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Opening photo: **The new, fully automated high-throughput system for testing coating formulations enables to cut down significantly on time spent searching for the optimal formulation.**

HIGHLIGHT OF THE MONTH

Test Track For Coatings

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About 40 million metric tons of coatings are used globally every year. These coatings protect surfaces from corrosion, impart special properties such as electrical or thermal conductivity, and increase the lifetime of consumer goods. Expectations for coatings are steadily increasing. Due to increasing complexity and regulatory pressure, customers are continuously faced with new challenges.

Formulating a coating is an intersection of art, craft, and science. In addition to the main binder component, coatings may also contain organic solvents or water, pigments and fillers, catalysts, co-solvents, and additives. For each of these components, a variety of different chemical substances may be used. The number of possible combinations of the various ingredients is enormous. If in developing a coating formulation, only ten curing agents, ten binders, ten pigments, and ten additives are being considered, then the number of possible combinations is already 10^4 or 10,000. And that doesn't even take into account variations in the relative amounts of the components.

In practice, it is impossible to cover the full range of possibilities and to test the properties of all combinations and proportions.

Figure 1: A track system for transporting panels and containers extends through the entire 120-square-meter HTE system.

On the other hand, the prerequisite for a systematic search for the optimal coating formulation is to investigate this range as extensively as possible at reasonable expense in terms of finances and manpower — because this is the only way of ensuring that nothing is left to chance. Evonik's coatings experts have now found a way to overcome this challenge, by developing a high-throughput system that is exactly tailored to meet the requirements for developing and formulating a coating (**ref. Opening photo**).

Faster testing for the perfect formulation

With this system, Evonik has improved its chances of zeroing in on the additive or specialty binder that perfectly matches the other system components and accounts for the coating's purpose (end use). This is not an easy task. Additives, for example, make up a very small percentage of the formulation, but they have a major influence on the coating's properties. They can impart a defoaming effect, prevent pigment agglomeration, and ensure that the coatings behave thixotropically — that they are easily applied but do not sag or drip on vertical surfaces while drying. Other additives can increase the scratch-resistance of coatings. Many additives are available for perfecting a formulation. The search for the optimal additive(s) is also complicated by the fact that, depending on their chemical composition, the additives can interact with other coating components. The new system is invaluable because it allows for the systematic testing of more formulations than previously possible within a given time frame. For customers, this means they can optimize and develop coating formulations faster, saving valuable time in the market launch of new products. The system is two meters high and occupies a 120-square-meter area in an air-conditioned room (**Fig. 1**). It doses the raw materials automatically and, in the first step, formulates them into coatings on the "formulating island." In the second step, substrates — also known as panels — are coated with the formulated coatings, dried, and transported to the test island, where the properties of the formulations are characterized as soon as the coating formulation is cured (**Fig. 2**). All of the steps run automatically in accordance with a precisely defined program that is reproducible at any time — and that is one strong point of the system.

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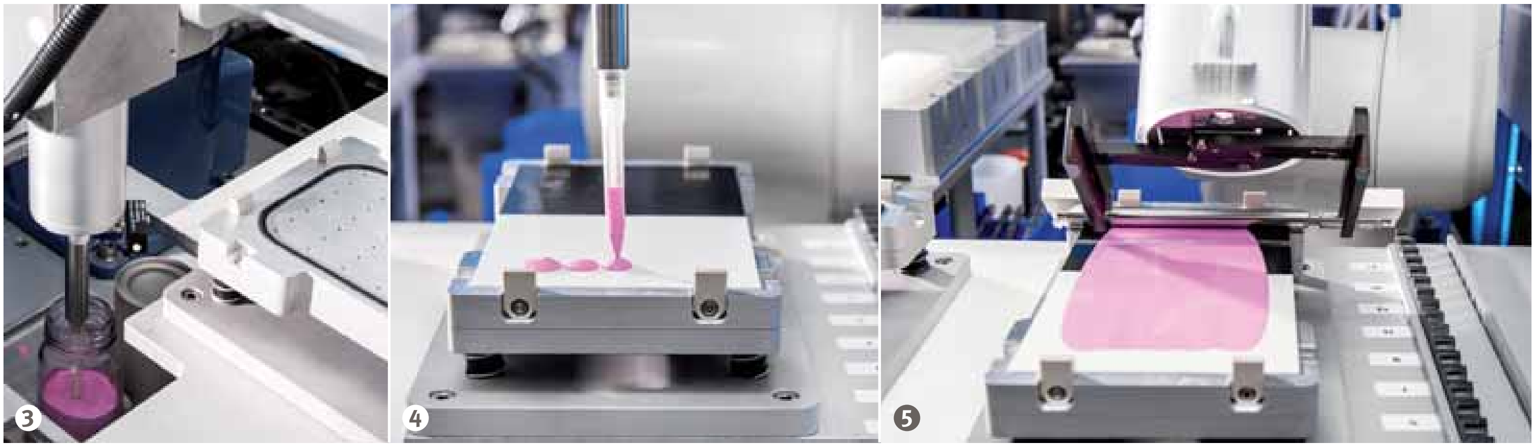
Figure 2: Formulations from various projects.



Figures 3, 4 and 5: Precise and cautious: A robot designed for this unique purpose draws the coating into a pipette, applies coating droplets onto a panel, and uses a doctor blade to produce a precisely defined coating thickness.

