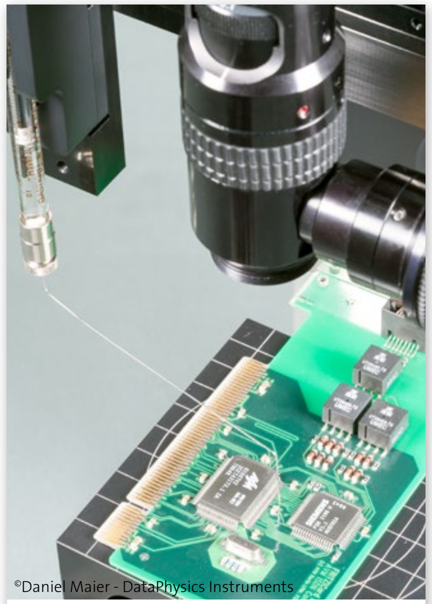
Additionally, they offer customer support and training seminars regarding DataPhysics Instruments' devices. They also conduct contract measurements, for example, when a company wants to outsource its measurement procedures or requires highly specialised devices. Lastly, the scientists also ensure that all DataPhysics Instruments innovations are tested to their limits.

The MultiScan MS 20 dispersion stability analysis system was recently fitted with a handheld scanner to make handling sample containers easier.

Showing Customers What is Possible with the Devices From DataPhysics Instruments

As fascinating as surface science can be, it is not the easiest topic to understand. Determining which device and measurement method are appropriate for a particular use case requires much experience. Potential customers are often unsure which device is suitable for their needs. Schaubach says, "Potential customers want to know if we can solve their individual measurement challenge." Norbert Heil, Senior Application Specialist, adds, "About eighty percent of our devices are sold after an in-depth discussion about possible measurements and methods beforehand." Therefore, the technical consultants in the sales department rely on the knowledge of the Application Centre to answer particularly challenging questions asked by potential customers. The scientists are more than happy to explain and discuss specific use cases with customers on a one-to-one basis.

Schaubach says, "As we believe in deeds rather than words, we encourage customers with particularly difficult measuring tasks to send in one or two samples for trial measurements. This way, the customers can see how a measurement can be done with their specific, often challenging, sample and have a look at the results." If the customer decides to buy a device from DataPhysics Instruments, the initial measurements performed by the Application Centre can be a valuable asset, which can be used to fine-tune later measurement procedures.



With the TVS Topview Video System, contact angle measurements in indentations are possible.

Obtaining the Best From the Device: Tailored Training for Individual Use Cases

The precise and versatile measurement systems from DataPhysics Instruments have been developed to be used intuitively. However, end users sometimes are happy to tap into the expertise of the Swabian company's in-house scientists. This often happens when difficult samples need to be measured or new employees start working for the end user's company. Here, the Application Centre supports customers with in-house and online training sessions. "As the wishes of our customers are very diverse, training sessions are individually tailored to the requirements brought to us by the practical assignments in real-life industrial and scientific applications," says Schaubach.

The scientists train not only the end customers, but also the technical sales consultants in DataPhysics Instruments' distributor network. This ensures the distributors can offer high-level support to customers during the procurement process, device setup and beyond. Schaubach says, "As a small company, we are fortunate to work in close collaboration with local distributors all over the world. The partnerships ensure that customers have a local partner answering their enquiries and questions in their native language. Our partners can also offer prompt help to end users on site."

In addition, DataPhysics Instruments aspires to offer prompt and encompassing services for all their devices. This includes updating devices to the latest software version, as well as fitting the devices with new accessories, if customers decide to widen their measurement portfolio. Schaubach explains, "For us, it is very important to support our devices as long as it is feasible. This is possible because all our devices and their comprehensive software packages are built in modular fashion, which means new measurement methods and system components can be added at any time."

Conducting Contract Measurements to the Highest Standards

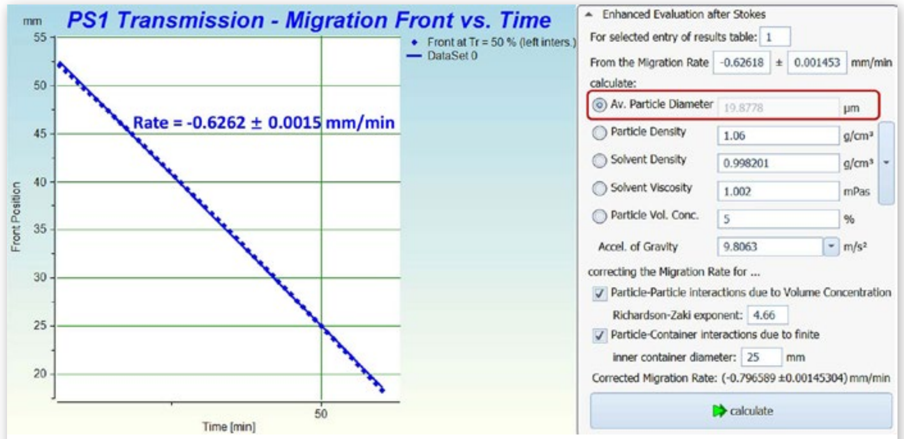
Not all customers consider buying a measurement device outright, as they sometimes do not have the manpower or space to conduct measurements themselves or because their measurement requirements call for highly specialised devices. In such cases, the _Application Centre is happy to function as a laboratory for contract measurements, following various DIN, ISO and ASTM norms relevant to the respective fields. The experts at DataPhysics Instruments can rely on their in-depth knowledge of their devices, which gives them several advantages compared to other contract laboratories.

First, as the company sells its devices in modular bundles, other laboratories might not always be able to offer all possible measurement methods. However, all accessories are available in DataPhysics Instruments' Application Centre.

Second, DataPhysics Instruments always uses the latest models of its devices – sometimes even before they are available on the open market. Customers can therefore be assured that measurements are conducted using state-of-the-art technology. Only recently, for example, the company developed a completely new software called dpiMAX for their contact angle measuring systems of the OCA series, which is already used in its laboratory.

Third, it is easy to customise the devices in the Application Centre to conduct highly specific measurement tasks, as the developers and engineers behind the device only work a few metres down the hallway.

©Dr. Qiongjie Liu - DataPhysics Instruments



Together with the scientists in the Application Centre, the software development team has recently added additional analysis methods to the MSC software package.

DataPhysics Instruments offers a wide range of accessories for its devices, enabling challenging measurements, for example, on the smallest surfaces.

Customers can therefore expect rapid and well-founded solutions to measurement challenges, as the knowledge to implement changes can be easily tapped into.

Fourth, where other laboratories are generalists and offer a large range of measurements, the scientists at DataPhysics Instruments' Application Centre are specialists in chemical and physical phenomena on surfaces and interfaces. Schaubach says, "If customers want to outsource measurements to our Application Centre, we are happy to support them not only with state-of-the-art devices but also with our scientist's expertise."

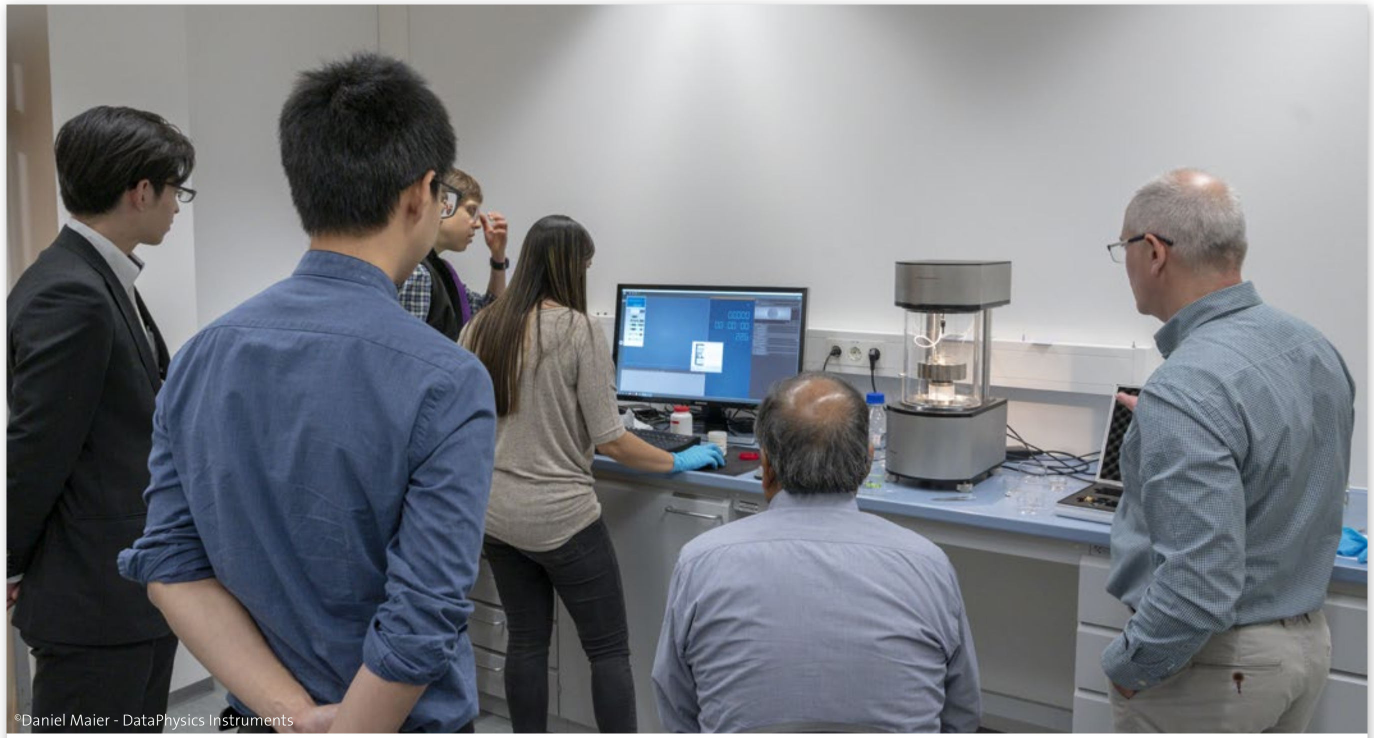
Viewing Things From a Different Perspective

Norbert Heil, who has worked in the Application Centre for over 20 years, recalls a specifically challenging assignment he completed successfully. He says proudly, "I recently helped a customer with quite a tricky measurement that called for very specific equipment and some out-of-the-box-thinking." The customer wanted to measure a tiny, soldered surface hidden inside a tube. No method was available to conduct a contact angle measurement using the traditional camera position, which views the dosed drop from the side. Fortunately, the contact angle measuring systems of the OCA series can be fitted with another camera. The so-called Topview camera can detect and view a drop from above and enables contact angle measurements for drops hidden in indentations or otherwise invisible from the side.

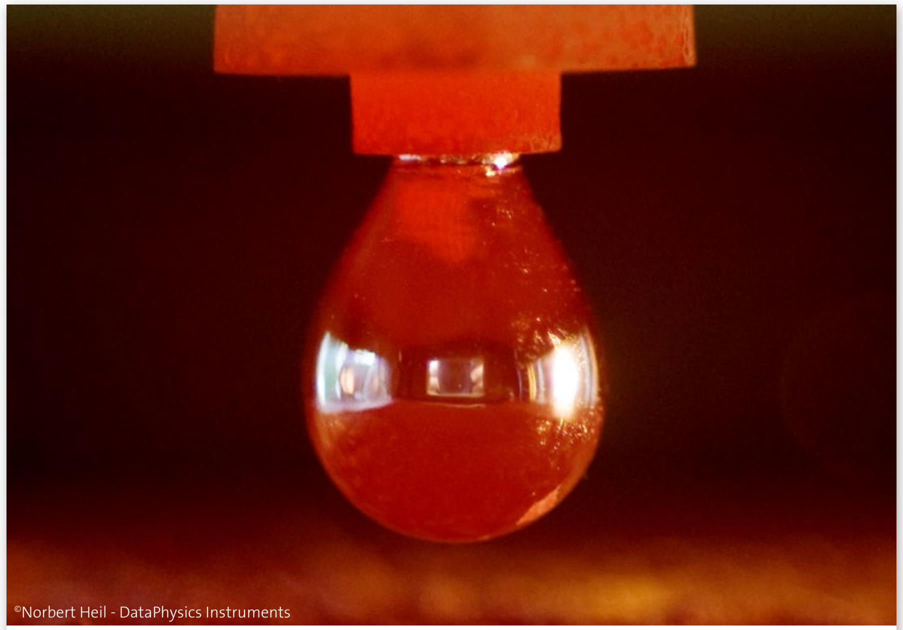
The Topview camera is normally used together with a bespoke liquid-dosing needle so that the view of the drop is not blocked. In this case, the solder's surface area was so small that the drop could only be dosed with the PDDS picolitre dosing unit, which allows drop sizes down to 30 pl. So, Norbert Heil had to find a way to move the PDDS out of the camera's line of sight for the measurement.

Another problem was that drops only a few picolitres in size evaporate very rapidly. Unfortunately, the first liquids Norbert Heil tested had already disappeared when the PDDS had been moved aside. He required a few trials to find a liquid that did not evaporate and allowed the measurement to be taken in time.

Such adjustments "on the fly" demand expert knowledge, which is only achieved with experience and expertise, both of which Norbert Heil could offer. The customer was very happy with the results of this complex measurement set-up.



At regular intervals, DataPhysics Instruments' distribution partners are invited to the Application Centre to learn more about recently developed devices and other updates to software and hardware.



Some use cases call for specialised equipment. In this use case, the surface tension of liquid aluminium was measured at 700 °C.

Testing Upgrades and New Devices in Practical Application

The Application Centre not only supports end users, partners, and technical consultants in the sales department. It also works closely with the company's development engineers. The Application Centre tests all new devices and upgrades in real-life measuring scenarios to ensure that all new software and hardware changes perform well. Schaubach says, "It is especially important to us that all components – of the device itself and in the accompanying software – are particularly user-friendly."

Currently, Product & Application Manager Dr. Qiongjie Liu works closely with the development team to upgrade the software accompanying the MultiScan MS 20 dispersion stability analysis system. Liu says, "We want to make the software simpler and easier to use. Additionally, we constantly add measurement and analysis methods to the portfolio of our devices and their accompanying software." Every change implemented by the developers is tested extensively under different measurement conditions to prove its reliability.

Recently, Liu and the software developers worked together to add a new function to the MultiScan MS 20 software. It is now possible to calculate the mean particle size in a solid-liquid dispersion as well as the mean foam bubble size in a gas-liquid mixture.

However, it is not only the software that attracts the developer's attention. Until the beginning of 2022, the MultiScan MS 20 was only sold with an integrated barcode scanner at the top of the device. Liu explains, "Now, we have added a handheld scanner to the device." This upgrade makes sample handling easier and quicker. Like many other device accessories developed by the company, the handheld scanner can be added as a modular component to the purchase package.

Liu's latest series of improvements cover the temperature range of the MultiScan MS 20. It is now possible to measure dispersions at temperatures down to -10 °C. Liu explains, "A big challenge is that the relative humidity increases drastically when the temperature is lowered, which can lead to condensation. The condensation can stick to the inside of the sample vial and falsify measuring results." This problem quickly solved, another issue arose on the to-do list: "We needed additional insulation; otherwise, the cooling process would have been not as energy efficient as we would have liked," says Liu. Here, too, a solution was found, and Liu could confirm reliable measurements at -10° C.

Practical applications for the widened temperature range include measurements of the shelf life of foodstuff such as milk products or medicinal formulations. "While the upgrade of our temperature range was initiated internally, we already have a first customer interested in measurements at low temperatures," says Liu. However, it is often the scientist's close relationship with DataPhysics Instruments' customers which initiates new development projects. This ensures that the company consistently develops its product portfolio in accordance with market needs.

Where Science Meets Technology

The Application Centre is a versatile department within DataPhysics Instruments. Its scientists explain surface science measurements to potential customers, train existing customers and distributors, conduct contract measurements and support the developers when new software and hardware needs to be tested under real-life conditions.

Schaubach concludes, "The Application Centre is an integral part of our company and at the heart of our relations to internal and external stakeholders." He continues, "One could say that the Application Centre is the main gateway to surface science – the place where theory meets reality."

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Patented Measurement Method Allows Accurate and Rapid Measurements

IPF Dresden Validates ZPA 20 Zeta Potential Analyzer

DataPhysics Instruments from Filderstadt has successfully validated the measurement results of its ZPA 20 zeta potential analyzer at the Leibniz Institute for Polymer Research in Dresden. A long-standing cooperation between the measuring instrument manufacturer and the institute has ensured that the measuring system meets the highest quality requirements. The bidirectional, oscillating measurement method of the ZPA 20 zeta potential analyzer has been patented and allows high-quality data sets in shortest measurement times.

