Coatings Development:

Moving to the Fast Track

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The desire to speed up the product development process has always been present in the coatings marketplace. However, the drivers for reducing time-to-market have never been as numerous and the pressure as intense as they are today. Many leading formulators and raw material suppliers have discovered that high throughput experimentation (HTE) techniques can have a measurable impact on the development process, allowing for not only reduced time in the lab, but often creation of higher performing products.

High throughput technology, also referred to as high throughput screening and sometimes as combinatorial chemistry, has been widely implemented in the pharmaceutical industry for many years. Catalyst manufacturers have been more recent adopters of HTE techniques. Many specialty, consumer, and commodity chemical companies are now benefiting from application of the technology as well.

HTE involves the use of robotics to enable parallel screening of very large numbers of micro-sized experiments at the same time, accelerating scientific research. For coatings, the technique can be applied at all stages of the product development process, including initial formulation, application, and performance evaluation.

In fact, the increasing interest in HTE by the coatings industry likely reflects the growing awareness that the technology can be applied to formulation and performance testing, two activities that are critical for coatings research, according to Damian A. Hajduk, vice president of Performance Materials and Formulations for Symyx Technologies, Inc. Initially, HTE was seen as being useful outside of the pharmaceutical sector for synthesis-related development work, and the impact for industries such as coatings was limited.

"Coatings manufacturers aren't just selling chemicals. They are selling performance, and this performance can be difficult to predict from chemical composition alone. For coatings, then, synthesis is only the first step,"

Hajduk explains. "Any newly synthesized resin often has to be formulated into a model coating, applied to a surface, cured, and tested across multiple performance dimensions before its value can be estimated.

With the recent advances in HTE capabilities, it is now possible to synthesize resins, formulate them into coatings, apply them to several different substrates, and measure properties such as flexural modulus, glass transition temperature, and chemical resistance at rates of 400–500 formulations per week.

"A couple of years ago, researchers from industrial labs were somewhat skeptical regarding HTE techniques, but this attitude has disappeared over the years and we are now seeing HTE adoption by leading performance materials companies," notes Dr. Philipp Hauck, business development manager with hte AG.

Not only has the technology improved significantly, but the sector has become much more receptive as well. "Another important key driver has been changing environmental regulations, leading to a shift to waterborne, high solids, and energy cured coating formulations which require extensive testing to demonstrate the performance and benefits of new compounds," Hauck adds. HTE helps the industry shorten the development cycle and can fulfill this task in time to meet legislation deadlines.

The primary reason for investing in high throughput capabilities, though, is the growing realization that HTE has an economic benefit, according to Sarah Eckersley, technology director and head of coatings HTE for Dow Chemical. "It enables faster speed to-market and higher performing, more novel solutions," she states.

High throughput equipment suppliers are offering new capability specific to coatings preparation, application, and testing, while major raw material suppliers are finding that HTE allows them to develop a much richer understanding of how their products can contribute to enhanced performance.

"One of the main reasons for the increased amount of interest is the effectiveness of the technology," affirms Abdullah Ekin, Ph.D., head of High Throughput Testing at Bayer MaterialScience LLC. "Companies that have HTE have a significant competitive advantage due to realizing faster development." He notes that the interest level in Europe seems to have grown more rapidly than it has in North America.

This advantage can be gained in many different ways. One of the most common applications for HTE is screening a wide variety of resin families and types for a specific application, which serves as a basis for where to start to formulate, according to Ekin. Another is optimization of a coating by changing different parameters within the formulation. Most agree, though, that high throughput techniques are not very well suited for finetuning of coating formulations or optimization of a final resin formulation.

Coatings formulators are faced with the task of optimizing a very large number of chemical compositions and process conditions. In the majority of cases, these factors are ... strongly interdependent and therefore have to be investigated in a non-decoupled way. "The main advantage of a well thought out HTE concept is the complete workflow and data integration including Design of Experiment (DoE), formulation, application, and characterization. Therefore, HTE techniques enable better understanding of formulation-substrate interaction and are much more than an increase in the number of experiments." Hauck comments.

The results can be dramatic. "What previously took years, now takes months; and what took months, now takes weeks," Hajduk

HTE Supplier Info

hte AG offers technology and software solutions for automated formulation R&D and automated coating R&D, together with consulting services on how to implement these systems into an existing laboratory environment.

Symyx Technologies performs contract research for coatings manufacturers involving the synthesis, formulation, chemical characterization, and physical characterization of coatings. It also manufactures and sells high throughput instruments used to carry out these activities.