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Complex steps fully automated

Formulate, coat, characterize: It sounds simple enough, but in fact the machine is tasked with performing a large number of complex steps. The system consists of 52 elements that combine to give 30 functionalities. Each functionality represents the performance of a particular task, such as applying a coating formulation on a panel.

The 52 elements are linked by a track system that extends through all parts of the HTE system; on this track system, containers and panels are transported by shuttle. There are also 13 robots performing various tasks, such as loading shuttles or placing panels in the oven. On average, 120 samples can be formulated in the system within 24 hours. While they are being applied to a panel and characterized, experiments for a new project can be initiated, hence the name High-Throughput Equipment (HTE).

The dosing and mixing of the raw materials are particular challenges for the system. For example, the viscosity of the liquid raw materials being used can vary from extremely free-flowing to so viscous that the substances must be heated before they can be dosed and filled. Moreover, certain liquids can attack the materials of containers and tools by dissolving them. Pigments and fillers in powder form can also be tricky to handle. Their bulk density and flowability varies; depending on the powder, this could complicate accurate weighing and dosing. While a well-trained technician quickly learns how to handle these raw materials, this is not an easy task for an automated system. But the new HTE system rises to the occasion, thanks to well-developed software that allows for the setting of all the parameters required for dosing. For liquids, these parameters include pressure, temperature, and the opening times of valves. For powders, on the other hand, these parameters include the speed of the metering screw during gross dosing, the region of fine dosing, and the fine-dosing amplitude. In the master RaceLab software, developed by Evonik itself, all of the input and output data required to operate the system are collected in a single database. Thanks to optimized engineering, the HTE system doses reliably and reproducibly. It registers any deviations from target values and saves the information. In case of anomalous results, it is then easy to trace whether irregularities occurred in the workflows. The strengths of the system are also evident in the production

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and testing of pigment concentrates. Pigments must normally be ground by grinding media in a highly time-consuming process. The HTE system has a special mixer for this purpose, in which double rotation of the mixing cup ensures significantly faster grinding and dispersion. Moreover, a specially provided air cooler prevents excessively high heat generation, which could destroy the raw materials. The subsequent separation of the grinding beads from the sometimes highly viscous mixtures, as presently performed in the laboratory, is highly labor-intensive; in the HTE system, this is done by a centrifuge. A specially developed filter adapter allows the coating material to pass through while retaining the grinding beads; the necessary movements are performed by a robot.

Objective parameter instead of a subjective test

To evaluate the storage stability of a pigment concentrate in the laboratory, a visual inspection and the use of a spatula reveal whether a sediment has been formed. Transferring the corresponding optical and tactile capabilities to a machine would have required enormous expenditure and effort. The developer of the HTE system found another solution: A measuring head determines the mechanical resistance it encounters when lowered into the sample. In this way the somewhat subjective human assessment is replaced by an objective parameter. Another step that poses a challenge to the fully automated system is the application of a coating formulation using a doctor blade. The panel must not slip during application, the quantity of the coating must correspond to the desired coating thickness, and the surrounding area must not be soiled, because that would contaminate the following panel. These tasks are assumed by a robot specially designed for this purpose. Panel after panel, the robot applies exactly the same quantity of coating in exactly the same thickness and cleans the doctor blade in various wash solutions before repeating the procedure (**Figs. 3, 4 and 5**). The HTE system also offers advantages in the rub-out test for assessing pigment stabilization. In the laboratory, this is performed by rubbing the freshly applied coating layer with a finger; if the pigments are not sufficiently stabilized, the shear forces applied result in a visible color change of the rubbed surface. The skill in this test lies in not displacing material while rubbing and not causing gaps to occur in the coating layer. The HTE system replaces this subjective test with a precisely coordinated control system whose parameters are individually adjustable for any coating system via the software. Altogether, the high-throughput system reduces the burden on laboratory staff by taking over onerous routine tasks, leaving employees free to concentrate on planning experiments and analyzing the data.

> **“RaceLab allows all workflows to be planned in detail. No fewer than 40 different work steps can be precisely defined with up to 600 parameters in the HTE system. This allows Evonik to freely combine individual steps into a workflow so as to satisfy the most diverse customer requirements. The HTE system can therefore be used very flexibly – and around the clock.”**

Flexible use around the clock

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Figure 6: **Since 2012 Claudia Bramlage is the responsible of the High Throughput Equipment (HTE) System at Evonik Resource Efficiency, Coating Additives business line.**

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Figure 7: **Ellen Reuter is the responsible of the raw materials development for the particles stabilization and for the High Throughput Equipment (HTE) system.**

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The strengths of the HTE system

- Operates fully automatically, provides reproducible results, can be used flexibly
- Formulates coatings from a wide variety of different raw materials
- Characterizes the fluid formulations prior to application
- Coats the panels precisely and dries or cures the coating
- Characterizes the finished coatings
- Produces a continuously growing data pool as a basis for future analyses via data mining.

The experts

Claudia Bramlage (**Fig. 6**) and Ellen Reuter (**Fig. 7**) are responsible for the operation of the new HTE system in Innovation Management of the Evonik Resource Efficiency Segment.

Discover the new test track for coating formulations from the inside:

1 High Troughput Equipment - VR shuttle tour | Evonik

2 High Troughput Equipment - VR technical tour | Evonik