The zeta potential is a measurable quantity that characterises the charge situation at a solid surface in solution. It is measured at the boundary between the ion layer firmly adsorbed on the solid surface and the solution. Dr. Astrid Drechsler, a scientist at the Leibniz Institute for Polymer Research Dresden e.V. (IPF) and an expert in the characterisation of polymer interfaces, explains why the zeta potential is an important parameter, “When a solid surface is submerged in an aqueous solution, it becomes electrically charged due to dissociable surface groups and adsorption of ions and molecules. The zeta potential provides information about these chemical functionalities and charge formation processes. Thus, it can be used to, for example, predict interactions such as adhesion between different surfaces.”

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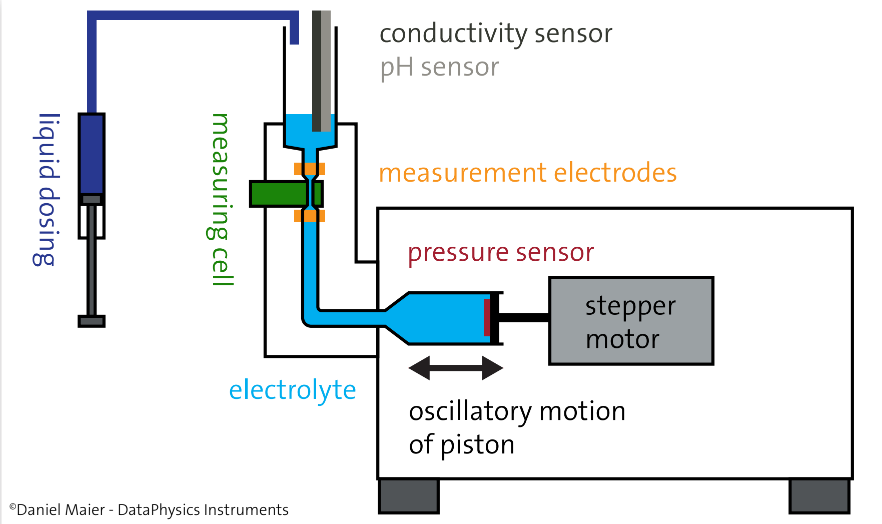
The ZPA 20 zeta potential analyzer uses the patented method of bidirectional, oscillatory streaming potential and streaming current analysis.

The ZPA 20 zeta potential analyzer from DataPhysics Instruments uses the streaming potential and streaming current analysis method. The device can be equipped with the MC-ZPA/S, a measuring cell for plate-shaped solids, and the MC-ZPA/PF, a measuring cell for fibres, powders and granulates.

Zeta potential measurements are important in a broad range of applications, such as the investigation of fuel cell membranes, fouling processes of filter membranes, bacterial growth on food packaging, and washing of textile surfaces. Customers who are unsure whether the ZPA 20 zeta potential analyzer is the right device for them can not only rely on the in-depth knowledge of the DataPhysics Instruments’ sales team, but also request trial measurements with their samples. Dr. Sebastian Schaubach, Chief Innovation Officer at DataPhysics Instruments, says, “Our laboratory offers contract measurements for all possible applications, with the ZPA 20 as well as all other devices of our portfolio.”

Leibniz-Institute for Polymer Research Validates Results

The ZPA 20 zeta potential analyzer had to meet the highest quality standards from the beginning. Thus, the manufacturer DataPhysics Instruments cooperated with a partner with a worldwide reputation in zeta potential measurement: the Leibniz Institute for Polymer Research Dresden e.V. (IPF). As an IPF scientist, Dr. Astrid Drechsler has been dealing with the physical chemistry of polymer surfaces, which includes zeta potential measurements of different kinds of materials. Since 2017, Drechsler and her team have accompanied the development of the ZPA 20 zeta potential analyzer through several project phases. “In the first phase, we validated data measured with the prototype of the zeta potential analyzer,” says Drechsler. A second project phase enabled further development of not only the device but also its measuring cells. “The focus was simplifying the handling of the measuring cells and achieving reproducible sample preparation,” explains Anja Caspari, a chemical laboratory technician at the IPF.



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The measuring method of the ZPA 20: the stepper motor drives the piston, which pumps the electrolyte fluid up through the measuring cell and back down again.

Patented Measuring Method Enables Accurate and Rapid Results

The streaming potential analysis for investigating the zeta potential works as follows: An electrolyte solution is pumped through a measuring cell containing the sample material. Specifically, it is pumped between two flat solid samples (e.g. plates, membranes or foils) or through the gaps in fibre or powder packages. The flow of the electrolyte solution separates ions near the surface and carries them with it. This results in an electrical current or potential difference (or voltage) between the electrodes situated at both sides of the sample. Electrodes measure these differences, depending on the experiment, as streaming current or streaming potential. These values, plotted against the pressure difference in the cell, allow the calculation of the zeta potential.

The ZPA 20 zeta potential analyzer uses a patented measuring method to achieve rapid results with high accuracy. The electrolyte solution is pumped over or through the sample not only in one direction but alternately in opposite directions. Additionally, the flow rate of the electrolyte liquid changes during repeated cycles, resulting in pressure changes. The device records up to 100 pressure values together with the corresponding streaming potential or streaming current in one second. Thus, results with excellent statistical quality are generated within a short time frame. The device software can easily process large amounts of data and evaluate them automatically. Schaubach explains, “The ZPA 20 zeta potential analyzer is the only measuring device on the market today working by measuring the streaming potential in a bidirectional and oscillating fashion.”

Measurement Procedure Reduces Sources of Error

The oscillating measurement creates both positive and negative pressure differences and consequently positive and negative values of the streaming potential or current. This may prevent electrode polarization.

Additionally, the bidirectional flow can reveal asymmetries of the sample surface, such as, for example, inhomogeneous fibre or powder packages or insufficiently fixed flat samples. In this way, error sources can be identified and measurement errors prevented.

Modular And Open Design Approach

During the development of the ZPA 20 zeta potential analyzer, special care was taken to ensure that all surfaces were easy to clean to avoid cross-contamination. This aspect is especially important for obtaining unbiased time- and pH-dependent zeta potential measurements.

Additionally, the ZPA 20 zeta potential analyzer can be expanded with optional modules. For example, with the LDU 25 liquid dosing system, the electrolyte composition can be changed automatically to determine the isoelectric point or adsorption kinetics of surfactants.

“The cooperation with the IPF helped us to tailor the ZPA 20 zeta potential analyzer to the needs of our customers right from the start and to design it with the highest scientific standards in mind,” says Schaubach, “We are especially proud that the patented measurement method renders zeta potential measurements quicker, easier and more reliable than ever before.”

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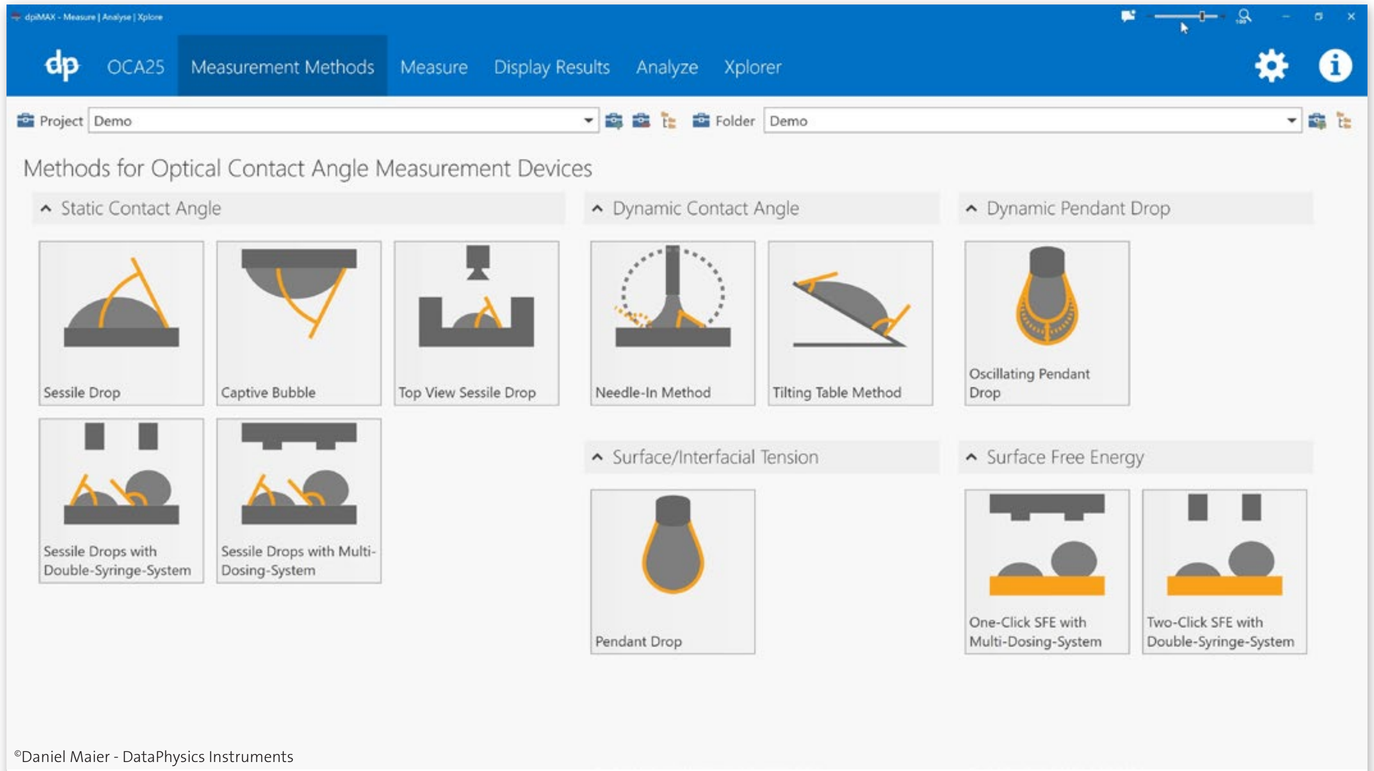
Measuring-Device-Software dpiMAX Now Available

Changing How You Look at Surfaces

The measuring device manufacturer DataPhysics Instruments has developed new, innovative software for the contact angle measuring systems of its OCA series: dpiMAX. The dpiMAX software guides the operator, step by step, through the entire workflow and can therefore be operated intuitively. Additionally, dpiMAX impresses with a well-organised user interface, which facilitates focused work. Furthermore, several smart functions make measuring easy and convenient, especially when an operator needs to perform many repetitive measurements.

The software for professional measuring devices is sometimes challenging to operate. Various parameters must be set accurately to obtain valid measurement results. To achieve this, operators are often confronted with many software settings simultaneously, which makes it difficult to choose the correct configuration. However, this does not have to be the case: measuring devices can be operated easily and intuitively when combined with modern software. The measuring device manufacturer DataPhysics Instruments from Germany confirms this with its latest product release: the dpiMAX software for contact angle measuring systems of its OCA series. The software guides the operator through the workflow, has a well-organised user interface, and contains additional smart features.



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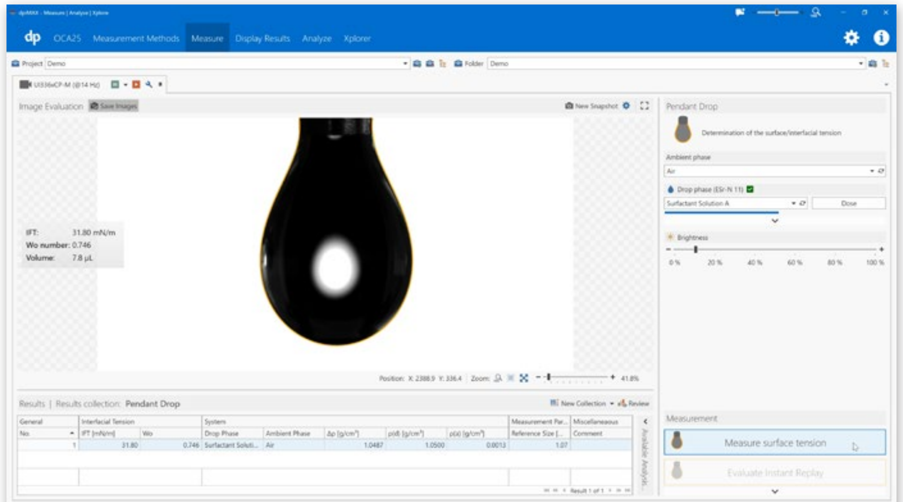
On the well-organised dpiMAX user interface, the different measurement methods can be selected easily and intuitively.

Software Development: Usability as Guiding Principle

Daniel Scholz, Sales Manager and Product Manager for the dpiMAX software at DataPhysics Instruments, explains, “The continuous update of our previous device software has lead, over time, to an expert software with many options to choose from. However, this large selection also made the software more complicated.” Therefore, the company decided to develop a new software altogether. Scholz says, “dpiMAX focuses primarily on easy and straightforward usability. In addition, numerous technical innovations have been implemented so that we can now offer our customers a comfortable state-of-the-art software solution.”

The dpiMAX software was designed using an agile management approach, working according to modern principles, to focus on the needs of the operators as early as possible in the development. This way, the proficient software development team at DataPhysics Instruments built not only on its decades of expertise with measuring device software, but also on state-of-the-art development tools. Scholz evaluates this approach positively. He says, “Our guiding development principle was to ensure simple and intuitive usability, which enables anyone to perform measurements quickly and conveniently. According to feedback from our first customers, we have achieved our goal.”



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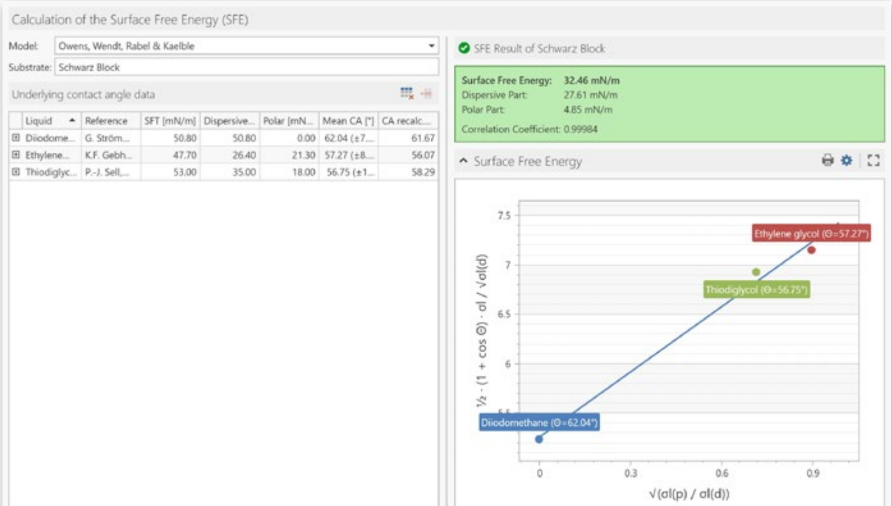
During the measurement, the operator is guided through all the necessary steps via a method-specific control panel (on the right).

The dpiMAX Software Accompanies its Operators Through the Workflow

True to the name-giving motto of dpiMAX – Measure quickly, Analyse easily and Xplore intuitively – the software accompanies its operators step by step from the device settings, through the measurement and evaluation, to the creation of reports and presentation of the measurement results. Thus, beginners are guided comfortably through the entire process. Individually adjusting additional settings remains possible for experienced operators facing complex measurements. For example, this applies to advanced camera settings, which can be used to optimise difficult image situations. In summary, intuitive operation is ensured for every operator, regardless of their experience level.

Well-organised User Interface Facilitates Focused Work

The dpiMAX software supports focused work by displaying only relevant information on the well-organised user interface. For example, during a measurement, the user interface shows the drop image, the results table, and a customised control panel containing only the parameters and control elements required for the selected measurement method. The results are consistently seen at a glance. For example, the contact angle is displayed directly at the three-phase point in the drop image. The surface energy analysis additionally contains an easy-to-read colour code: the colour directly indicates the robustness of the obtained result. Hence, operators can easily remain focused on the essentials due to the well-organised user interface.