Flanders Materials Centre has installed HTE equipment and software to enable experimental workflows in the areas of viscous coatings, high speed curable coatings, and chemical vapour deposition (CVD), and makes this expertise available for projects with interested partners. The company also offers modeling services for eliminating experiments with little likelihood of success, allowing focus on the potentially most rewarding areas.

BASF's High-Throughput Screening facility offers services on robotic equipment designed to test coating formulations quickly and economically, enabling optimization of pigments, resins, and additives for desired performance characteristics. Solvent and waterborne formulations containing solid and liquid ingredients can be mixed and/or cured, applied, and tested on various substrates. The company's intelligent data management system enables quick processing of results.

Bayer MaterialScience offers a range of services and products for high throughput experimentation from the provision of state-of the art reaction and formulation technologies, through high-performance data processing, to the application of mathematical processes in planning experiments (DoE).

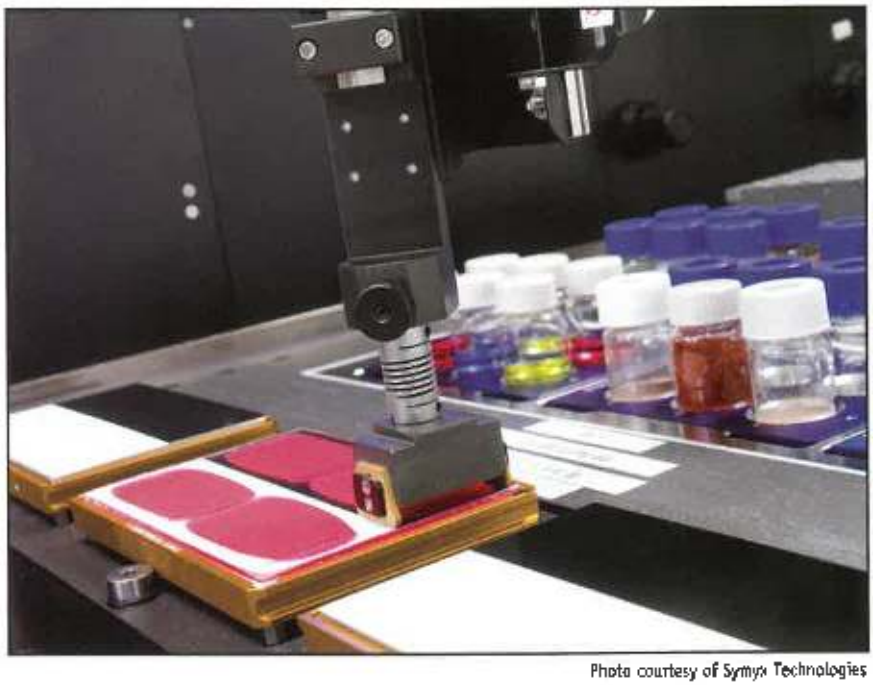


Photo courtesy of Symyx Technologies

states. Turnaround times improve, and the number of projects a team can take on in a given year increases. Some companies, he says, have used this to conduct more thorough research, identifying the best performing formulations for commercialization and patenting while leaving areas of weaker performance for their competition. Others have used it to increase the number of projects a team can take on in a given year.

HTE also lets manufacturers invest their research budgets efficiently. "The expansion of HTE into physical testing lets researchers estimate the performance of *every* formulation at an early stage in the research," explains Hajduk. Large quantities of material, often requiring scaleup, are no longer required before these properties can be measured. Teams can therefore identify promising directions at an early stage and focus their efforts there. Less promising research directions can also be identified quickly, and the time spent on them can be minimized.

Formulation development using high throughput technology is one area receiving a lot of attention in the coatings sector. "HTE is particularly well suited for solving problems with multi-component formulations where there may be synergistic or antagonistic interactions between components," remarks Hajduk. And the greater the number of formulations to be explored, the greater the advantage of HTE.

"Formulations represent a huge challenge for the researcher," agrees Nick Gruber, who is responsible for Global Strategic Management of Resins with BASF AG. "Numerous different ingredients, varying in type and concentration, have to be handled; structure-property relationships are hard to predict; and the variation of chemical composition and process conditions is almost infinite. In this field, HTE methods are a powerful tool for increasing the efficiency and effectiveness of R&D."

Specific problems include ingredient substitution and simplification, according to Hajduk. HTE can be very useful when a key ingredient must be replaced for cost or environmental reasons. Because the replacement material is not exactly identical to the original ingredient, its impact on performance is hard to predict. HTE also allows manufacturers to identify what each ingredient in a formulation contributes to performance, and the level at which this performance is optimized. The use of expensive ingredients can be reduced to the level that yields the desired performance, and additives that contribute little or nothing to the coating can be eliminated.

