High-throughput formulation and coating workflow (Symyx Discovery Tools, US)

This platform, developed by Symyx® Discovery Tools Inc., includes a complete suite of software tools for experiment design, equipment operation, data storage, and database retrieval and analysis. All libraries are bar-coded so they can be tracked as they move from tool to tool.

1. Overview of the High-Throughput Workflow

Combinatorial or high throughput experimentation is based upon the concept of a workflow, as shown below (Figure 1). The workflow is a series of process steps taken during the course of an experiment starting with experiment design and ending with analysis of the data.

Each process within the workflow is designed to accommodate regular arrays of miniaturized samples referred to as libraries. Libraries typically contain 2, 6, 24, 48, or 96 samples (Figure 2). A centralized database is used to track samples and store data for efficient data management.

![Figure 1: Platform for combinatorial or high throughput formulation and coating screening.](image)
The libraries are evaluated using unique rapid analytical or testing methods to identify promising material candidates referred to as hits. Hits are used as the basis of further experimentation or scaled-up to laboratory scale to validate performance and generate additional performance data.

2. Experiment Design

Library Studio™ from Symyx Discovery Tools is the primary design tool (Figure 3). Designs are stored in a common database and information is used to control dispensing of reagents in synthesis and formulation systems. Statistical experimental design can also be used to design experiments with the output from these programs providing input directly into Library Studio™. Design Expert™ from Stat-Ease is available for designing experiments using statistical methods.
3. Formulation and Powder Dosing Station

The preparation of formulations involves the dispensing and mixing of a variety of polymers, crosslinkers, and other additives. Many resins and crosslinkers are relatively high viscosity materials and can be difficult to handle. Dispensing of these resins is accomplished through the use of dilution, disposable pipet tips and mixing by magnetic stirring. By using a disposable tip for each dispense, the chance for cross contamination is reduced and the need to thoroughly wash the pipet between each use is eliminated (Figure 4).

Additionally, the formulation station is fitted with a custom designed viscosity measurement tool, which can measure the viscosities of the coating formulations (up to 1000 cps) as they are created. In this way for example, by setting a target viscosity for the entire library, the station will automatically measure the viscosity, dilute with solvent, mix, and measure again until all of the samples in the library have similar viscosities. This is important as the library might be passed onto the next station where the polymers are coated onto substrates. Any changes in the compositions of the library are automatically updated in the database to maintain accurate information as the library progresses through the workflow.

![Figure 4: Dual-Arm Liquid Handling Robot.](image1)

![Figure 5: Many-to-many Autodose Powdernium.](image2)

Besides liquids, also powders can be dosed very accurately. This station consists of a plate storage hotel for 9 microtitre plates, a balance, and hopper storage racks holding 10, 25 and 50 ml storage containers (Figure 5).

4. Coating Application System

After a library of polymers has been formulated into a coating, it is then passed on to the Coating Application System where the individual coatings are applied to a substrate panel (Figure 6).

This system is set-up to handle the 4x6 array library format and will generate a 24 element array of coating "patches" applied to panels made of metal, glass, plastic or wood. The system is designed to accept 4” x 8” standard test panels.

The liquid coating is applied to the substrate using a disposable pipet, and then a doctor blade spreads the coating into a film. The gap of the doctor blade can be adjusted to yield different wet film thicknesses (up to 8 mils) and is automatically cleaned and dried between applications. Once cured, the coatings can be evaluated for surface energy, corrosion protection, abrasion resistance, biofouling, adhesion, color and gloss.
Thicker films up to 1,000 microns can be prepared using a multiwell aluminum panel. Coatings are dispensed into the wells until a target volume is reached and leveled through vibration. These thicker films are particularly useful for measuring foul-release characteristics of coatings.

5. Coating Screening

Coating screening can be carried out using a variety of methods depending on the goals of the specific study.

Coating Surface Energy

This automated system measures the surface energy of coatings by placing a droplet of liquid on the surface and measuring the contact angle. Dynamic contact angle measurements are also possible with this instrument (Figure 7).

The coating surface energy station receives two standard 4x8 inch metal coating panels that have been prepared by the coating application station. Each of the 24 elements within a library can then for example be subjected to three water droplets and three methylene iodide (MI) droplets. The contact angle of each is measured using image analysis and the readings are in this case averaged for both water and MI. Finally, the Owens-Wendt method can be used to calculate the surface energy.
6. Data Analysis

Data analysis is carried out using a number of software tools. Renaissance™ PolyView™ software provides a flexible way to search for and retrieve data from the Renaissance Application Server™ (RAS) based on data stored during synthesis and screening activities (Figure 8). The user can view combinations of data from multi-step synthetic processes, which are cross-linked by the RAS to provide consistent and clear access to all data collected through the lifetime of a test sample.
This data may be displayed in custom reports, including spreadsheet tables, 2D or 3D scatter plots, 2D or 3D bar charts, grid views, and library summaries. Reports can include numeric and textual data as well as an extended set of custom data types, including spectra, chromatograms, and images. Links are also provided to send data to external text files, Microsoft Excel™, and Spotfire DecisionSite™, for additional visualization and analysis.

7. Additional Equipment

Gas chromatograph. A gas chromatograph equipped with a CTC autosampler is used to determine the product distribution of high-throughput liquid phase catalytic reactions, formulations, …

Automated Balance. A Bohdan Automated Balance is used to automatically weigh arrays of vials. It can be used to verify dispense accuracy of the automated dispensing systems, perform weighing of vials before and after solvent evaporation to determine polymerization conversion, and other tasks.

Centrifuge. A centrifuge is used to e.g. separate heterogeneous catalysts from the liquid phase reaction mixture, …

Curing Ovens. Several ovens are available for thermal curing of coating array samples.