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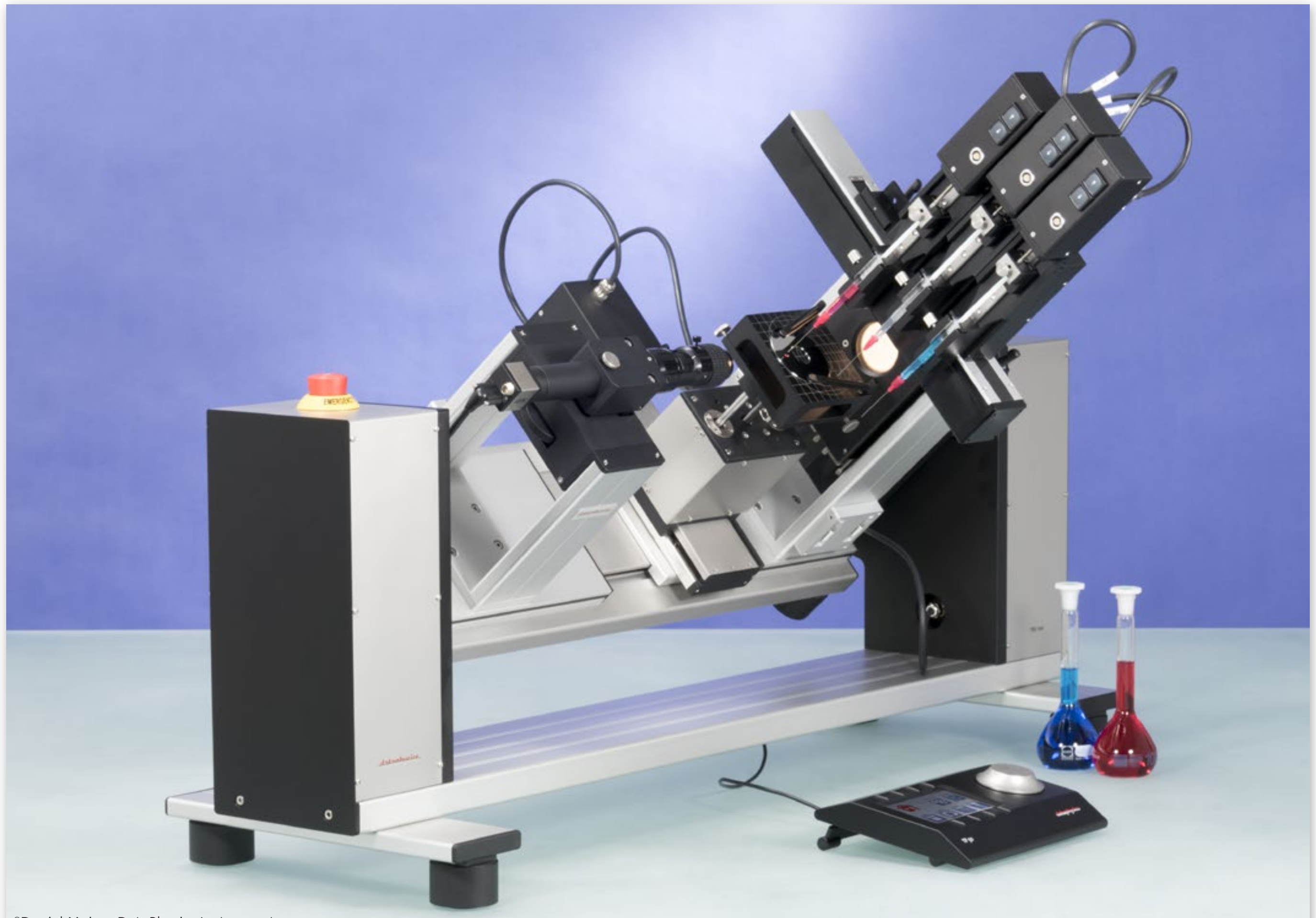
When determining the surface energy of solids, additional values can be easily added for evaluation via drag and drop.

Additional Smart Functions Make Measuring Easy and Convenient

The innovative dpiMAX software not only performs basic tasks, such as controlling the measuring device and executing analysis algorithms, but also supports its operators far beyond such tasks with additional smart functions, which make measuring particularly easy and convenient.

With these smart functions, the drop shape evaluation is performed rapidly: dpiMAX immediately evaluates the surface tension of a hanging drop, or the contact angle of a sessile drop.



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Measurement equipment can be individually assembled to fit a specific use case. For example, using the TBU tilting base unit, a sessile drop can be tilted to measure the drop's receding and advancing contact angle.

The software then directly displays the result in the live camera view. Additionally, videos can be subsequently recorded and evaluated. Even if the operator forgets to start recording, the measurement is not lost due to the Instant-Replay-Function. The dpiMAX software saves the last seconds of the camera image so that it is easy to "rewind" to a specific image situation.

The dpiMAX software also enables predefined processes as simple one-click measurements, which is particularly convenient when operators need to conduct repetitive measurements. Additionally, dpiMAX reminds the operator with smart hints to input necessary information on material specifications and suggests suitable dispensing needles when measuring the interfacial tension.

The dpiMAX software not only offers a host of useful functions, which make measuring and evaluating straightforward and even fun. dpiMAX is also available in several languages, making it easy to use for operators worldwide. Furthermore, operators can set substances as their personal favourites and add substance names commonly used in their laboratory. It is even possible to adapt the user interface of the dpiMAX software to different screen sizes, thanks to an integrated zoom function.

Compatible with All Current OCA Models

The dpiMAX software is compatible with all current contact angle measuring systems of the OCA series from DataPhysics Instruments and is available now. The software is modular in design, so that the methods and analyses required by each operator can be assembled individually.

The experts at DataPhysics Instruments are happy to offer customised packages. Additionally, existing customers with OCA devices can currently buy an upgrade to dpiMAX under favourable conditions.

The dpiMAX software from DataPhysics Instruments supports the realisation of high sample throughputs, investigation of the surface properties of new materials, and development of innovative products. This makes dpiMAX a valuable asset for research laboratories as well as quality and development departments worldwide.

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Interview with Daniel Scholz, dpiMAX Product Owner at DataPhysics Instruments

"Improving Teamwork and Increasing Effectiveness"

Working at DataPhysics Instruments for over ten years, Daniel Scholz knows and understands the needs of the company's customers. This was an excellent premise when he took over responsibility for the product development of the new contact angle measuring system software, dpiMAX. As dpiMAX is developed in an agile way, Scholz fulfils the role of product owner within the scrum team. In this interview he gives an insight into the agile development process and explains its advantages.

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Daniel Scholz
Dipl. Biophysics
dpiMAX Product Owner

***Dr. Michaela Laupheimer:* Can you tell us what a scrum team is and how it differs from a team which follows a classical project management approach?**

Daniel Scholz: A scrum team is a powerful squad that is particularly dedicated to solving complex tasks, like developing a new measuring software. The scrum framework, developed by Ken Schwaber and Jeff Sutherland, specifies that the scrum team consists of a self-organised development team, a product owner, who is responsible for the product's development path, and a scrum master, who helps the team manage the scrum process. According to this process, we have several regular meetings, such as a "Daily" every morning, where we briefly talk about our current tasks. The biggest difference compared with classical development methods is that we work in consecutive, two-week-long "sprints". These sprints always start with a sprint planning meeting, where we agree on our development goals for the next two weeks, and end with a sprint review, where we present the latest version of our software product with the newly implemented features. Finally, we do a sprint retrospective, where we review the effectiveness of the process. The whole system is geared towards improving our teamwork and increasing our effectiveness.

***Laupheimer:* That sounds very well-organised. And what are your personal tasks and duties as the product owner in the team?**

Scholz: As the product owner I act as a link between the development team and the users and stakeholders of the developed software product. This means that I constantly communicate with these groups, collecting user feedback and notes for improvement as well as new ideas for future developments in the dpiMAX software. I summarise all the gathered input in the form of 'user stories', which explain what a user wishes to accomplish and why. Then I discuss those user stories with the development team. Together we determine possible software solutions, whereupon the developers estimate the implementation time and effort of the latter. On this basis, I can prioritise all the ideas for future development steps with regard to their complexity and potential benefit for the users and, hence, shape the roadmap for the upcoming sprints.

***Laupheimer:* I see you have a truly diverse and responsible assignment. What do you personally like best about your product owner job and the agile way of development? And do you maybe see something that could boost your work and the work of your team?**

Scholz: It is great to collect and discuss innovative ideas and then see how, step by step, they become reality. I also like the rapidity of the agile development. We do not work for months or years, to fulfil an initially compiled specification sheet before we finally come up with a software product. Instead, we deliver a new software version every two weeks, after every sprint. In each new version, we implement several new features that effectively expand dpiMAX. So, whenever a great idea comes up, which promises improvement, we have the flexibility to implement it directly within the next sprints. Hence, in just a few weeks, we can present the new solution to our software users and hear their feedback. This feedback, by the way, is always greatly appreciated. Thus, I want to invite everyone to tell us whatever they have to say about the software! We value all feedback, because when we get to know what works well for the users and where there is room for improvements, we have the chance to continuously enhance dpiMAX for everyone.

***Laupheimer:* Thank you very much, Daniel, for these interesting insights into the software development process. I am sure the dpiMAX users greatly appreciate your and your teams' commitment and will be happy to support you by providing feedback.**

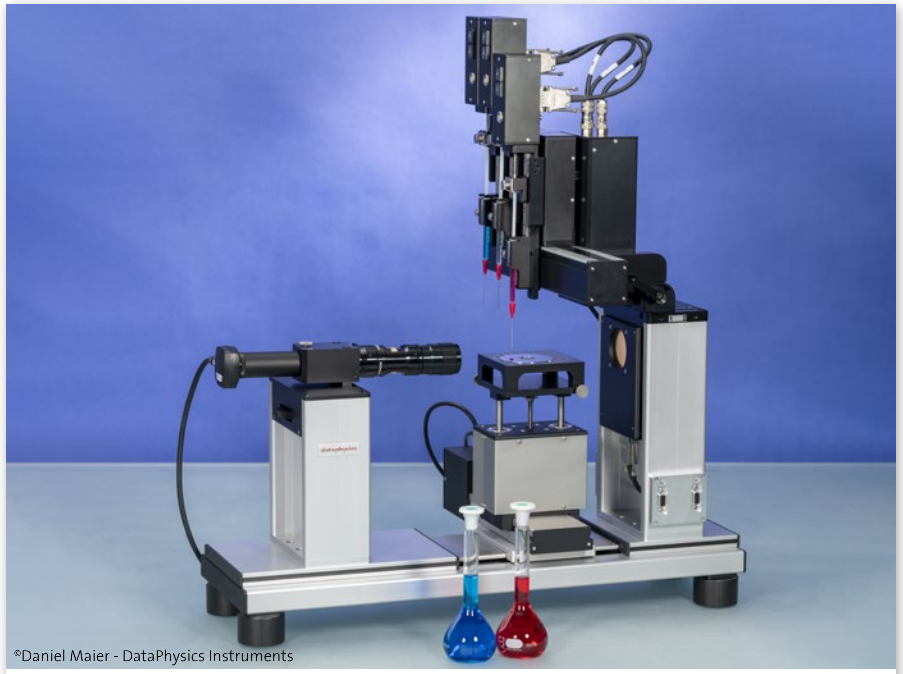
Future of Pharma Manufacturing

Use Case: Developing Innovative Tablets Using 3D Printing

As the pharmaceutical industry steps into the future, companies developing excipient blends for formulating pharmaceutical drugs are investigating 3D printing as a new, innovative way to produce tablets. The technology is especially interesting whenever small batches of tablets are needed, such as in clinical trials. DFE Pharma, one of the largest producers of excipient solutions, believes 3D printing can become a viable way to produce tablets. The company is using an OCA 50EC contact angle measuring system from DataPhysics Instruments to verify the wettability properties of excipient blends.

Pharmaceutical production is a highly regulated field, but at the same time always in need of innovative technologies to keep pace with the rapidly developing demands of modern medicine. While in the past, "blockbuster" drugs were produced in high quantities and given to a wide range of patients, today's drugs are more diverse and tailored to different patient groups.

In clinical trials, too, drugs are only needed in small batches. This presents a challenge to traditional bulk manufacturing, much like special demands regarding drug properties and performance. For example, using traditional methods to combine excipients in a tablet with a high load of the active pharmaceutical ingredient (API) with fast dissolvability is not easily done.



The OCA 50EC contact angle measuring system from DataPhysics Instruments helps to characterise powder wettability.

However, producing tablets using 3D printing represents a promising solution to these challenges.

What 3D Tablet Printing Can Do for Pharma

The basic materials of drugs produced in the form of tablets, capsules, or sachets are so-called excipients, which are mixed with the API. Together, API and excipient determine the drugs' properties, such as their dissolvability, flow, wettability, and compressibility.

DFE Pharma is one such company investigating the advances in 3D printing. The company's 3D printing initiative is headed by Senior Product Developer Korinde van den Heuvel, and she explains the two significant benefits 3D printing can have as an innovative production technique for pharmaceuticals, "For clinical trials, which demand small batches and additionally call for variations in the API concentration for every drug tested, you only need a couple of thousand tablets. Such small quantities are not easily realised with traditional methods but can be produced with 3D printing technology." Moreover, she explains, "Using the same powder blend in a traditional compression machine and in a 3D printer yields different results, as the principle of binding the tablet together differs between approaches. Working with a 3D printer, multiple settings can be tweaked so that tablets with different characteristics are obtained, even with the same initial material mix." Hence, when there is a need for a certain special tablet, which cannot be produced with traditional methods, 3D printing can be the solution.

3D-printed lactose-starch tablets, developed by DFE Pharma

To date, the only fully FDA-approved 3D-printed tablet is the epilepsy drug Spritam, developed by the pharma company Aprecia in the US. Aprecia uses 3D printing to produce Spritam because the technique allows the combination of fast dissolvability and a high drug load (that is, API concentration). "The higher the drug load, the harder it is to create a tablet which falls apart easily. Aprecia has used 3D printing because it was the only way to combine both characteristics," explains van den Heuvel.

With all these benefits in mind, van den Heuvel, an organic chemist by training with over 10 years of experience in the pharma industry, works hard today to produce 3D-printed tablets that will be widely available in tomorrow's world. For this purpose, she and her team are developing excipient-API-powder-blends, which constitute suitable starting materials for 3D-printed tablets.

3D Printing Explained

While several 3D printing methods are available, these methods vary in suitability for printing tablets. The method used to produce Spritam is called "powder bed printing" – the technique also researched by DFE Pharma. "We believe that powder bed printing has the best qualities for tablet production compared to the other 3D printing methods which are available today," says van den Heuvel. Another advantage is that powder bed printing is closely related to an already widely used technique called wet granulation. With powder bed printing, it is even possible to handle thermosensitive APIs. Moreover, powder bed printing is scalable and applicable under real-life conditions.

The technique of powder bed printing resembles building a brick wall, layer by layer. The ingredients are the powder blend, which contains the excipient and APIs, as well as an "ink" (i.e. a liquid which is dosed onto the powder). This liquid is most commonly an ethanol-water-mix. First, a thin layer of the excipient-API-blend is spread out on a flat surface, forming the powder bed. Then, a nozzle sprays small drops of the liquid onto well-defined areas of the powder where the tablets are to be built. Once the drops have solidified, a layer of fresh powder is added. These steps are repeated until the desired tablet height is reached. Finally, the solidified tablets are scooped out of the powder bed.

Important Parameters When Studying Powder Blends

Three parameters are of importance when developing powder blends for 3D printing: the wettability of the powder, its flow, and the consolidation of the printed tablets[1]. While the wettability determines how the powder interacts with the liquid drops, the flow governs how easy it is to spread the powder. Furthermore, the consolidation after 3D printing describes the integrity of the printed tablets when dried.

Van den Heuvel knew from the beginning of her research that wettability was an important parameter to gain additional insights into the usability of different powder blends. Hence, she decided to invest in dedicated measurement equipment for wettability studies.

