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Developing New Adhesive Formulations

Laboratory Automation for New Adhesive Formulations

The automation of laboratory work in adhesives formulation relieves chemists of manual sample handling and generates solid bases for statistical evaluations. A system was developed and built for the Henkel Adhesive Technologies research center that mixes small quantities of adhesive formulations precisely, and applies these to sample carriers without loss.

The Adhesive Inspiration Center, which opened in 2021, will help to accelerate the market launch of new adhesive formulations, while at the same time testing as many variants as possible and scientifically exploiting the results. The purpose of the system developed by Füll Lab Automation is to produce adhesives fully automatically and apply them to specific carrier materials or to generate material samples for further testing. In this way, new and customer-specific adhesives are constantly being developed for a wide range of industries.

Dosing different raw materials, mixing them and applying the produced formulations directly to sample carriers or substrates is one of Füll Lab Automation's core competencies. In the case of the system for Henkel, the aim was to take the special properties of adhesives and raw materials for adhesive production into account when designing the system (*Figure 1*). The spectrum of raw materials and their consistency ranges from powdery, solid, pasty or highly viscous to liquid. Here the task was finding a process that allows as many different formulations as possible from these raw materials. Various raw materials are automatically added to a liquid base and mixed in accordance with the desired formulation. The final formulation is then applied to substrates or injected in molds for further testing.

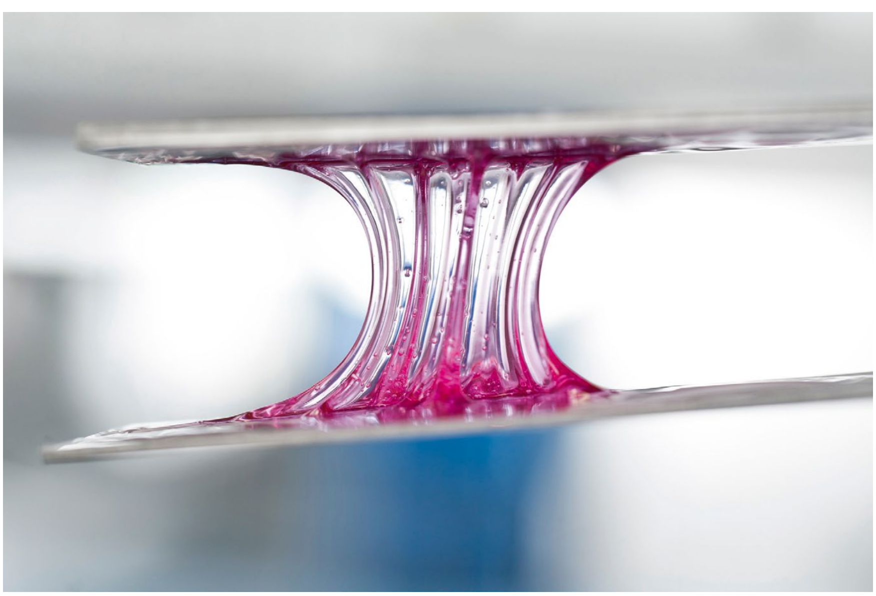
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Figure 1 > With ever new formulations, new adhesives can be developed for different application areas.

With the BLS syringe Füll Lab Automation has an appropriate dosing technology for this process in its product portfolio (*Figure 2*). This syringe consists of a cylinder, which is used as a formulation container, and a separate piston, which can be automatically inserted into the cylinder. The main advantage of this technology is that the formulation is created and then applied directly from the same container. This principle works even with higher viscous liquids or pastes, which in principle cannot easily be aspirated. The vacuum which can be reached in a standard syringe or direct displacement pipette is usually not sufficient to move for example adhesives without bubbles.

Applying a formulation directly from the BLS syringe to a substrate and creating a thin layer by doctor blading is a well-known process from the paint and coatings industry. The system at Henkel uses

this application method to produce samples which can be used to investigate the curing behavior or adhesive strength of adhesives, for example.
In addition, the production of “dog bones” adds an application method that Füll Lab has implemented for Henkel. Here, similar to 3D printing, the adhesive is filled along defined paths into small molds reminiscent of chewing bones for dogs. The resulting dog bone shaped samples are suitable for testing the tensile strength and elasticity of the adhesives. Samples with overlapping joints for shear tests can also be prepared, to determine the resistance of a bond against shear forces.


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Figure 2 > The BLS syringe is suitable for high-viscosity adhesives as for paints and coatings or low-viscosity formulations.

Automated system with 21 active modules

A so-called Integrated Lab Station (ILS) forms the base for the system at Henkel (*Figure 3*). This modular, fully automated solution is particularly suitable for handling a large number of samples. All elements for handling and the desired automation steps are laid out by Füll Lab on a customer-specific base. Only the initial loading of raw materials, formulation containers and substrates is carried out manually; all subsequent steps are automated. The entire system consists of 21 active modules and comprises three modules for dosing liquids and three for powders in the formulation section. The formulation is mixed directly in the syringe cylinder in one of four so-called Speedmixers, three of which additionally operate with vacuum (*Figure 4*). In this mixing process the container is centrifuged in an inclined position and simultaneously rotated around its own axis. Through the combination of both movements and the forces that occur, the components are mixed homogeneously, and air or gas is removed from the sample. This degassing is additionally supported by the vacuum.
A major advantage of this process, especially when handling adhesives, is that no

Figure 3 > Ten meters long and four meters wide, the system houses 21 active modules.

additional stirring or mixing elements are required to which raw materials or the formulation would adhere. The formulation is prepared and mixed in the BLS syringe cylinder and afterwards applied directly from it. This prevents the composition of the formulation from changing through the process steps and eliminates the need to clean a mixer or agitator.

