Anton Paar

Automated rheometry at Procter & Gamble: Making complex analyses routine

**Brussels Innovation Center (BIC),
Procter & Gamble (P&G),
Brussels, Belgium**

**Measuring instrument:
High Throughput Rheometer HTR**

From its foundation in 1837 producing candles and soap, Procter & Gamble has developed into one of the largest corporations in the world, serving around four billion people in more than 180 countries. The corporate purpose statement, "to provide branded products and services of superior quality and value that improve the lives of the world's consumers", is reflected in the constant drive for innovation throughout the company. In Western Europe alone, Procter & Gamble has eight centers of innovation and research with almost 3,000 scientists working on products ranging from razors to baby wipes, detergents to hair colorants.

Dr. Broeckx and Liesbet Detroch about to load some samples

The research behind the washing detergent

Ariel, Dawn, Downy, Gain, and Tide: These are some of the billion-dollar brands produced by Procter & Gamble in the field of household care. However, the product behind a brand is not static, it is constantly being improved. Once available only as a washing powder, most brands are now also on sale as liquid detergents.

To keep Ariel, Dawn and Co. in the list of the most popular detergent brands, Procter & Gamble employs teams of researchers at its Brussels Innovation Center (BIC) in Belgium. With liquid detergents, the focus is on optimized formulation and responding to the subjective perception of the consumer.

When producing a liquid detergent, for example, it is important to ensure that no unexpected reactions occur when mixing the ingredients. These reactions may have unfavorable knock-on effects in the heat exchanger or result in undesirable sedimentation in the end-product. If the formulation for a liquid detergent is changed or a new formulation is created, research teams at BIC investigate the best sequence of mixing the product.

The consumer's opinion counts

At Procter & Gamble feedback from consumers is taken seriously. Liquid detergents which are perceived by consumers as 'too thin', 'too runny', 'too thick' or 'too gluey' are often rated poorly, affecting the consumers' overall evaluation of the product. Formulating a liquid detergent which exactly corresponds to the consumers' expectations and wishes is an important task of researchers at BIC.

Such investigations into the formulation and processing of washing liquids require specialized equipment and know-how. At BIC, the work of measuring, analyzing and interpreting physical parameters is frequently undertaken by the aptly named "Measure Group", headed by Dr. Walter Broeckx.

"We are here to help people at BIC to understand the processes," he explains. His team works in a well-equipped laboratory. If the task requires it, new instrumentation and technologies are purchased to extend the range of analysis on offer.

Validation is key

Rheological tests on samples by the Measure Group are used to interpret the behavior of the product and make predictions about its physical parameters. Models are built and changes to formulations and processes are made based on these models.

As Dr. Broeckx explains: "You want to have a measurement so that you can make a lot of products, test them quickly and say based on a measurement: 'This one will be stable,

this one will not be stable, this one might be stable.' Then you go and test the ones you believe will succeed. The problem is that you also have to create the reassurance, you have to be sure that the number the rheometer generates really reflects the physical parameter. If not, you are going to discard maybe very good technologies without having a good reason for doing so."

To deliver this reassurance, Dr. Broeckx relies on thorough validation of the models the Measure Group uses. As he explains: "We work with complex products, so inherently these complex products give a lot of variation. To understand what the noise is and what real differences there are between products you need to understand what a variation is, what you can expect within one sample. Unfortunately, in the lab, if I do rheological tests on three samples it just kills half my day. Validating rheological procedures really takes a lot of time."

The power of building good models

However, the need for validation is so great that Dr. Broeckx and his team invest considerable time in their models, as he explains: "For me the power of building good models lies in doing rheological tests on more samples." Typical tests for building models for liquid detergent are investigations into the viscosity at low shear rates.

"It was the time-consuming aspect of rheology that bothered me the most," remembers Dr. Broeckx. For this reason, in the 1990s Dr. Broeckx began to consider a more automated approach. By chance, he learnt about the Automatic Sample Changer ASC 32 from Anton Paar, a rheometer with a sample changer which automatically measures and cleans 32 samples in one cycle.