Methods to Study Powder Wettability

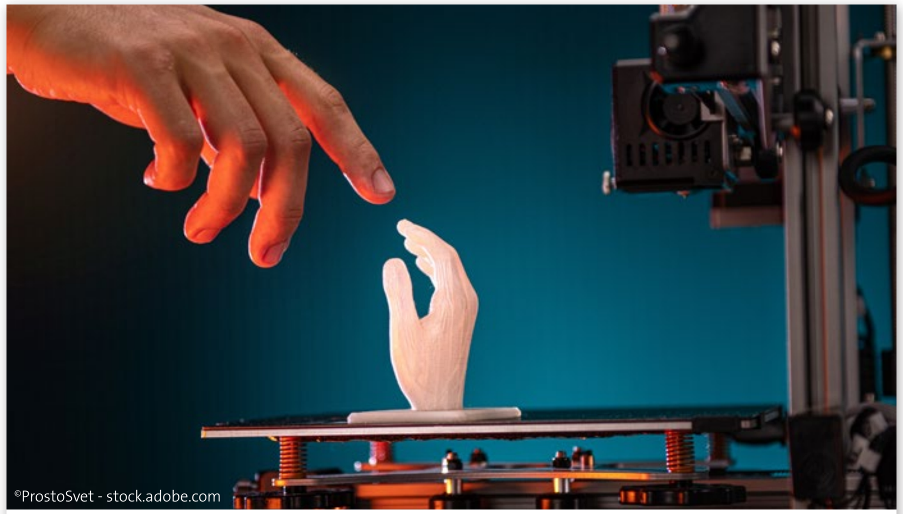
Technical measurement experts from DataPhysics Instruments could offer van den Heuvel two established techniques for studying powder wettability. One possibility is to use a tensiometer and conduct the Washburn method. For this method, the powder is placed in a small cylindrical vessel with a porous bottom, which is fixed to a high-precision weighting system in the top part of the tensiometer. A sample container filled with a liquid is then positioned below the vessel and lifted by the electric sample stage until the liquid touches the bottom of the vessel. Driven by capillary forces, the liquid enters the vessel through the porous bottom and soaks through the powder. As the powder is soaked with the liquid, the weight of the vessel increases. The instrument's software then calculates, based on the rate of weight increase, the liquid's advancing contact angle on the powder as a measure of its wettability.

The second available method does not use a tensiometer but an optical contact angle measuring system. In this case, the powder is spread flatly on a substrate, which is placed on the instrument's sample table. The liquid is placed in a dosing system, such as a syringe, which then doses precisely controlled, single drops of liquid on the powder. The entire process is closely monitored by the high-performance camera of the contact angle measuring system, whose comprehensive software executes a detailed video image evaluation.

"The measurements with the contact angle measuring system mirror the process of powder bed printing," explains van den Heuvel. The system doses liquid drops on a powder bed, just as in a 3D powder bed printer. In addition, the interaction of the liquid drop with the powder bed can be analysed in detail by evaluating the captured video images. DFE Pharma is not alone in its decision to examine drop penetration times for its powder wetting studies. Universities doing research in the field of 3D printing, such as Deakin University, Australia, or the University of Connecticut, US, are also performing drop test experiments for their measurements[2, 3].

Fundamental Studies for 3D Printed Tablets

When van den Heuvel had a contact angle measuring system at her disposal, she and her team started conducting extensive wettability tests on their different excipient-API-blend powders. She explains, "In detail, we are measuring the penetration time (i.e., how long it takes for the drop to be absorbed by the powder once it comes to rest on it)." If a powder absorbs liquid quickly, the blend is suitable for further investigation.



Many believe 3D printing can change how industries produce products in the future, including tablets and implants.

Unsurprisingly, with prior knowledge of 3D printing, van den Heuvel and her team were eager to do more than mere standard measurements with their contact angle measuring system. So, in order to adapt the instrument to their specific measurement needs, they worked with the contact angle measuring system service team to programme it to work as a "prototype printer". "This gives a first indication whether a blend is suitable or not," says van den Heuvel.

In addition to the wettability tests, the flow of the powder is measured. "Only if those preliminary tests for wettability and flow are satisfactory do we start printing tablets in the 3D prototype printer and study their disintegration and hardness," explains van den Heuvel. "Only if the disintegration and hardness of the first printed tablets are satisfactory, we move on to the official testing stage to gain a bigger data set," she says.

On the Journey Towards the Future of Pharmaceutical Manufacturing

DFE Pharma strongly believes in the enormous potential of 3D printing, which has already made a significant impact in many industries. For the company, the innovative technique is a promising route into the future of pharmaceutical manufacturing. Van den Heuvel says, "When I started my position at DFE Pharma, little research had been published on 3D printing in the pharma industry – and even less about specific questions like which excipients to use and which particle sizes are needed for powder bed printing." Van den Heuvel and her colleagues decided to change that: In 2021, they published a paper in the scientific journal *Powder Technology* for which they evaluated different lactose and lactose-starch formulations as excipient blends for 3D printed tablets. The research was conducted together with TNO, the Netherlands Organisation for Applied Scientific Research, which provided the 3D-printing-equipment for the study.

Exploring new ways to tap into the advantages of 3D printing, DFE Pharma is about to embark on an exciting journey. However, the company does not want to go alone. Van den Heuvel says, "We would like to invite other organisations to work with us. We are looking forward to collaborations in the area of 3D printing and are more than happy to supply our partners with advice or customised blends. Other industries have already committed to 3D printing and gained a lot from it. We hope the same will be possible in the pharma industry."

This article is taken from Pharmaceutical Manufacturing and Packing Sourcer, August 2022, pages 40-41. © Samedan Ltd.

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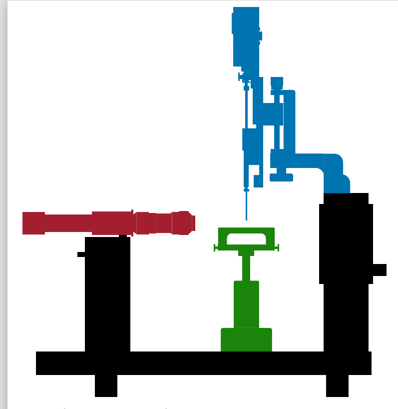
Contact Angle Measuring Systems of the OCA Series in Detail

Optical Measurement Methods Explained

The optical contour analysis and contact angle measuring systems of the OCA series are capable of a wide range of measurements to characterise surfaces and interfaces. This overview explains the most important measurement methods.



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OCA systems combine high-resolution optics (red), exact liquid dosing (blue) and precise sample positioning (green) into powerful measuring systems.

Pendant Drop Method: Measuring Surface and Interfacial Tension

The forces that determine the shape of the pendant drop are in particular its surface tension and gravitation. The surface tension seeks to minimise the surface area and desires the drop to obtain a spherical shape. Gravitation, on the other hand, stretches the drop from this spherical shape and the typical pear-like shape. The surface tension of a liquid and the interfacial tension between two liquids can be determined with every contact angle measuring system of the OCA series from DataPhysics Instruments.

The experimental setup for such measurements is achieved by placing the sample liquid in a syringe, which is mounted on the dosing unit (picture above, blue). The liquid is dosed until the drop hanging at the needle orifice assumes the largest possible pear-shape without falling off. The camera (picture above, red) captures an image of the drop shape. From this shape, the absolute size, and the density difference between the drop phase and the surrounding phase, the device software, dpiMAX, calculates the surface or interfacial tension.

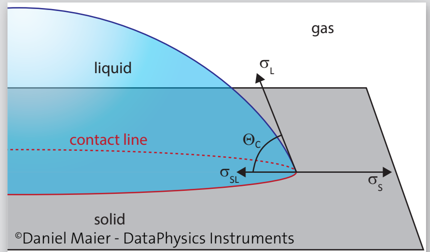
Sessile Drop Method: Measuring Contact Angles of Liquids on Solid Surfaces

With every instrument of the OCA series, it is possible to measure the static contact angle of a liquid on a solid surface (picture below). This is achieved by placing the solid sample on the sample stage (picture above, green). The liquid is filled into a dosing system mounted on the dosing unit. A drop is then dosed on the solid surface. The camera detects the shape of the sessile drop, and the software calculates the contact angle at the three-phase-point between the liquid, solid and gas (often air). In general, low contact angles indicate a good wettability, while higher contact angles indicate a poor wettability. For example, the "Lotus effect" describes surfaces on which water-based liquids display a very high contact angle, which means that drops easily roll off the surface.

Dynamic contact angle measurements refer to techniques which measure the contact angle during movement. One way for measuring dynamic contact angles is the so-called "roll-off"-method, during which the sample or the complete device is tilted. Another way is the "needle-in"-method.

Surface Energy of Solids

The surface energy of a solid describes the "additional" energy available at the solid's surface. The contact angles of at least two different test liquids (with known characteristics) are measured on the solid surface to determine the surface energy experimentally. From the contact angles, the software calculates the surface energy with its polar and dispersive parts. Generally, a higher surface energy indicates better interaction, that is, better wettability of the surface. Additionally, a similar ratio between the polar and dispersive parts of liquids and solids allows more interface interactions, indicating better adhesion between the materials. If a solid has a low surface energy, activating treatments, such as with plasma, can increase the polar part of the surface energy.



The contact angle Θ_C at the solid-liquid-gas contact line is measured optically, based on the shape of a sessile drop.

Work of Adhesion: Liquid-Solid-Interactions

If the liquid's surface tension and the solid's surface energy are known, the dpiMAX software can analyse the work of adhesion, that is, how well a liquid and a solid stick together. Such predictions are particularly useful when searching for a matching liquid-solid-pair or when the liquid-solid-interactions must be optimised.

Oscillating Pendant Drop Method: Viscoelastic Behaviour

The oscillating pendant drop method can be used to determine the viscoelastic behaviour and stress relaxation of a surface, that is, how quickly the molecules react to a change in surface area. This is achieved by suspending a drop from the needle. The drop volume is then increased and decreased repeatedly in oscillating fashion. The camera measures the drop contour and the software can then calculate how quickly the surface active molecules react to the change in the surface area.

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Comparing the Effectiveness of UV-light and Plasma Treatments

Restoring a Perfect Smile with Imaginary Contact Angles

Treating the surfaces of dental implants immediately before implantation optimises their wetting behaviour and therefore their biocompatibility. Most dental implants can be wetted well with water; a behaviour also known as superhydrophilic. In measurements, this is expressed by very small contact angles. The tensiometers of the DCAT series by DataPhysics Instruments can quantify those using the concept of imaginary contact angles. In this use case, the wettability of dental implants, treated with UV-light and plasma, are compared.

The surface-treatment of dental implants, conducted by the dentist shortly before implantation, has attracted increasing attention. It allows optimising the implant's wetting behaviour, which significantly increases its biocompatibility. Studies have shown that the initial attachment of osteoblasts is improved by increasing the surface energy or charging the surface positively, which subsequently leads to increased formation of new bone after implantation. A high surface energy is normally achieved by treating the surfaces of dental implants, giving them a water contact angle of 0° or below. Imaginary contact angle measurements have evolved into a powerful technique to study these superhydrophilic surfaces[1,2]. Tensiometers of the DCAT series from DataPhysics Instruments are the only measuring systems so far featuring reliable and reproducible imaginary contact angle determination in their software. The following use case presents the application of this method to samples of UV- and plasma-treated dental implants.

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The tensiometers of the DCAT series can calculate contact angles even for extremely hydrophilic surfaces, such as the screw threads of dental implants.

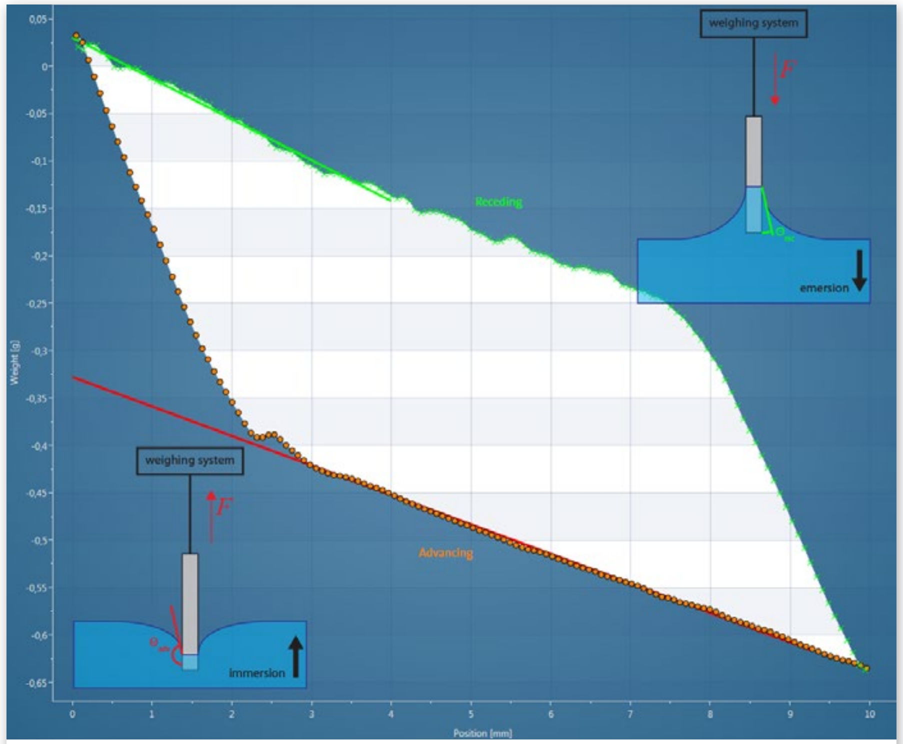
Technique and Method

A tensiometer of the DCAT series from DataPhysics Instruments is a universal measuring system for the force-based study of surface and interfacial parameters. With the software module DCATS 32 and suitable sample holders, it can be used to measure dynamic contact angles on various solids, such as implants, plates, films, powders, fibre bundles, and even single fibres. This is particularly useful for studying hydrophilic samples. When optical contour analysis reaches its limits, one still obtains reliable and accurate results when measuring dynamic contact angles with a DCAT tensiometer due to its precise weighing system.

Dynamic contact angles are measured by fixing the solid sample to a holder. The holder is attached to the device's high precision weighting system, located at the top of the instrument. Then, the solid sample is dipped into and pulled out of a test liquid with a known surface tension σ. The measured weight m of the liquid lamella linking the sample at the contact line of length L is related to the contact angle Θ by equation 1 (Eq 1):

$$\cos\Theta = \frac{m_{(h=0)}g}{L\sigma} \qquad \text{(Eq 1)}$$

where g is Earth's gravitational acceleration of approximately 9.81 m/s². The buoyancy effect of the sample is eliminated by extrapolating the measured weight to an immersion height h = 0 before calculating the advancing contact angle Θ_{adv} or the receding contact angle Θ_{rec} for dipping in and pulling out, respectively.



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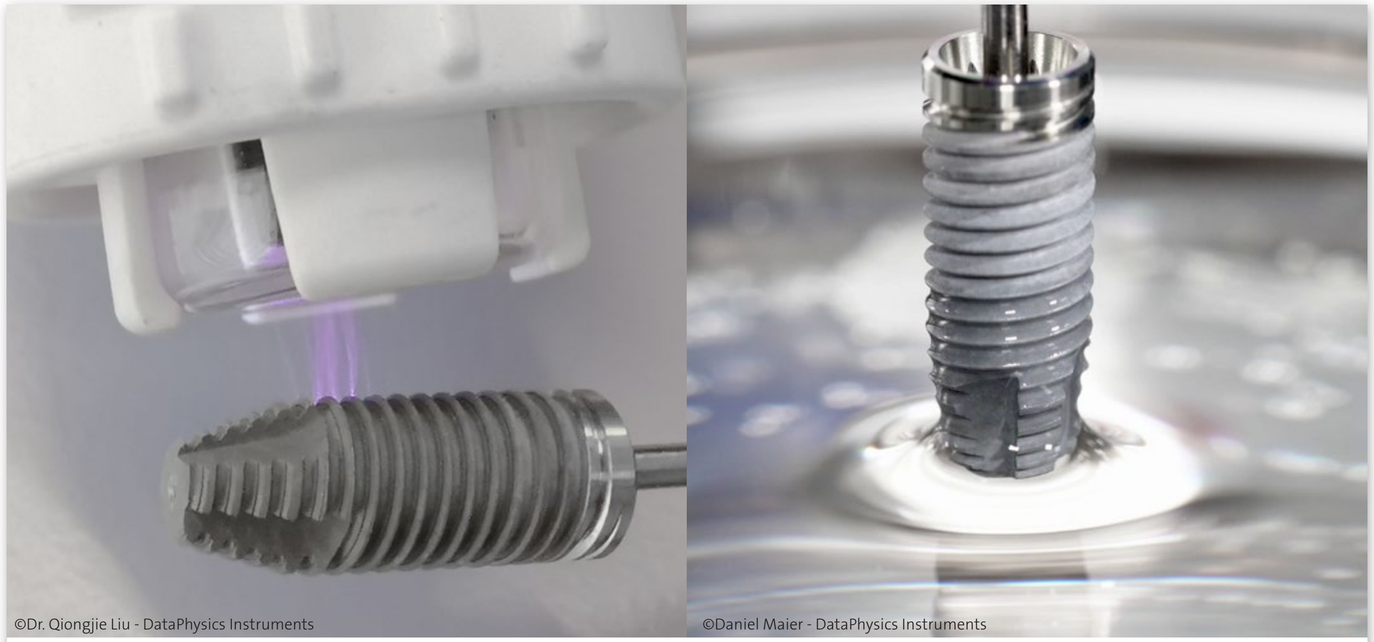
Dynamic contact angles can be measured when the solid sample is dipped in and pulled out of a test liquid. The surface tension of the test liquid must be known.