Other typical development problems where HTE offers significant support for R&D are compatibility studies; the influence of process parameters such as curing time or energy on coating properties; development of new color shades by formulation; and adjustment of additive packages to resins and vice versa, according to Gruber. "From a coating technology point of view, there are, in principle, no limitations. Radiation-curable coatings can be tested in an HTE setup as well as thermally cured coatings. Both 1K and 2K formulations with a potlife can also be studied, but for 2K systems, timing of workflow is essential because of viscosity issues. Our experience has shown, though, that this challenge can be realized in fully integrated automated robotic systems."

The advantages of high throughput technology are not realized, though, by simply running a lot of experiments in parallel on robotic systems. "The sheer amount of experiments that can be done will not lead per se to a better understanding and faster result," notes Hauck. HTE is only effective if the search is narrowed down to relevant materials or processes to be tested early on in the research process. Therefore, the Design of Experiment stage is critical in determining the successful application of any HTE program.

HTE equipment maintenance requires unique skills as well. "Dedicated personnel with the requisite expertise are needed to fully capitalize on the potential of the in-

vestment, which can be significant," says Eckersley. "The most important thing to consider is having the right team of people who will be able to understand and use the equipment. This includes expertise in software and hardware, as well as knowledge of designing and executing large experimental designs, and typically involves a scientist, chemist, technician, and engineer," agrees Ekin.

Knowing how to manage the large quantities of data that are produced and be able to convert that data into useful information is also important. "In order to tap the full potential of the powerful HTE techniques, a fast evaluation of the huge data sets as well as the acquisition of structure-property relations is absolutely essential," asserts Gruber.

Data management systems with graphic display of the results offer good solutions, but there is still room for improvement. "The bottleneck is no longer execution of the experiment but the processing and intelligent interpretation of the data," adds Hauck. Gruber further believes that, "Only if this challenge is tackled successfully will HTE techniques really make a contribution to increasing the efficiency of R&D."

Dow Chemical's coatings business, which has partnered with Symyx on its HTE activities, has invested in an integrated set of robotic instruments and modeling and data management software that can be used to formulate coating compositions, create coatings on a variety of substrates, and analyze both liquid formulations and coating samples for several key properties. Facilities have been implemented in Dow's new coatings R&D labs in Midland, MI, and Shanghai, China.

"Dow has made a substantial and strategic investment in HTE as a transformational way of doing R&D. We use HTE throughout our product development process—from early stage research through designing specific formulations with optimized performance," Eckersley notes. In all cases, the company chooses to use the HTE approach when it makes technical sense and will result in new value.

"Using HTE technology, we are changing the way we develop new products and explore new solutions with our customers," explains Wendy Hoenig, global R&D director for Dow's coatings business. "Through new polymer development and formulation latitude studies, we are working on innovative solutions that meet historic and emerging industry needs with chemistries we would not have considered before. Current projects include enabling new environmental or 'green' coating formulations, enhanced environmental durability, reduced application and formulation costs, smart coatings, and others."

With its HTE capabilities, Dow optimizes coating performance by finetuning the complex interactions that occur between the numerous different ingredients. For example, Dow recently conducted a series of experiments using HTE targeted towards low VOC and low odor results in an architectural paint. In just 11 weeks, 15,000 end-use performance tests on 288 paint formulations were completed.

"We found a formulation region of low odor and low VOC that had significantly better performance than the commercial paints on the market," Eckersley notes. "HTE allowed us to probe regions of the formulation space that would never have been tried using a traditional ladder approach."

In addition to development of water-based coatings, HTE is also well suited for coating application processes for UV-cured lacquers, according to Hauck. HTE can simulate coil coating processes for UV or IR curing or dual curing, and short to ultra-short exposition experiments can be run that could not be achieved manually.

"Exact reproducible timing is something automated systems are especially good at," Hauck says. "There is a range of different HTE coating technologies that are well developed and reliably reproduce the corresponding manual laboratory procedures. They even excel, with higher reproducibility and higher integrity compared to the standard manual operations."

Abdallah Ekin also stresses that making assumptions about correlations between conventional methods and high throughput methods must be avoided. "Before a new method is generated using HTE, feasibility studies need to be done to confirm the correlation between conventional methods and high throughput methods."

Many HTE approaches simplify existing practices in order to achieve high throughput, but such simplifications can inadvertently eliminate key steps needed for meaningful results. One good example, according to Hajduk, is the use of drawdowns for sample creation. Most measurements of interest can be performed on drawdowns, but there are others, such as surface finish measurements, in which specific deposition methods like spraying need to be used in order to obtain results representative of the real application.

