Combinatorial compounding allows the rapid production and testing of large numbers of polymer and additive formulations. **Nadya Anscombe** reports on two very different approaches to the technique

Rapid compound development

Polymaterials carries out its rapid compound development work on a specially modified injection moulding machine (above right) fitted with multiple feeds and mixers plus automated testing systems

Combinatorial chemistry is a technique that has revolutionised many industries including chemical synthesis, pharmaceuticals and biotechnology. But this idea of trying various combinations of materials to create large arrays of new materials is only now making inroads into the compounding industry.

Conventional methods of developing new compounds can be tedious and time-consuming. The iterative process of choosing a compound that needs improving, experimenting with additives and testing the materials is slow and laborious. This method often does not give the best possible product because not all combinations of polymers and additives were tested due to time constraints. In today's market, where the focus is on new formulations, rather than entirely new plastics, and where recipes are becoming increasingly complex, there is a real need for a faster development technique.

One of the main reasons why the compounding industry is a few years behind other industries in adopting a combinatorial approach to product development is the compounding industry is interested in the physical properties of the polymer blends. Testing hundreds of samples quickly, ideally on-line, is a huge challenge.

At the **German Polymer Institute** (DKI) in Darmstadt, Germany, researchers have met this challenge head-on and designed a combinatorial compounding line that integrates as many as 10 different on-line tests. "Our system uses a melt divider to divide the melt into three streams," explains Juergen Wieser, head of the technology department at the DKI. "With the first melt flow we perform tests on the melt such as viscosity. The second melt flow is pelletized and these pellets can be used for off-line testing such as injection moulding. The third melt flow is fed into a flat film line and this is where most of the testing takes place."

The film first passes through a non-destructive testing station where various different kinds of spectroscopy can be used to test a wide range of film properties such as colour, clarity, degradation, crystallinity and particle size distribution. These tests can be carried out on the moving film without damaging it. However, mechanical tests require a stationary piece of film in order to perform the test. So that these tests can also be performed continuously and on-line, the DKI researchers incorporated a dancer roll into the system. This acts as a buffer while the extrusion line is running and gives the researchers a minute or so to perform the mechanical tests. These include lateral and longitudinal tensile strength, as well as tear strength.

The system is also able to monitor the degassing process inside the extruder by using an FTIR spectrometer at the degassing vent. "We dilute the gasses with nitrogen before they are analysed by the FTIR spectrometer," says Wieser. "The spectrometer can give valuable information about the chemical processes taking place inside the extruder."

The combinatorial compounding line at DKI is able to change recipes continuously. For example, one additive can be ramped up from 0 to 2% while another additive could be ramped down from 5 to 0%, giving a gradient

of different properties over a length of film.

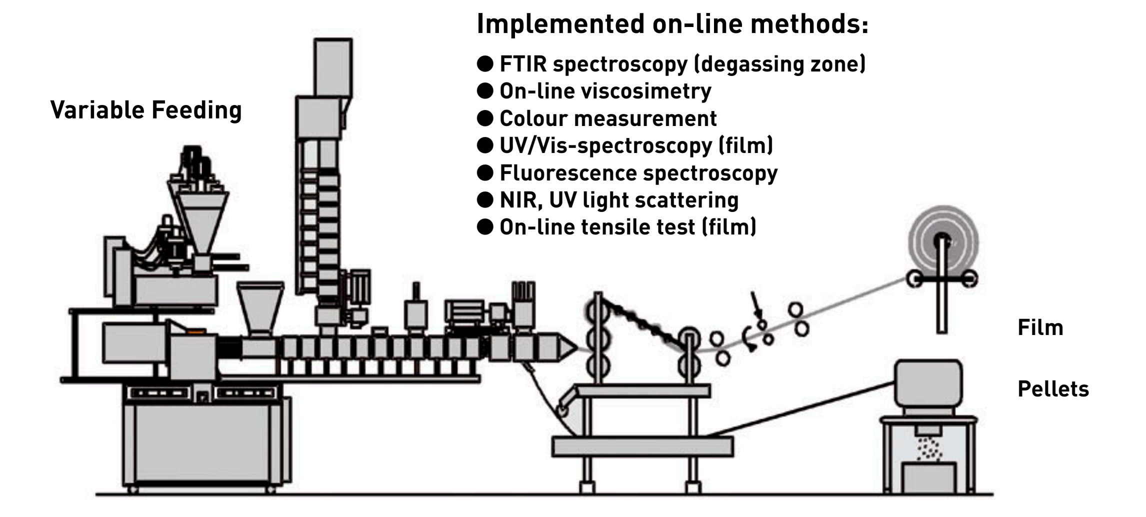
Researchers at German company **Polymaterials** have taken a different approach. Their system uses an injection moulding machine to produce standard test bars which are then tested using standard test procedures including tensile strength. "When we designed the equipment it was important to us that it could produce standard test pieces which could then be used in internationally recognised test procedures," says Dr Gerhard Maier, chief technology officer of Polymaterials. "Our high-throughput screening technique is much faster than conventional methods of compound development and also needs smaller amounts of material." This enables the system to work with experimental materials which are not available in large amounts.