The fraction in the equation should theoretically not be larger than 1 (for which the contact angle is 0°). However, in practice, measurements of very hydrophilic surfaces do yield values exceeding 1, particularly for rough surfaces where an additional force is generated during wetting by the capillarity of a porous surface[3].

Rather than assigning a contact angle of 0° in all these cases, the DataPhysics Instruments' software calculates the imaginary contact angle, that is, the complex number fulfilling Eq. 1. This opens the possibility of distinguishing between very hydrophilic materials and studying surface treatment techniques such as the UV or plasma treatment of, for example, dental implants.

Experiment

In this application note, the advancing and receding contact angles of six samples were measured using a DCAT 25 tensiometer. Sample 1 was an untreated dental implant manufactured in 2015 (Untreated from 2015). Sample 2 was also an untreated dental implant, but manufactured in 2021 (Untreated from 2021). Sample 3 was a sample treated with plasma for 1 minute, sample 4 was treated with plasma for 2 minutes. Sample 5 was treated with UV-light for 5 minutes, sample 6 treated with UV-light for 12 minutes. Sample 3 to 6 were all manufactured in 2021. The treatment technique and treatment time are shown in the table on the next page.



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The threads of a dental implant are designed to be very hydrophilic to enhance its biocompatibility. Dental implants are treated with plasma or UV-light just before implantation to further strengthen this characteristic.

The measurements were conducted on three identical implants for each sample category 1 to 6. The samples were carefully taken out of their packaging and then analysed without further cleaning. In a preliminary test, the surface tension of the water, which was later used as the known test liquid, was measured using a Wilhelmy plate, ensuring its purity. The measurement yielded a surface tension of 72.8 mN/m, which is consistent with the literature values.

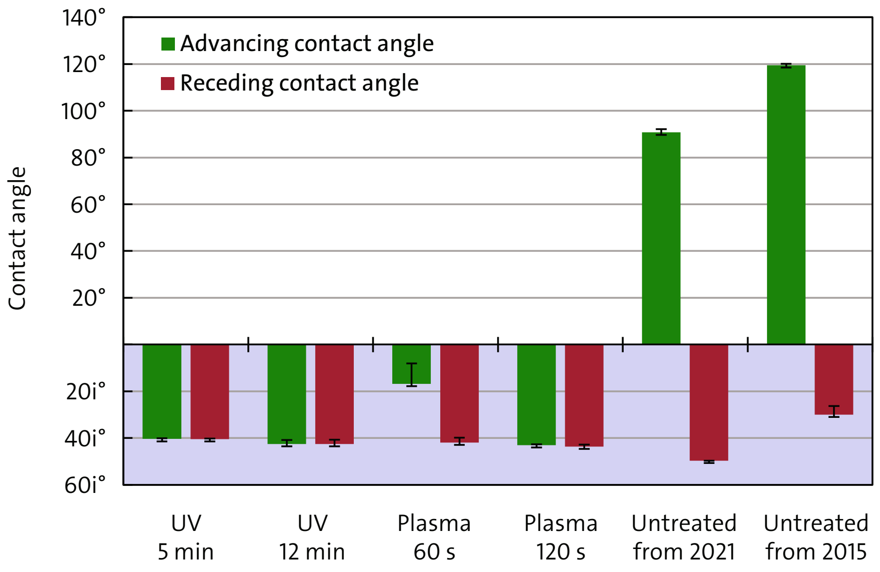
For the dynamic contact angle measurement, an implant sample was attached to the sample holder. The dynamic contact angle method was selected in the software and the sample diameter was entered (4.7 mm). Note that the implants are slightly tapered, the tip being a little smaller than the given diameter. The immersion depth was set to 8 mm. The measurement was then started, and the instrument automatically dipped the sample into the water and pulled it out again, whereafter the software calculated the dynamic contact angles.

Results and Discussion

The graph on the top right of this page shows the advancing (green) and receding (red) contact angles determined for the dental implants. For all implants, only small deviations were found between the repeated measurements, resulting in small error bars. As can be seen in this graph, both advancing and receding contact angles of UV-light and plasma-treated implants show imaginary contact angles, indicating that these two surface treatments result in superhydrophilic surfaces with extremely good wetting behaviour. The data in the same graph shows that the imaginary contact angles on surfaces that have been UV-light-treated for 5 min and

Treatment technique	Treatment time [min]
UV	5
	12
Plasma	1 (3 cycles of 20 s)
	2 (6 cycles of 20 s)

Treatment technique and treatment time



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Dynamic contact angles for differently treated and untreated implants

those treated for 12 min ("the gold standard" of implant treatment) are similar, indicating that the treatment time of 5 min could be sufficient regarding wettability. Conversely, the advancing contact angles on implants plasma-treated for 1 min are smaller than those of implants that have been treated for 2 min, suggesting that the treatment time of 1 min is too short to achieve the best possible wetting.

Interestingly, as shown in the graph on the top right of this page, the advancing contact angles of all untreated implants are contact angles exceeding 90°, indicating the original surface is not hydrophilic and the wetting rate is extremely low. Additionally, the advancing contact angle of the implant from 2021 was smaller than that of the implant from 2015.

Hence, both UV and plasma treatment techniques can improve the wetting behaviour of dental implants and make them superhydrophilic. Additionally, an UV-treatment of 5 min exhibited a similarly good wettability as the gold standard of 12 min treatment. Overall, a 2 min long plasma-treatment is probably a promising candidate to replace the more time-consuming UV-treatment.

Summary

The tensiometers of the DCAT series and their software from DataPhysics Instruments can be used to research surface treatment techniques and treatment times of dental implants reliably and reproducibly. Imaginary contact angles enable an easy study of superhydrophilic materials and, notably, quantitative comparison of the results even in cases where conventional methods would simply yield a contact angle of 0°. Hence, this is an ideal technique for developing and improving surface treatments for superhydrophilicity.

Written in collaboration with De Kliniek voor Tandheelkunde, Netherlands.

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Measurement Devices of the DCAT Series in Detail

Tensiometric Measurement Methods Explained

The dynamic contact angle measuring devices and tensiometers of the DCAT series are versatile measurement instruments that are capable of measuring a wide array of surface and interfacial parameters. This brief overview presents the most common measuring methods and their application in practice.

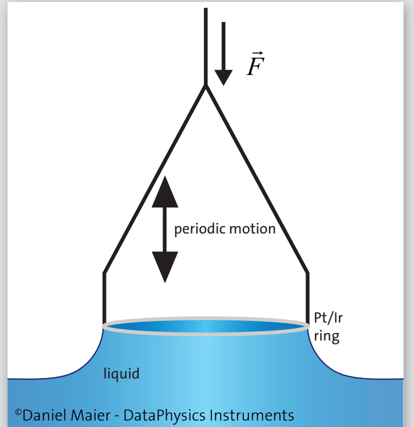
Surface and Interfacial Tension

A tensiometer can be used to measure the surface tension of liquids against air as well as the interfacial tension between two liquids. These measurements can be performed using standard test bodies such as the Du Noüy ring or the Wilhelmy plate. With tensiometers of the DCAT series, such measurements can be done at controlled temperatures between -15 °C and 300 °C and at relative humidities between 5% and 90%.

Measuring Dynamic Contact Angles

Surface tension and interfacial tension are parameters describing liquids. However, one can also use a tensiometer to analyse solid samples. This is achieved by measuring the solid sample's dynamic contact angles against a liquid with known characteristics.

Dynamic contact angles are measured by placing the test liquid in a small container on the sample table of the tensiometer. A solid sample is suspended from a high-precision



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The Du Noüy ring method achieves a high accuracy since it features a large surface area that interacts with the liquid.

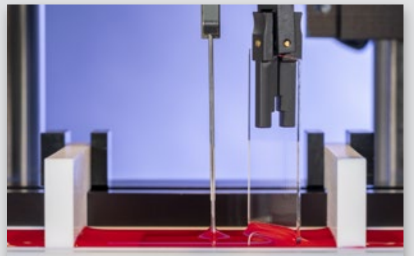
weighting system located in the upper part of the tensiometer. The sample table is raised until the liquid touches the sample. Then, the sample table is slowly lowered. The liquid sticks to the sample surface, and a liquid lamella forms. The tensiometer's weighting system detects the weight change resulting from the lamella formation. The software calculates the dynamic contact angles using the detected weight changes. In detail, it calculates the advancing contact angle (dipping into the liquid) as well as the receding contact angle (pulling out of the liquid).

Even highly hydrophilic solid surfaces can be characterised using imaginary contact angles (see pages 26 to 28). Challenging materials such as single fibres, fibre bundles, granulates, and powders can also be characterised.

Surfactant-Studies: Finding the Critical Micelle Concentration

Surfactants are surface-active substances. When added to a liquid, surfactants adsorb at the interface of this liquid with another liquid, solid, or gas. The surfactant thus lowers the interfacial tension of the mixture. The higher the surfactant concentration, the lower the surface tension – until the critical micelle concentration (CMC) is reached. Afterwards, the surface tension remains constant, even when the concentration is increased further.

A DCAT tensiometer with the LDU 25 liquid dosing unit, for example, enables the surfactant concentration to be changed automatically, and the surface tension can be measured at different concentration values quickly and precisely. The critical micelle concentration is easily determined by plotting the surface tension as a function of the surfactant concentration.



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With the Langmuir trough module it is possible to study monolayers at temperatures up to 90 °C.

Monolayer and Thin Film Studies

Thin films of surface-active molecules at the interface between liquid and air, a solid material, or another liquid can be investigated using a tensiometer together with a Langmuir-trough module. Two barriers on either side can move towards or away from each other. Thus, the reaction of thin films to expanding or compressing the surface area can be quantified.

Adhesion Studies

The force of adhesion is defined as the force needed to pull two materials apart. To measure this parameter experimentally, a drop in a special holder is suspended from the tensiometer's weighting system. A solid, flat sample is placed on the sample table, which is raised until the drop touches the solid surface. The table is then lowered until the drop is pulled off the solid surface. The detected weight change can be used to calculate the force of adhesion. The measurements can be correlated with optical measurements using the additional camera module, UpVideo DCAT. The camera module enables the calculation of the force of adhesion per area unit.

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Stability Study of a Temperature-Responsive Water-Polymer-Mixture

Turning it off and on again: Reversible Hydrogels

Hydrogels are polymer structures dissolved in water. Some of them switch back and forth between a liquid and a gel-like state with a change of ambient conditions, such as temperature or pH. This characteristic makes hydrogels interesting for many applications, for example, in the pharmaceutical or agricultural industries. In this application note, a temperature-sensitive hydrogel is analysed using the MultiScan MS 20 dispersion stability analysis system from DataPhysics Instruments.

Hydrogels consist of hydrophilic polymer structures in water. The polymer chain can bond to water molecules with its functional groups (OH-groups) via hydrogen bonds or bond to itself through “cross-links” between its own functional groups.

If the hydrophilic groups of the polymer chain are bonded with its own functional groups, the hydrogel acts as a two-phase suspension, in which solid polymer chains float as individual particles in the surrounding water. However, when the polymer chains bond with the water molecules, the mixture obtains a gel-like, semi-solid consistency. The change between the two states is often described as gelation or a “sol-gel-transition”, in which “sol” describes the liquid state as colloidal solution, and “gel” the semi-solid state of the mixture. Some hydrogels can repeatedly form and break the bonds with the water molecules and therefore reversibly change between sol and gel state.

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The MultiScan MS 20 dispersion stability analysis system from DataPhysics Instruments can be equipped with six independent sample chambers, called ScanTowers.

The change between liquid and semi-solid state can be induced by a host of external factors. Mixtures react, for example, to changes in pH, electricity, light, ionic concentration, pressure, or temperature. Temperature-sensitive hydrogels have been widely studied for their specific structure and easily tuneable properties. Their excellent temperature-sensitive performance and ability to repeatedly change between the two states are highly valued in various applications, such as drug or pesticide release, cell cultures, protein purification, industrial coatings, and the development of sensors.

In this context, it is essential to study the sol-gel-transition and destabilisation mechanisms occurring during this process. In this article, the reversible sol-gel-transition of a temperature-sensitive pharmaceutical hydrogel is studied, using the MultiScan MS 20 dispersion stability analysis system from DataPhysics Instruments.

Technique and Method

The MultiScan MS 20 dispersion stability analysis system is a measuring device for an automatic optical stability and ageing analyses of liquid dispersions. The MultiScan MS 20 can characterise time- and temperature-dependent destabilisation mechanisms such as creaming, sedimentation, aggregation, and agglomeration. The MultiScan MS 20 comprises a base unit and up to six connected sample chambers, so-called ScanTowers. The ScanTowers of the MultiScan MS 20 can be individually controlled and operated at different temperatures or temperature curves between -10 °C and 80 °C.

With its software, MSC, the MultiScan MS 20 is an ideal partner for stability analyses, since even the slightest

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The sample, a pharmaceutical hydrogel, shows ideal temperature reversibility between 20 °C and 50 °C.

MultiScan MS 20 Dispersion Stability Analysing System in Detail

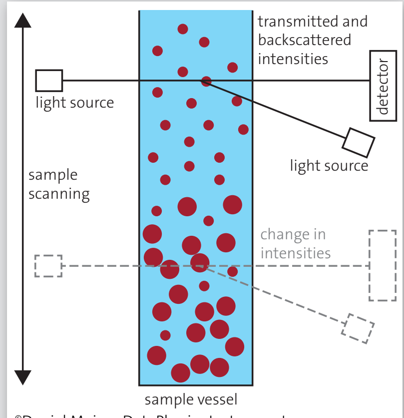
Dispersion Stability Measurements Explained

In this short overview, the measurement principle of the MultiScan MS 20 dispersion stability analysis system is explained in detail. The device can visualise and analyse destabilisation mechanisms such as sedimentation, creaming and clustering by recording the light intensities transmitted through and scattered back by the sample.

Over time, destabilisation mechanisms occur inside dispersions or liquid multicomponent mixtures. Dispersed particles can sediment at the bottom of the sample container due to gravity. They can also cream, meaning lighter particles collect at the top of the liquid. Moreover, phase separation processes can occur due to the interfacial tension between the two phases. Particles can cluster to minimise their interfacial area with the surrounding liquid phase. When the particles cluster together permanently, this process is called agglomeration. If the process is reversible for example by shaking the sample vial, it is called aggregation.

An optical dispersion stability analysis can be used to measure those destabilisation processes. The light transmitted through and scattered back by the sample is captured using two light sources and a detector (see picture). Those so-called transmission (light penetrating through the sample) and backscattering (light reflected by the sample) intensities depend on the number, size and type of dispersed particles or drops. Hence, the recorded intensity changes can indicate as to which destabilisation mechanisms have occurred. A stability analysis tracks these changes over time by measuring the sample repeatedly within set time intervals for a certain duration. For every measurement, the sample is scanned over its entire height, from top to bottom. Hence, local intensity changes in the sample, for example, only at the top or in the middle layer, can be detected.

For practical applications, it is essential to not only receive the transmission and backscattering intensities, but also be able to analyse them. The MSC software, accompanying the MultiScan MS 20 dispersion stability analysis system, offers various ways to analyse a sample. For all analysis methods, the operator must set the position range (from and to which sample height should the analysis occur) as well as the time period (how long should the measurement take overall and how long should the measurement intervals be). After the measurements are complete, the operator can choose between four main analysis methods: “Values”-, “Migration Front”-, “Peak Width”-, as well as “Peak Area”-analysis. Further interpretations, such as according to Mie’s or Stokes’ theory, are possible. Additionally, the stability index for the whole measurement time can be calculated.