Four additional modules are available for capping and uncapping of sample containers, for example before and after mixing in the Speedmixer or at the end of the workflow. Other active modules take on the doctor blading and creation of dog bones, as well as heating of adhesives samples.

Both the range of raw materials and their individual properties are important for selection of the dosing technology. The BLS syringe is particularly well suited for materials with high viscosities, which are otherwise difficult to dispense, as well as for liquids with very low viscosities. For powders or powder-like raw materials, a special, closable powder container is used. This container can also be used to dose fibrous solids in such a way that the fibers are retained. The handling of the syringes, for example the raw materials and the formulations, is classically carried out by a robot (*Figure 5*).

Sample sizes of 80 ml or less

The BLS syringe makes it possible to significantly reduce the amount of material needed for new formulations. While quantities of several 100 ml are common in the manual process in the lab, the BLS syringe works with a sample quantity of 80 ml or even significantly less. This is possible because both the syringe and the powder container can precisely dose even the smallest amounts of raw materials. As a result, waste volumes can also be reduced considerably – compared with the typical preparation volumes of several kilograms in the laboratory. The considerably reduced sample quantity and the use of the BLS syringe contribute significantly to saving of raw materials in several respects: The amount of raw material required for preparing the formulation is lower, there are no losses due to decanting of the finished formulation, since no additional containers, pipette tips or other disposables are required, and the amount of solvents required for cleaning is much lower. In total, this reduces the amount of waste significantly.

If larger quantities of adhesive, for example more than 80 ml, are required for certain tests, the system also works with larger formulation containers without the need for any conversions or modifications of the system. The modular concept allows further functions or additional equipment to be connected without any problems.

No research without statistics

Research has long been moving away from intuitive approaches based on the experience of individuals and toward statistical methods. These can be used to determine how changes to individual parameters affect the result. The "Workflow Manager" software package developed at Füll Lab is responsible for the processes within the ILS plant, for example the recipe and the mixing process. The transfer of results then takes place via an appropriately configured software interface to the customer's system.

A decisive factor in any scientific work is the reproducibility of results. This means that all data must be recorded in such a way that they can be referred to and compared with other values at any time. The goal is to use Design of Experiment (DoE) to determine formulas that allow predicting properties. DoE software calculates how many experiments are needed to determine the coefficients or factors of the formula. The software also specifies the parameter variations that are important for the mathematical evaluation of the test results. The more input factors there are, the more complex the formula becomes, and the more tests are necessary to determine the factors. It

Figure 4 > Vacuum speed mixers enable precise mixing and complete deaeration of high-viscosity formulations, even in small quantities.

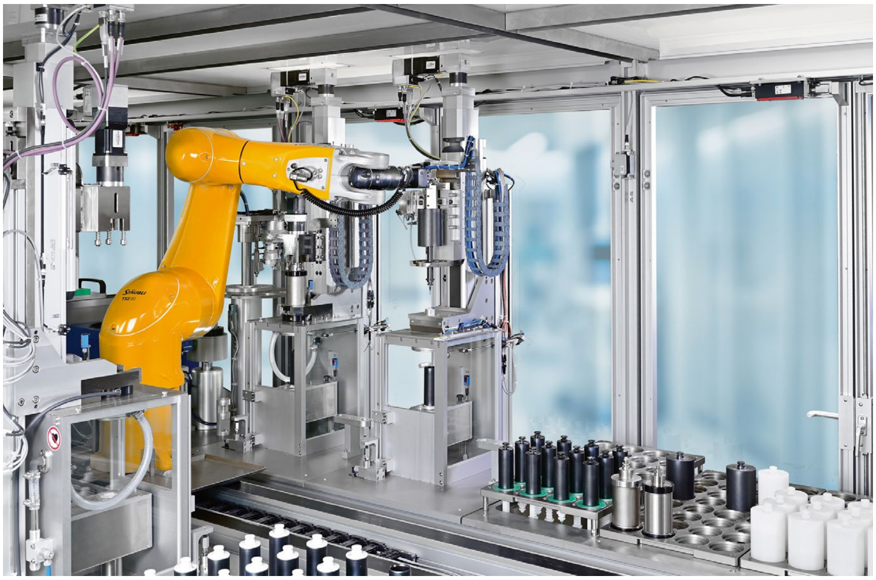

Figure 5 > The handling robot transports a raw material syringe.

is important that all the tests as well as the results in an automated process are documented. On this basis, it is possible to determine which of the many factors in a formula is the most decisive in order to best achieve the specified goal.

Conclusions

The advantage of an automated solution is that it is easy to log all the key data of a test, for example the amount of components added, the duration of mixing or the temperature, and all the data determined with measuring equipment. Furthermore, the repeatability, and the linkage of already existing data with newly acquired data enables verification of results or allows the prediction models to be made more precise. //

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