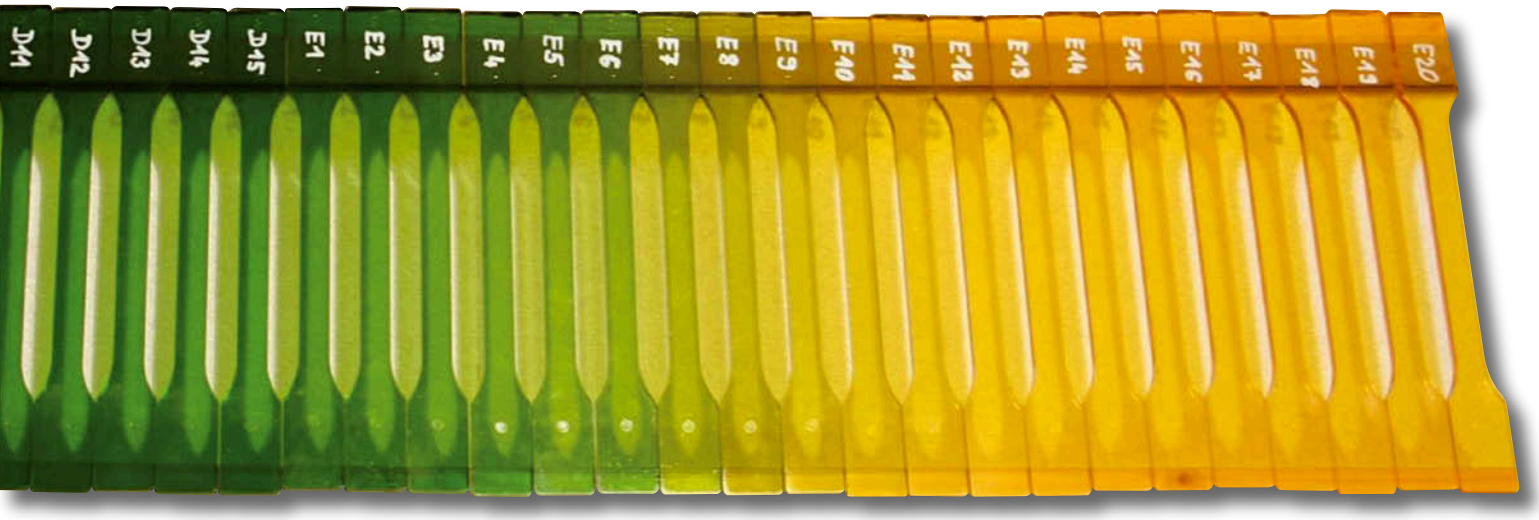
While combinatorial compounding is based on the idea of combinatorial chemistry used in the pharmaceutical industry, it can not process millions of samples as is the case in that market. "Experiments still need to be designed and individual components need to be carefully selected," says Maier. "But our system is still much faster than conventional methods. In one week we can do two or three iterations of a compound development. Using conventional methods this process would take several weeks."

Polymaterials combinatorial compounding system is based on an all-electric injection moulding machine with two plasticising units. Each plasticising unit has four dosing units giving the system the capability to process as many as eight different materials. The melt streams are homogenised using static mixers, then combined and subsequently remixed using more static mixers. "Many people have expressed doubts about the use of static mixers and an injection moulding machine as opposed to a traditional twin-screw extruder for compounding," says Maier. "But our results are comparable to those obtained from a traditional compounder, even for reactive compounds where the residence time is critical."

He points out that the main aim of combinatorial compounding is to identify trends and promising recipes, and not to obtain absolute values for mechanical properties of certain blends. Polymaterials' system changes the recipe in a step-wise fashion, producing a fixed number of test pieces using one recipe, followed by a purging step where the test pieces are discarded. A fixed number of test pieces are then produced using the new recipe. These test pieces are then fed into tensile strength and impact testing machines. The company uses software designed by Bayer Technology Services to

THE DKI COMBINATORIAL PROCESS SPLITS THE OUTPUT FROM A COMPOUNDING EXTRUDER INTO THREE STREAMS: ONE FOR TESTING THE MELT; ONE THAT THAT IS FED INTO A FLAT FILM LINE; AND ONE THAT IS PELLETIZED





The Polymaterials process can change the recipe in a step-wise fashion

generate recipes, design experiments and also analyse the test data. "We are also able to perform other, off-line tests, such as softening temperature and surface hardness," says Maier. "We also plan to add other tests - such as flame retardancy - to the line depending on our customers' needs."

Both DKI and Polymaterials are still developing their equipment and plan to increase automation and add more testing stations. They are both interested in working with compounders to further improve their systems and help compounders develop new material blends in a fast and efficient way. DKI says its system can handle up to 200 mechanical tests per day while Polymaterials system can process up to 40 different material compositions in a day. Imagine what a difference that could make to your product development.

www.dki-online.de

www.polymaterials.de