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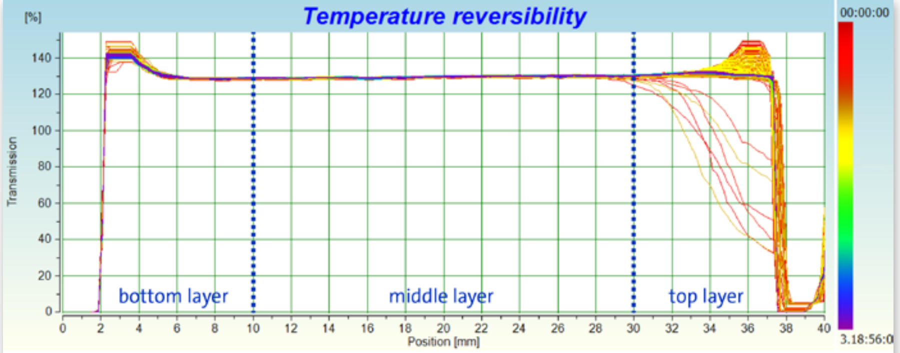
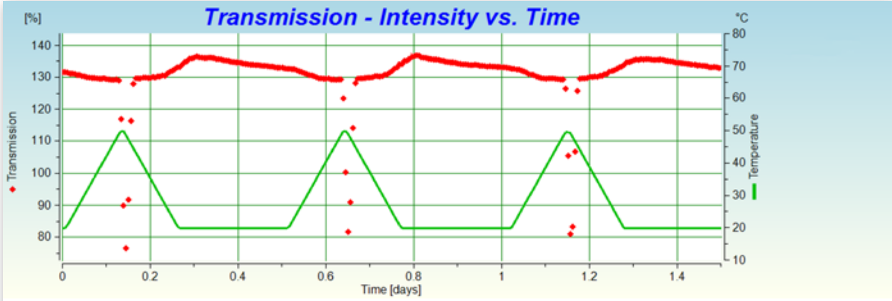
Light transmission and backscattering are measured along the sample height.



Transmission intensity diagram of the hydrogel sample measured during temperature cycles between 20 °C and 50 °C.



Changes in transmission intensity of gel over time (red) and corresponding temperature diagram (green).

changes within dispersions can be detected and evaluated. The MultiScan MS 20 enables rapid and objective analysis of the dispersion stability as well as conclusions on possible destabilisation mechanisms.

Experiment

The material for this experiment was a pharmaceutical hydrogel, which is typically stored at room temperature. For each of the two samples, 20 g of the hydrogel was poured into a transparent glass vial. To ensure that there were no air bubbles in the two samples, the vials were vibrated using an ultrasonic bath and afterwards settled for two hours at room temperature. Then, the measurements were started.

A temperature profile was set using the MSC software. In this profile, the temperature initially increased by 6 °C per hour, from 20 °C to 50 °C, then remained steady at 50 °C for 10 minutes. After that, the temperature decreased from 50 °C to 20 °C at a rate of 6 °C per hour. Subsequently, the sample was kept at a temperature of 20 °C for six hours. The temperature cycle was repeated three times. The samples were scanned every 15 minutes for a total duration of 36 hours. The measured position lay between 0 mm (bottom of the vial) and 57 mm (top of the vial).

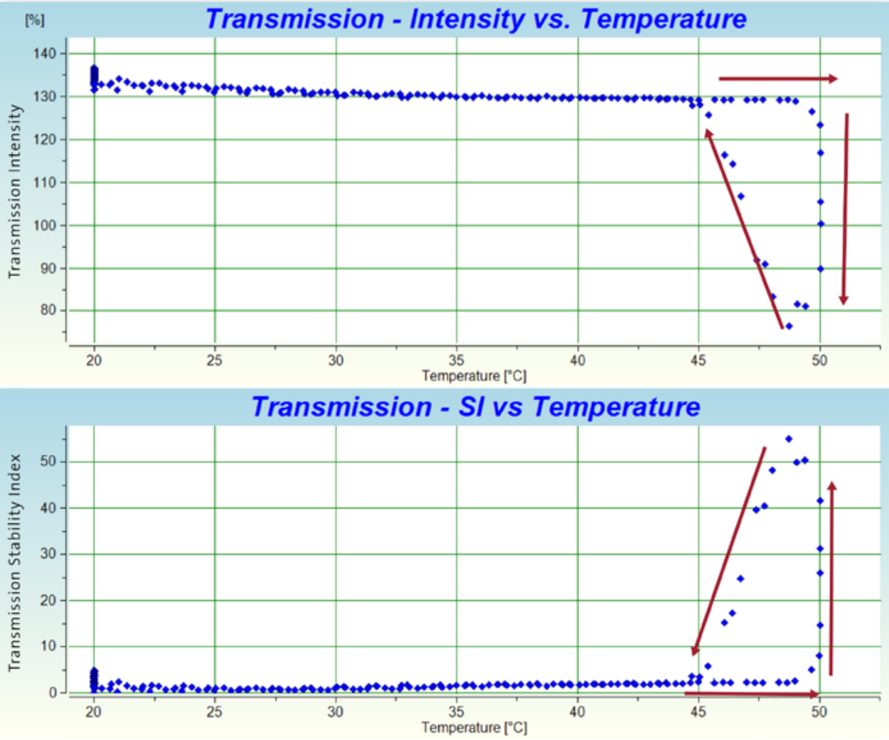
A second experiment was conducted at a constant temperature of T = 50 °C, with scans every five minutes for 22 hours. This second experiment aimed to study possible destabilisation mechanisms at a higher temperature. The picture on page 31 shows the first sample at the end of the first experiment at 20 °C as well as the second sample at the end of the second experiment at a temperature of 50 °C.

Results and Discussion

1. Temperature Reversibility

The picture at the top left on this page shows the transmission intensities against their position in the sample vial containing the first hydrogel sample measured in three cycles between 20 °C and 50 °C. The colour-coding of the curves indicates the time at which they were recorded, from red (start of the experiment, t = 0 s) to purple (end of the experiment, t = 36 h). The transmission diagram shows a clear, time-dependent and position-dependent change in the signal in the top layer between 30 and 39 mm. The MSC software offers various methods to evaluate such changes, for example, using the “values method”.

The changes in transmission intensity (red dots) and the test temperature (green curve) over time are overlaid in the graph on page 32 (middle left). This evaluation can be conducted easily within the MSC software and is one of several overlay graphs which can be generated for further analysis and evaluation. The transmission intensity is relatively constant between 20 °C and 45 °C, indicating that the hydrogel stability is very high in this temperature range. However, the transmission values drop dramatically when the temperature reaches around 50 °C, indicating that the interaction forces between water and the polymer chain are insufficiently strong at higher temperatures, leading to a more liquid sol-gel system, that is, a colloidal system in which individual polymer particles float in water. The transmission returns to its previous values of around 135% when the temperature is reduced to around 45 °C. The hydrogen bonds are reformed between the polymer chain and the water molecules, again resulting in a semi-solid, gel-like state.



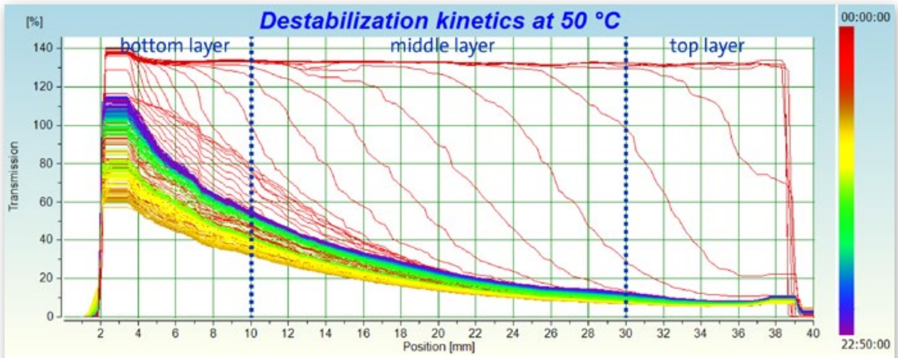
Changes in transmission intensity and transmission stability index (SI) of the hydrogel vs. temperature.

Notably, the transmission change is reversible over all three temperature cycles. It is concluded that the hydrogel exhibits a sensitive temperature response and excellent reversibility.

Consistent with the above results, the changes in transmission intensity and the stability index (SI) over temperature, shown in the graph on page 33 (bottom), confirm that the hydrogel shows temperature-sensitive, full reversibility between 20 °C and 50 °C.

2. Destabilisation Kinetics at 50 °C

The sample stability was studied at 50 °C to obtain an insight into the destabilisation mechanisms of the hydrogel at high temperatures over time. The graph on the top of this page shows that the transmission intensity decreases over time and increases towards the bottom of the vial. This suggests that the hydrogen bonds between the water and polymer molecules are broken at high temperatures and the polymer drops coalesce, leading to a more liquid sol-gel system.



The transmission intensity diagram of the hydrogel at a constant 50 °C shows the destabilisation mechanisms at this higher temperature in detail.

The graph at the bottom of this page shows that the transmission decreases rapidly in the first hour with a change rate of 137.9% per hour, indicating that most of the water molecules are removed from the polymer chains. Additionally, a slow increase of the transmission intensity is exhibited after six hours, suggesting that the coalescence of drops is predominant.

Consistent with the temperature reversibility test, the top layer of the sample is more unstable than the other layers.

Summary

Using the MultiScan MS 20 dispersion stability analysis system and the MSC software, a quantitative and precise way to study the temperature reversibility, phase transition and destabilisation mechanisms of a hydrogel system was demonstrated. Changes can be detected sensitively and allowing the producer to reliably anticipate and quantify stability issues and thus optimise the time and cost of product development.

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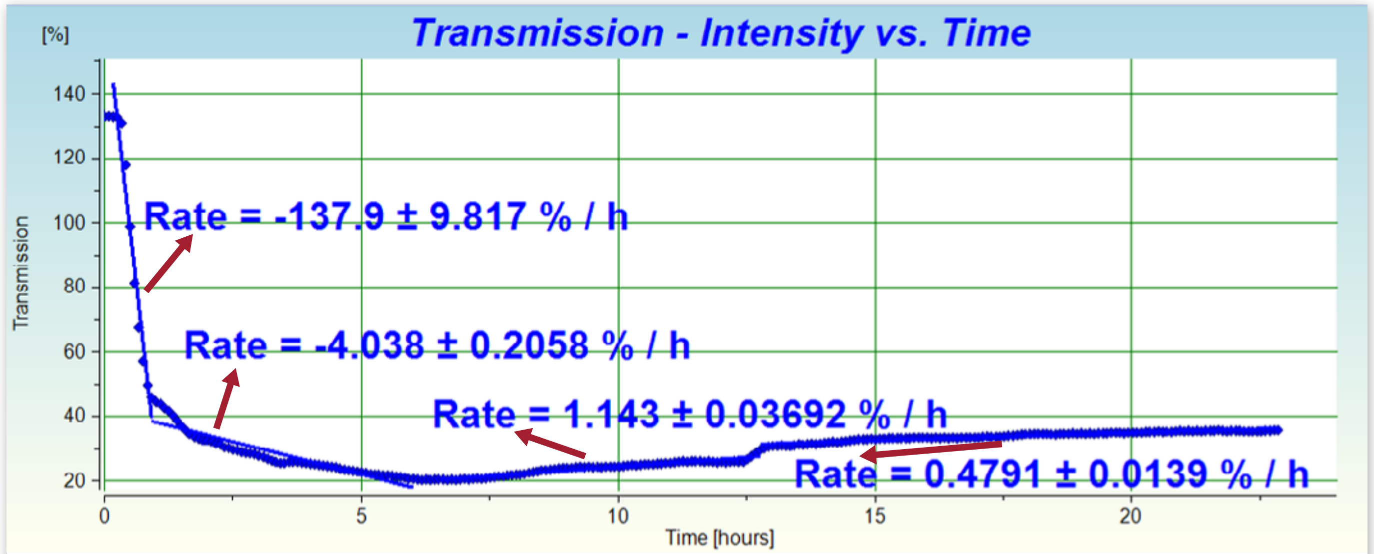
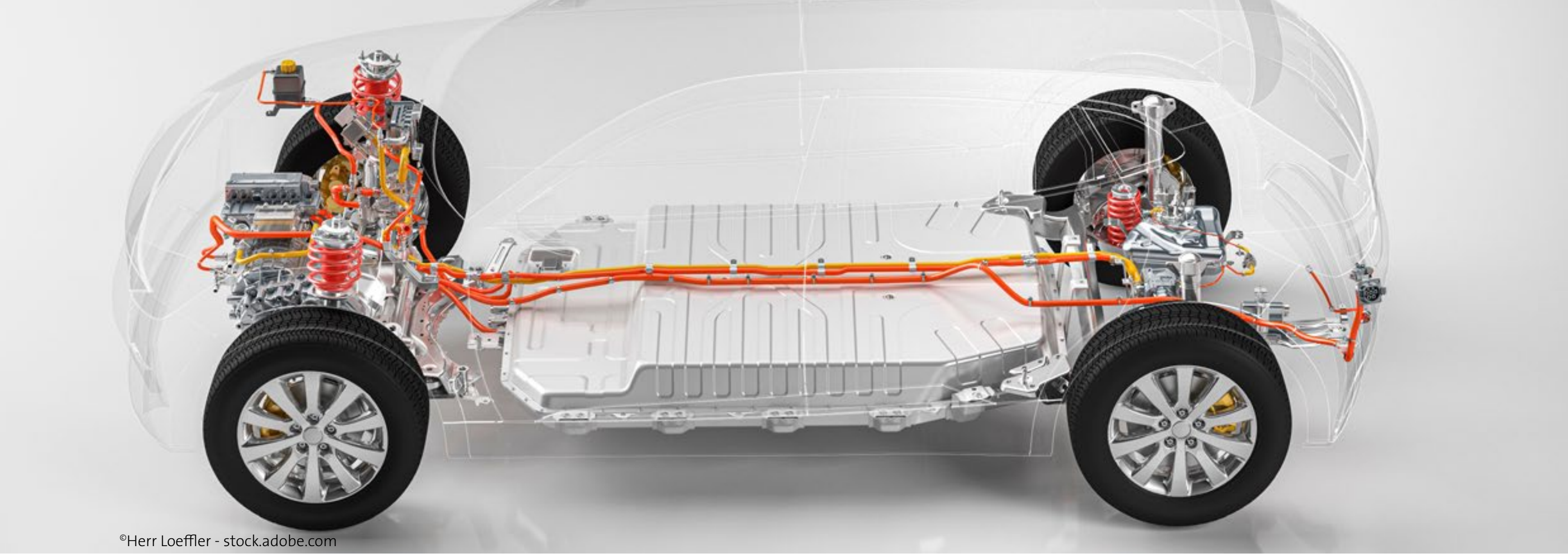


Transmission intensity at position heights between 3 to 35 mm of the hydrogel sample vial vs. time.



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Analysing Surface and Interface Phenomena of Energy Storage Solutions

How Surface Studies Enhance Battery Technology

The energy transition calls for new forms of energy storage solutions. Additionally, consumers demand access to power "on-the-go", from smartphones to electric bikes and cars. Therefore, more and more powerful rechargeable batteries are needed. For good performance, batteries should be light and small with a high energy density. Longevity, fast rechargeability, safety, and low production costs are objectives of today's battery research. In this context, surface and interfacial measurements can provide important insights.

The energy transition aims to replace fossil fuels with low carbon energy sources. Wind power and photovoltaic (PV) systems have long proven to be cost-effective ways to generate renewable energy. Other forms to produce green energy, for example, using the tidal rise and fall of the ocean's surface, are currently being actively investigated.

Making Power Available Anytime

A challenge of the energy transition lies in storing renewable energy for later use. After all, we cannot influence when the wind blows and the sun shines. Energy that is, for example, produced by a PV-system during the day needs to be saved, so it can be used at night when consumers need electricity to turn on their lights. Therefore, energy producers need solutions to store energy and release it to the electric grid when it is needed.

The most mature technology to store energy for later use is offered by pumped hydroelectric energy storage (PHES) plants. When energy sources offer a surplus of energy, these plants use electricity to pump water from a lower to a higher reservoir. When the demand for energy peaks, the plants convert the stored gravitational potential energy back into electricity by driving turbines with the elevated water. While PHES plants have been used for several decades, the energy transition calls for additional energy storage solutions. "Power-to-X"-technologies offer a newer approach. When a surplus of energy is produced, these units convert electricity to gas or heat; if electricity is needed, the units convert the gas or heat back to electrical energy.

PHES plants and "power-to-X"-technologies both offer stationary, central ways to store power. However, mobile and decentralised storage solutions have become interesting in different capacities. Most PV-units sold to end users come with their own energy storage units in the form of a lithium-ion battery. This allows PV unit owners to store the energy they have produced and use it later in a closed circuit without using the electric grid[1]. Such decentralised battery solutions can also be used in parts of the world which do not have a fully developed, centralised electric grid, offering a green alternative to mobile generators, which are often powered with diesel or gasoline.