"When setting up HTE programs, it is always wise to check the extent to which HTE results on 'known' samples correlate with 'known' results—and if the correlation is weaker than expected, consider whether key steps of conventional practice have been changed or eliminated," Hajduk emphasizes. It is essential, Gruber adds, when employing high throughput methods, "to formulate, apply onto substrates, and characterize coatings in a manner as close to reality as possible in order to guarantee the transferability of the results for practical purposes."

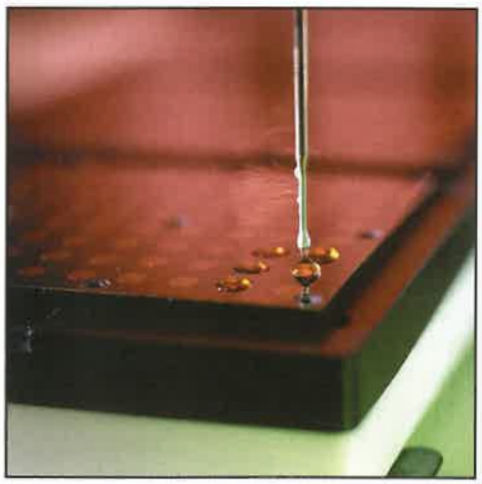
It is also important to realize that high throughput techniques cannot

practically replace all development efforts. For example, large-scale performance tests (such as ASTM tests) that are expected by customers take a considerable amount of time and resources to perform, but automating such tests at the relevant scale would require a very high level of investment.

Symyx looks not to *replace* large-scale performance tests, but to *restrict them to coatings most likely to succeed*, according to Hajduk. High throughput physical testing is used to measure combinations of fundamental physical properties that are thought to underlie the properties measured in typical large scale performance tests. For example, glass transition temperature measurements could be used to estimate performance on a softening temperature test, or surface friction measurements in the presence of different solvents could be used to assess chemical resistance of a cured coating.

Coatings that show acceptable performance in the high throughput tests are then scaled up and sent through the battery of large-scale tests that manufacturers use today. Dow, which follows this strategy, uses scheduling and logistics to keep output maximized when the time duration of different tests does not perfectly align.

Bayer uses HTE for many internal applications such as catalyst screening for a new chemistry or resin, optimization of different parameters in a coating formulation, and understanding of raw material properties and the performance of materials in different types of formulations, according to Ekin. High throughput is also a very important customer service offering for the company. "We use HTE to help our customers solve problems, particularly when time is of the essence. And we also use it for our potential customers' needs to show feasibility for a project," he explains.



Substrate depositing (Photo courtesy of Symyx Technologies)

It is large companies like Dow, Bayer MaterialScience, and BASF that are implementing R&D strategies that increasingly rely on high throughput methods. "The best fit and highest added value of applying HTE techniques is with companies employing clearly defined long-term development objectives," notes Hauck.

Smaller companies are often hesitant to adopt the technology because of the high initial investment. HTE centers like the Flanders Material Centre in Belgium—which has installed equipment and software for coatings development—or other institutes, could serve as places where HTE technology is accessible on an R&D service basis, Hauck believes.

"The coatings industry is still in the early adoption phase with regard to HTE. Major raw material suppliers and larger coatings companies are leading the way, often conducting joint projects to explore the potential of this new technology," states Eckersley. "Interest will continue to grow as economic and technical value is demonstrated one project at a time."

Just in the past 5–10 years, according to Ekin, many questions about the value of HTE technology for coatings formulation development have been addressed by different instrument suppliers. Also, there are many more companies who are supplying instruments and introducing new capabilities compared to five years ago. "The increasing availability of new equipment will further interest in both the short and longer term," he notes.

As high throughput techniques are more widely used, shorter time-to-market for novel raw materials and formulations will be realized. "Eventually, HTE techniques will make it possible for companies to react immediately to customer requests and offer highly targeted solutions," says Gruber.

The ability to explore complex formulation spaces efficiently will continue to make HTE attractive to the coatings industry, according to Hajduk. "As more and more measurements become available in high throughput form, the value of this approach will only increase," he says. Hauck points to future developments such as laboratory devices for characterization that are more suitable for integration into automated workflows, centralized data acquisition, and processing and improved informatics tools for more sophisticated visualization and data mining.

"We must always recognize that HTE supports but does not substitute classical optimization in the lab," asserts Gruber. "Keeping that in mind, I firmly believe that high throughput techniques, which are already increasing the efficiency of coatings development today, will see even wider use in the future." CT