"Suddenly we could do a lot of measurements."

"Buying the ASC 32 was a huge step forward because suddenly we could do a lot of measurements," remembers Dr. Broeckx. Two ASC 32 rheometers are still at work in his laboratory, busily running through thirty-two samples while lab staff analyze the results of previous samples on the PC. These 'sampler rheometers' are mainly used for tests on samples at ambient temperature. However, Dr. Broeckx was keen for more automation. When he heard of a solution with the possibility of running 96 samples, he was very interested. The new rheometer system was a fusion of rheometer and robot inside a closed cabin. Named the "High Throughput Rheometer", or HTR, this was a new system from Anton Paar, the producer of the sampler rheometers. Within a few months, Procter & Gamble had purchased a "HTR" for the Measure Group. It was installed in the summer of 2010.

"It opened more possibilities."

As Dr. Broeckx recalls, "The HTR goes one large step further than the ASC: Not only for the number of samples but also the flexibility becomes wider. You can do temperature sweeps easily. It more reflects what you do in reality. The robot just replaces the person. It's otherwise exactly the same. It's a standalone rheometer with a robot."

The HTR is a rheometer within a glass booth. The robot arm in the middle dominates the setup. Once loaded with samples, the robot arm swings between the sample magazine, the rheometer and the cleaning unit. Two measuring systems are used simultaneously: as one is in the rheometer, the other is being cleaned. Liquid samples, such as Dr. Broeckx's liquid detergent, are poured into sample vials by laboratory assistant Annelies Verbesselt before starting the operation. Paste-like samples can be squeezed onto measuring plates and placed ready for use in the booth. Before each measurement the robot inserts a new blade into the integrated trimming system, which cuts around the rheometer's bottom plate to remove excess sample material. After the measurement, the robot removes the blade and places it in a container ready to be cleaned. This ensures the same regulated filling for each sample, every time.

Cost-effective solution

The idea behind the technology is that the HTR also works overnight, running tests on 96 samples which are then completed when laboratory staff arrive in the morning. Dr. Broeckx aims to use the machine at its highest capacity: "It runs 24 hours a day, or close to 24 hours. And seven days a week. We fill it completely and analyze, analyze."

The results generated keep him and his team busy evaluating and interpreting - even when on business trips. Via the internet, it is possible to access the PC running HTR from anywhere in the world. As Dr. Broeckx explains: "I spent some time in September 2010 at a university in the UK and I just asked people back at the Measure Group in Brussels to load samples into the HTR and I did the rest. I programmed the machine remotely from the UK. I started it, checked the results, did the analysis. I could do everything I wanted, it was great."

"A valuable piece of kit"

The arrival of the High Throughput Rheometer has changed the speed at which samples can be analyzed, allowing thorough validation of the models used and building in the reassurance that these models are delivering useful results.

Besides this, Dr. Broeckx uses the HTR to run more complex analyses on his samples: "I love the fact that it opens more possibilites in the number of samples and that it has a huge flexibility in the procedures you can use, and so on. But as a researcher, it is also the possibilities, mainly the possibilities regarding temperature that I love. You can go to minus 5 °C easily – It's beautiful. You can look at and probe rheology and you can do that in an automated way. So you can build even more complex analyses and make them routine."

More information about Procter & Gamble:
www.pg.com
More information about HTR:
www.anton-paar.com

Anton Paar GmbH was established in 1922 as a one-man locksmith's workshop. Today, over 1400 employees worldwide develop, produce and distribute high-quality measuring instruments for the determination of properties such as density, temperature and viscosity as well as high-precision mechanical parts and assemblies. Anton Paar has strong links with universities and research laboratories worldwide. The company is owned by the Santner Foundation, which invests in research in the field of science and technology as well as in the rehabilitation of drug addicts.