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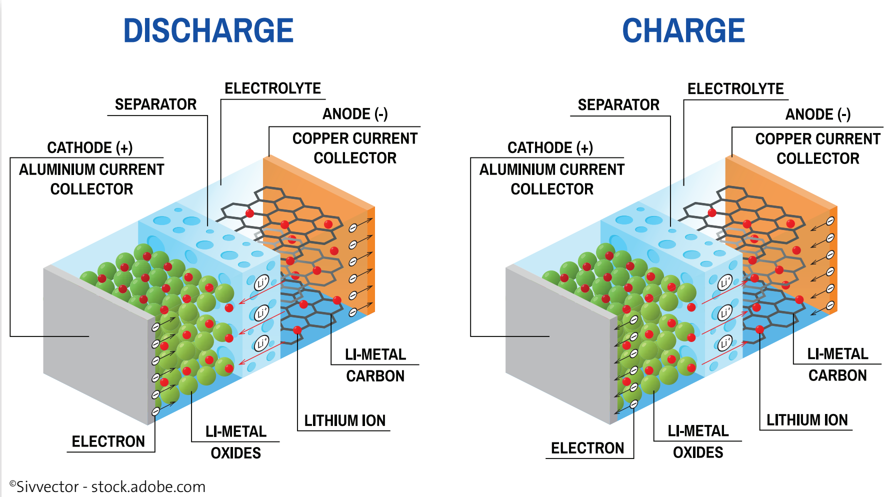
In 1973, the pumped hydroelectric energy storage plant in Ludington, USA, began operation and achieved a power output of 1,872 MW. Its total storage capacity is 19,548 MWh.



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The setup of a lithium-ion battery comprises a current collector, a cathode made from a Li-metal oxide, a separator, an anode made of carbon, and a second current collector. The cathode, anode, and separator are submerged in an electrolyte.

Making Power Available Anywhere

It is not only the energy transition towards intermittent, renewable energy sources that demands new energy storage solutions. Additionally, demand is growing for electric power to be available "on-the-go" for electric mobility solutions as well as mobile electronics.

According to statistics by the Center for Climate and Energy Solutions, 15% of all global man-made greenhouse gas emissions are due to transportation, which is therefore the second largest contributor to gas emissions after electricity and heating[1]. The transition to a new form of mobility takes the form of electric cars on our streets, but also electric scooters and bikes in urban areas, as well as – in a more distant future – hydrogen-powered forklifts, trucks and planes. Most modern electric vehicles use lithium-based batteries.

While electric mobility solutions remain slow to penetrate the market, the sales of mobile consumer electronics are skyrocketing. In 2022, a staggering 91% of the world's population owns a mobile phone[2]. Most smartphones and other portable consumer electronics rely on lithium-ion batteries to power them on the go, as well.

How Lithium-Ion Batteries Work

Different types of lithium-based batteries are available or currently being developed. All lithium-based batteries comprise two electrodes, the anode and cathode; two current collectors; an electrolyte; and a separator. A schematic setup of the components is shown in the graph above.

The current collectors are connected to the electrodes. The two electrodes and the space between them are filled with the electrolyte and the separator ensures that the electrodes cannot make physical contact. The main differentiating factors between lithium batteries are their electrode and electrolyte material.

Lithium batteries are based on the intercalation of lithium ions into a layered material. Intercalation is a reversible process during which the lithium ions enter a layered material and become part of it while retaining the crystal structure of its host material. Hence, the lithium ions can change the charge of an electrode made of a suitable layered material. The first commercially viable lithium-ion battery was developed in 1985 by Akira Yoshino, who received the Nobel Prize for Chemistry in 2019, together with two colleagues[3]. The anode was made from petroleum coke and the cathode was made from lithium cobalt oxide.

Once an electric load, such as an electric motor or a phone, is connected to the collectors, the lithium battery discharges. During this process, the lithium ions stored in the anode are released into the electrolyte. The excess electrons are collected by the collector and can drive the electronic device. Simultaneously, the lithium ions travel inside the electrolyte through the separator and into the cathode material, where they meet with the electrons that travelled through the load. This process stops when the load is disconnected, that is, when the connected device is turned off, or when all lithium ions have moved from the anode to the cathode and the battery is depleted.

Since the process of intercalation is reversible, the battery can be recharged. When the battery is connected to an external power source, such as a phone charger, electrons are driven from the cathode to the anode. Inside the battery, the positively charged lithium ions are released by the cathode and migrate through the electrolyte across the separator into the anode to rejoin the electrons. Once all lithium ions are inside the anode, the battery is fully charged.

It is crucial to ensure optimal contact and adhesion between the battery components to reach the highest battery performance. Important interfaces to consider are situated between the electrodes and current collectors, the electrolyte and separator, as well as the electrolyte and electrodes. Surface and interface studies enable the investigation of adhesion and wetting behaviour of battery components to optimise their materials. For this purpose, several experimental techniques are available.

Measuring the Surface Tension of a Liquid Electrolyte

The laboratory measuring systems from DataPhysics Instruments can offer two different methods to measure a liquid electrolyte's surface tension.

The first method uses a force-based tensiometer of the DCAT series. The Wilhelmy plate and the Du Noüy ring method both yield the liquid's surface tension. The Du Noüy ring method is particularly well suited for low surface tensions, as desired for battery electrolytes.

Alternatively, the surface tension can be determined with an contact angle


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With a DCAT tensiometer, the surface tension of an electrolyte solution can be easily determined. Together with a Peltier temperature control unit, the sample vessel can be tempered from -15 to 135 °C.

measuring system of the OCA series. The so-called pendant drop method evaluates the surface tension based on the shape of a liquid drop hanging from a dosing needle.

Measuring Contact Angles on Electrodes and Separator

The OCA contact angle measuring systems are also suitable for studying the electrodes' and separator's wettability with the electrolyte solution by measuring the contact angles of sessile drops. This has been done, for example, by Sun et al. from the China Automotive Battery Research Institute in Beijing, who developed a novel tri-layer separator[4]. They compared contact angles measured on their newly developed separator to contact angles measured on common separator material and thus confirmed an advanced wettability, besides other superior properties.

Alternative methods for contact angle measurement are the Wilhelmy plate method and the Washburn method, which can both be employed using a DCAT tensiometer.

In the first case, a sample of the solid electrode material is dipped into and pulled out of the electrolyte solution, yielding the advancing and receding contact angle.

For the Washburn method, a small container with a perforated bottom is filled with a sample of porous electrode material. When brought in contact with the electrolyte solution, the electrolyte solution is absorbed due to capillary forces, and the advancing contact angle is calculated by the device's software.

Studying the Absorption and Penetration Rate

If the electrolyte wets a separator or electrode material so well that it spreads on the surface, or if the electrolyte is absorbed by a porous electrode material, videos captured with the high-speed camera of the OCA contact angle measuring system allow these processes to be studied in detail. Hence, for instance, the absorption rate can be determined. The absorption rate is a crucial factor in the manufacturing process, as the porous electrodes must be fully saturated with the electrolyte.

Studies analysing the penetration rate of an electrolyte have already been conducted in practice by researchers from the Leibniz Institute for Polymer Research and the Fraunhofer Institute for Ceramic Technologies and Systems in Dresden, Germany[5]. They used a DCAT tensiometer from DataPhysics Instruments and the modified Washburn method to study the rate of electrolyte penetration into a model battery cell. The aim was to optimise the time efficiency of battery production lines.


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With an OCA contact angle measuring system, the wetting behaviour can be analysed.

Studying the Stability of Battery Slurries

The current collectors are prepared with so-called "electrode slurries" to produce the electrodes. These slurries are complex and highly viscous suspensions containing electrochemically active materials, conducting additives, and polymeric binders, in the form of solid particles of different sizes and shapes. For good battery performance, it is essential to ensure that the electrode has a homogeneous composition and that the current collector is completely covered with the electrode slurry. In this regard, it can be useful to conduct contact angle measurements of the electrode slurries on the current collectors using the experimental methods explained above.

Moreover, it is advisable to ensure that the electrode slurry remains uniformly mixed, at least until it is spread and dry-fixed on the current collectors. Hence, it is beneficial to study the electrode slurry's stability with the MultiScan MS 20 dispersion stability analysis system from DataPhysics Instruments. The MultiScan MS 20 yields quantitative results revealing separation mechanisms occurring in a sample, as the article "Stability Study of Battery Coating Slurries", on page 38, explains. Product developers can then optimise the slurry's stability by, for example, varying type or concentration of its additives and binders.

Surface Science Studies for Better Batteries

There are many surface and interfacial measurements which can help enhancing the performance of batteries and other e-mobilty solutions, such as fuel cells. DataPhysics Instruments is happy to provide the appropriate measuring equipment and be part of the journey into a greener future.

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How Measuring Systems from DataPhysics Instruments Can Improve Fuel Cell Efficiency

Surface Science Studies for Fuel Cells

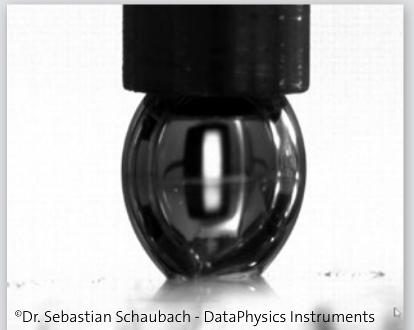
Fuel cell vehicles promise to be a green alternative to conventional cars. Similar to batteries, the interaction between fuel cell components is important, as it has an influence on the fuel cell performance. Researchers have used OCA contact angle measuring systems as well as DCAT tensiometers in their quest to find the perfect materials for efficient fuel cells.

Fuel cell vehicles are powered by an electric motor, just like electric battery vehicles. However, the required electricity is not stored in batteries, but generated in on-board fuel cells. The required fuel, such as hydrogen, is stored in the vehicle and can be quickly refilled, without having to charge the battery for a long time. Therefore, as a promising green mobility candidate, fuel cells are the focus of numerous research activities aimed at improving their efficiency, safety, and affordability.

Fuel cells are electrochemical cells in which the chemical energy of the fuel is converted to electrical energy, which can then, for example, power an electric motor. The cells contain an anode, where a catalyst oxidises the fuel and an adjacent electrolyte through which the generated ions migrate to the cathode. There, they recombine with the electrons and react with another chemical to form a certain "waste product". The fuel is commonly hydrogen, which reacts at the cathode, while oxygen from the air reacts at the anode, so that water is the only "waste product" besides heat.

The contact of the electrolyte with the other fuel cell components is highly relevant in fuel cell electric cars, too. Hence, Halter et al. studied how porous materials in high-temperature fuel cells are wetted by a phosphoric acid electrolyte[6]. For this purpose, they conducted contact angle measurements at temperatures between room temperature and 160 °C, using an OCA contact angle measuring system equipped with an electrical temperature control unit, TEC 400, from DataPhysics Instruments.

Another exciting study in the field of fuel cells was published by Lu et al. from the University of Chemical Technology in Beijing, China[7]. They focused on fuel cells using liquids, such as hydrazine, as fuel. An issue with this technology is finding an effective catalyst on the electrode, where gaseous products are generated. If these gases are not released sufficiently quickly, they block the catalyst site and cause high pressure in the liquid flow area, limiting the fuel crossover. Hence, Lu et al. propose to work with nanostructured copper electrodes, which possess "superaerophobic" properties. They confirmed the effectiveness of such electrodes by measuring the force of adhesion of air bubbles on them in water. This was feasible with a DCAT tensiometer from DataPhysics Instruments using an optional video module.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The pull-off-process of a gas bubble from a surface can be evaluated in terms of its exerted force and optically with an optional video module.

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A Stability Study of Battery Coating Slurries

Charged up for the Future

The stability of coating slurries used to produce anodes and cathodes of batteries was tested using the MultiScan MS 20 dispersion stability analysis system, developed by DataPhysics Instruments. By analysing the time- and position-dependent backscattering intensity values of different slurries, it was possible to identify unstable slurry formulations within a short period of time. Studies, such as the one conducted here, provide valuable information for developing and optimising battery slurries.

Electromobility is a key aspect of climate-friendly transportation. Electric vehicles can make the environment cleaner and improve people's quality of life, especially in urban areas. The success of electric vehicles depends on the availability of efficient and affordable battery systems; thus, companies are devoting tremendous effort to improve electric batteries[1].

Lithium batteries are the most widely used battery systems in the electromobility industry[2]. Their electrodes are made from multicomponent mixtures manufactured from dispersions of microscale or nanoscale powders in viscous polymer solutions. These so-called "coating slurries" contain a high percentage of solid particles differing in their composition, size and shape. Guaranteeing the production of coating slurries of consistent quality requires them to be homogeneous and stable in terms of the distribution of their ingredients. When developing coating slurries, it is therefore essential to study the stability of the dispersion used in the process. This is challenging, because the separation mechanisms within the dispersion are often invisible to the naked eye for a long time. Consequently, technical tools are crucial for a rapid product development.

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The MultiScan MS 20 dispersion stability analysis system from DataPhysics Instruments can be equipped with six independent sample chambers, called ScanTowers.

The MultiScan MS 20 dispersion stability analysis system from DataPhysics Instruments (see picture at the bottom left of this page), with its MSC software, is ideal for conducting thorough stability investigations required when developing battery slurries. The MultiScan MS 20 can detect even the slightest changes within dispersions, enabling rapid and objective evaluation of separation processes. Every sample chamber, also known as ScanTower, contains a scanning unit, comprising two light sources for transmission and backscattering signals plus a light detector unit. The scanning unit moves up and down the sample container, scanning it at individually defined time intervals. This enables position- and time-resolved detection of both transmission and backscattering light intensities. The results are presented in the MSC software as multiple intensity profiles in intensity vs. position diagrams (see graphs on pages 39 and 40). A study of two different nanoparticle-based battery coating slurries is presented in this article.

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Battery coating slurry 1 in its sample container after the measurements

Experiment

In the experiment described here, the stability of two battery coating slurries with different formulations was investigated. For this purpose, 20 ml of each slurry were poured into a standard sample container, which was then placed into one of the ScanTowers. The temperature inside both ScanTowers used in this experiment was defined as t = 25 °C. However, different temperatures and temperature curves can be set for each ScanTower if required for a particular experiment.

A measurement routine was then set up. It scheduled scans of both samples (slurry 1 and slurry 2) every 5 minutes for a total measuring time of 26 hours (slurry 1) and 120 hours (slurry 2). The scanned height was between 0 mm (bottom of the sample container) and 50 mm (top of the sample container). The picture at the top of this page shows the sample container filled with battery coating slurry 1 at the end of the experiment.

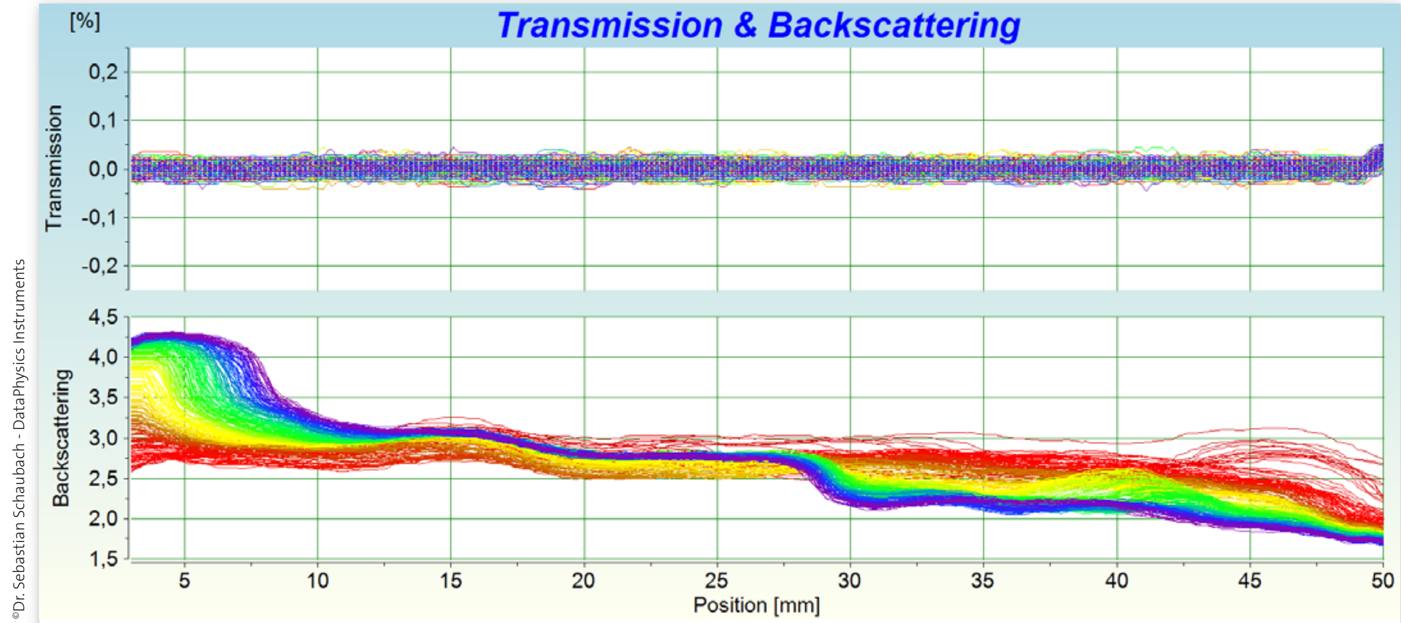
Results and Discussion

The graph at the bottom of this page shows the plots for transmission (top) and backscattering intensities (bottom) against the height position within the sample vial for slurry 1. The colour coding indicates the time at which the individual intensity profiles were recorded. The red profile marks the first measurement (t = 0), the purple the last measurement taken (t = 26 h).

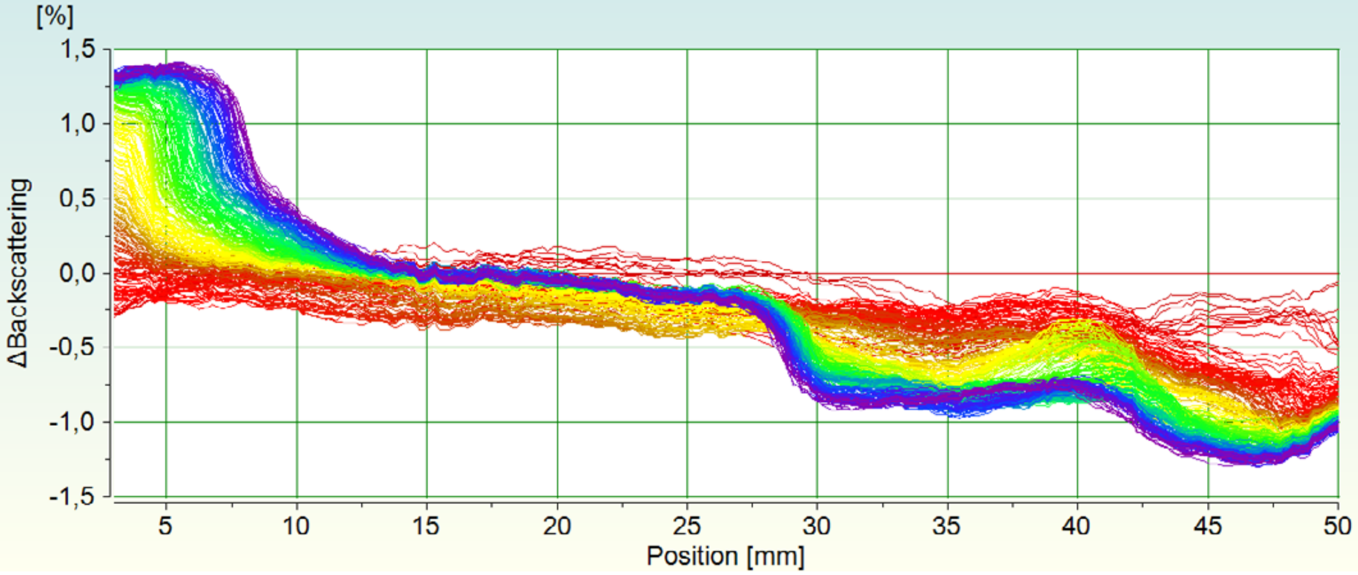
The transmission profiles of slurry 1 show a constant mean transmission intensity value of 0%, which does not change throughout the whole experiment. This profile can be explained by the turbidity of the slurry, which prevents the transmission of any incident light.

The second plot yields more information. The backscattering plot shows a clear time- and position-dependent change in the intensity signal. This plot indicates that slurry 1 is unstable over the investigated period, and that some destabilisation processes are occurring. This result becomes more evident in the relative plot depicting the change in the backscattering intensity compared to the intensity in the initial scan (see graph at the top of page 40).

Regarding the shape of the backscattering profiles in the graph at the top of page 40, it is possible to see an increase in the intensity at the bottom of the sample container along with a decrease in intensity at the top of the sample. This indicates a sedimentation process, with a layer of sediment building at the bottom and the sample and clearing towards the top. The sample could be analysed further to determine the sedimentation speed using the option to analyse the so-called migration front in the MSC software.



Transmission (top) and backscattering (bottom) intensity vs. position diagrams for slurry 1. Intensity profiles are colour-coded from red to purple from the first to the last scan, respectively.



Relative backscattering intensity vs. position for slurry 1; the profile illustrates the intensity relative to the intensity measured at t = 0 s

For the second sample, slurry 2, the results appear very different. As shown in the graph at the bottom of page 40, neither the transmission nor the backscattering intensities changed for slurry 2 during the whole measurement time of 120 hours (or five days). This indicates that slurry 2 is stable in this time frame and hence could be a good candidate for a battery coating slurry.

Summary

The stability of two different battery coating slurries was studied and compared using a MultiScan MS 20 dispersion stability analysis system from DataPhysics Instruments and the MSC software. By recording transmission and backscattering intensity profiles of slurry 1 and slurry 2 for periods of 26 and 120 hours, the researchers identified slurry 2 as stable, while the other, slurry 1, was shown to be unstable.

Due to the turbidity of the two slurries, these results could not be obtained using the transmission data, but only became evident when examining the (relative) backscattering profiles, which showed distinct changes for slurry 1 after a couple of hours. On the other hand, the profiles for slurry 2 showed no changes throughout the measurement period. The shape of the evolving backscattering profiles for slurry 1, indicated that the predominant destabilisation process was sedimentation, as a migrating sedimentation front and a growing layer of sediment were observed.

The MultiScan MS 20 dispersion stability analysis system from DataPhysics Instruments enables position- and time-resolved observation of even the smallest changes in a sample's backscattering and transmission intensities. It thereby enables producers of battery coating slurries to conduct quick, objective stability analyses, helping to anticipate long-term stability and develop batteries for electric vehicles quickly and cost-effectively.

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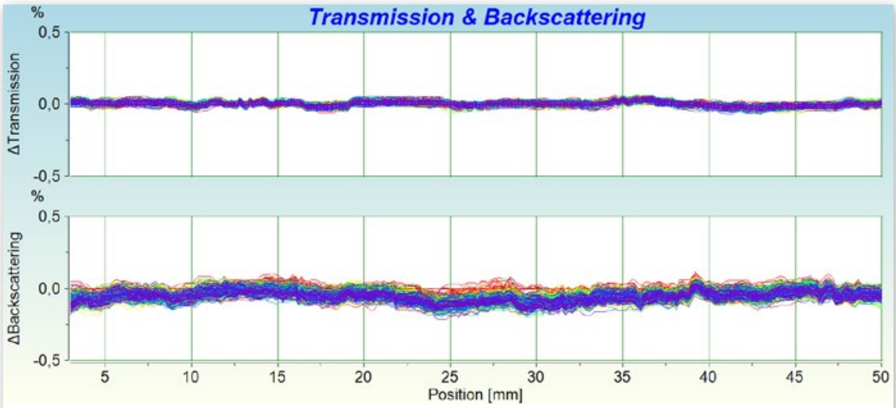


Relative transmission (top) and backscattering (bottom) intensity vs. position diagrams for slurry 2 (intensities relative to the one measured at t = 0 s)

Interview with Dr. Qiongjie Liu, Scientist at DataPhysics Instruments' Application Centre

"An Inspiration to What Our Devices Can Do in Practice"

Dr. Qiongjie Liu has been Product and Application Manager at DataPhysics Instruments since 2021. Among other responsibilities, she writes application notes – such as this one on battery slurries – showcasing how instruments from DataPhysics Instruments can be used in real-life use cases, spanning many applications and industries. Liu studied chemistry and was rewarded a doctorate from the Chinese Academy of Science in 2017 for her thesis: "Cu(II)/SaBOX Catalyzed Highly Stereoselective Synthesis of Poly-Heterocyclic Compounds".

Dr. Qiongjie Liu
M.Sc. Chemistry
Product & Application Manager

***Sanja Döttling*: DataPhysics Instruments regularly publishes application notes containing various measurements and experiments from different fields. Why is it so important to show these different use cases of DataPhysics Instruments' products?**

Qiongjie Liu: While we pride ourselves in making versatile and precise measuring systems, they only fully come to life when used by our customers in a myriad of exciting use cases. These use cases are as diverse as they are surprising. Therefore, our short application notes offer a good way to represent the whole range of our products and their versatile applications in real life, encompassing industries such as cosmetics, oil recovery, foodstuffs, coatings, surface treatments and foundational research in materials science, chemistry and physics.

We not only develop and produce measuring systems but also want to offer fully fledged solutions for our customers' measurement challenges. The different application notes my colleagues and I have written hopefully offer inspiration on what our devices can do in laboratory practice.

***Döttling*: As you have mentioned, the topics of the application notes are very widespread. How do you choose the most interesting topics for new application notes?**

Liu: As mentioned previously, our main goal is to provide solutions to our customers. Therefore, most topics for new application notes are chosen based on our customers' wishes and interests. This also helps us understand what customers might need in the future and which direction the company should take for further development of our products.

Additionally, we keep an eye on the latest academic research papers in the area of surface science, as our devices are used in many academic disciplines for fundamental research. In 2022, for example, Lizhong Wang et al. published an article in *Nature Communications* on how differently structured surfaces influence the dewetting behaviour of water drops during repeated icing and melting. Another academic paper by Xiaomei Li et al. published in *Nature Physics*, looks at how electrostatic forces influence the velocity of water drops rolling off a tilted surface. In both papers, a contact angle measuring system of the OCA series from DataPhysics Instruments was used. Combining our insight into the latest academic research and keeping an eye on our customer's challenges in industrial applications, we can offer a varied selection of experiments in our application notes.

***Döttling*: What, to you, is important when conducting the experiments at the heart of any application note?**

Liu: For me, from a scientific point of view, it is most important that the results obtained in the experiment are reliable and reproducible. I also strive to showcase experimental solutions which are more efficient, faster, or simply more precise than other, already known, experimental setups. In essence, I want to offer the reader concise, practical insights into many different experimental designs possible with our devices.

***Döttling*: As a scientist, you need to be objective in your work. But do you have any application notes that are your personal favourites, and if so, why?**

Liu: I personally like application notes with very well-known samples. Recently, I finished an application note studying the gelation process of yogurt and another one analysing the process of chocolate tempering. The samples seem commonplace, but the phenomena observed in the experiments are interesting and fascinating.

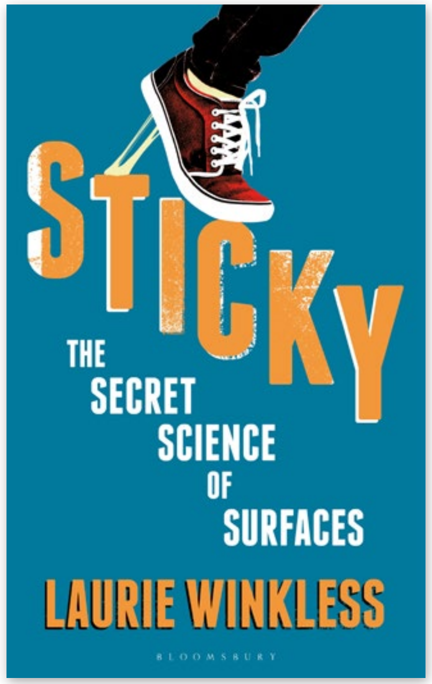
As a scientist, I also support our internal software development team with practical insights gained in the lab. Therefore I am especially proud of the application notes showcasing new experimental methods and analyses possible with our devices' software packages. Last year, for example, we managed to measure contact angles on dental implants with highly hydrophilic surfaces. Here, we were able to put into practice the theory of so-called "imaginary contact angles", developed by H. P. Jennissen.

***Döttling*: Thank you very much for this interview!**

Recommended Reading

Book Review: The Spaces in Between

Everywhere in daily life, different things meet: the car tyre meets the asphalt of the road, the skating blade meets the icy surface of the lake, and one human hand meets another. The study of things meeting, known as surface science, is a fascinating and often overlooked field. In her book Sticky: The Secret Science of Surfaces, *author Laurie Winkless talks about some eye-opening surface science applications. The stories she tells are wowing and insightful and show that surface interaction is a topic filled with cutting-edge science.*



Book Details

Sticky: The Secret Science of Surfaces by Laurie Winkless
Published by Bloomsbury Sigma, London, 2022.
Hardcover, 336 pages.
ISBN: 978-1472950833

What do a car tyre, a curling stone and your fingertip have in common? Laurie Winkless, the author of the book *Sticky: The Secret Science of Surfaces*, says they are all made from material surfaces interacting with other surfaces. In her book, Winkless examines several instances in which one material meets another – in the most interesting fashion.

The Wonders of Surface Science Explained

Winkless starts her book by discussing how a gecko sticks to walls and ceilings. Surprisingly, the forces by which it achieves this are poorly understood today, although scientists have been able to copy some of the gecko's stickiness and incorporate it into biomimetic technology, allowing humans to climb walls just as a gecko would. Another amazing feat scientists have achieved by mimicking biology is found in slick swimsuits. Their surface structure is based on the dermal denticles of sharks. Swimmers wearing swimsuits using such a design broke several international records, leading to the International Swimming Federation banning the suits from competitions.

The following chapter covers the interactions between air and solid materials – here, too, the surface structure leads to interesting results. Among other topics, the author talks about William Taylor, an engineer from England, who patented dimpled golf balls, as they offered a better performance than their smooth counterparts.

However, interactions do not only occur between air and solid surfaces. Winkless also examines the interaction between the tarmac of the road and car tyres. She describes the special properties of rubber, which makes it the ideal material for car tyres. Her research includes a visit to the factory of the Aston Martin F1 team in Silverstone, UK, which gives fascinating insights into the car racing industry.

The next section describes phenomena on a much larger scale: Winkless dives into the topic of geology and specifically examines plate teutonics, that is, how that the Earth's plates rub against each other at plate boundaries. Here, she asks how earthquakes work and why they are so difficult to predict.

Winkless sticks to the ground in the next chapter and asks a surprisingly difficult question: Why is ice slippery? Its slipperiness has some rules that are easy to explain: temperature and a liquid film on top of the ice play an important part in it. However, the topic is much more complex considering the nitty-gritty of ice surfaces.

The book's final chapter is even more challenging, as the author examines human skin. Among other things, she talks about fingerprinting evidence, which is not nearly as scientifically rigid as crime shows want us to think.

A Book for Curious Minds

What makes this book particularly gripping is not only the wide range of topics discussed but also the close attention to detail in every chapter. The knowledge and research the author displays are impressive. The main conclusion to take away from the examples considered is simple: do not underestimate how complex and multilayered seemingly "everyday" interactions between materials are and how difficult it is to explain them scientifically. It is eye-opening to read how much research in surface science is recent and ongoing. In a world such as today's, where we often think we know it all, the gaps in the understanding of interacting surfaces and interfaces are, on the one hand, sobering, and, on the other, a call to action for many researchers.

Winkless formulates this predicament well when she says, "It's really because knowing or observing something is several leaps away from understanding it. And that's the thing about many of the surfaces we're exploring in this book. It's not that we know nothing about them – just like speed-skating ice or asphalt on roads, we've been manipulating and designing surfaces to work for us for decades. But surprisingly often, and usually because of the 'it works, so why worry' attitude, we haven't taken that final step: to understand the complex physics and chemistry of surfaces, and why they behave as they do. And while purely practical knowledge can take us a very long way, it has limitations, especially when we start playing around at the edges of it."

About the Author

The author Laurie Winkless was born in Ireland. She graduated with a B.Sc. in Physics with Astrophysics from Trinity College Dublin and with a M.Sc. in Space Science from University College London. Winkless has worked at NASA's Kennedy Space Center and the National Physical Laboratory in Teddington, UK. Additionally, she has worked as a BBC radio reporter and wrote press releases for the Nobel Foundation. In 2016, she moved to New Zealand and now lives and works there with her husband. Her first book, *Science and the City: The Mechanisms Behind the Metropolis* was published in 2016; *Sticky: The Secret Science of Surfaces* followed 2021.

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Impressum | Imprint

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Handelsregisternummer: HRB 224335
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Druck
Printer

Möhle-Druck GmbH
Silcherstraße 26
86720 Nördlingen
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