AZOM.com | Interview conducted by Kal Kaur

insights from industry

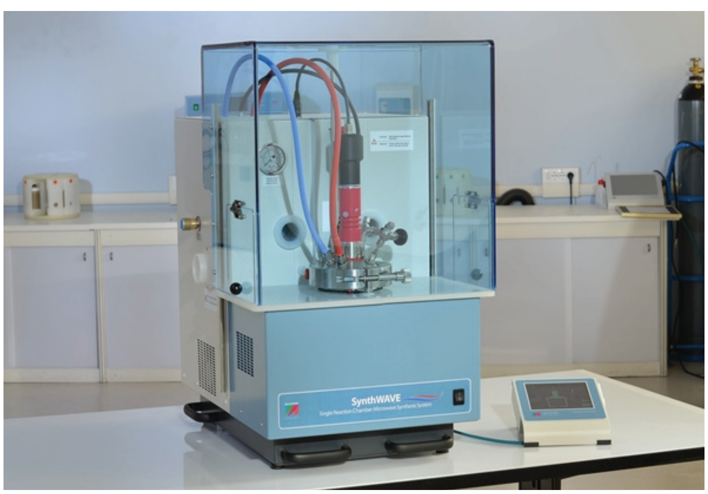
David GUNN

Applications Manager at Milestone Inc.

David Gunn, Applications Manager at Milestone Inc., talks to AZoM about Parallel Microwave-Enhanced Synthesis for Reaction Screening.

KK - You have a range of products within your Microwave Synthesis category. Why is the SynthWAVE single reaction chamber (SRC) technology a revolutionary addition to this spectrum of products?

DG - The SRC technology is a shift in design concept. Traditional microwaves have either used small scale-single mode reactors or benchtop multi-mode microwave cavities to run reactions in parallel. Outliers are flow-through reactors and large batch reactors. Our product line has built specific designs from a multimode cavity with flexibility on the inside, such as the RotoSynth and the Flow/BatchSynth, each with the same benchtop cavity, but with different features to explore specific types of chemistry. For example the RotoSynth is designed to provide better mixing of materials, while the FlowSynth allows samples to flow through the cavity over a programmed period of time.



The Single Reaction Chamber technology is a fundamental movement away from classical design. The idea is to have a large chamber where single or multiple independent reactions can be performed simultaneously. In order for that process to work, the chamber also becomes the microwave cavity. The chamber is designed for high temperature and pressure – the key to the process for multiple independent reactions to work is to have the chamber pre-pressurized with an inert (or reactive depending on the reaction) gas to a specified amount.

There is a microwave absorbing liquid, which sits at the bottom of the chamber (a pool if you will) where a rack full of multiple reactions are lowered into and the chamber closed. An internal thermocouple sits in the absorbing liquid to measure, monitor and provide feedback to the magnetron to supply microwave energy to the chamber. Pre-pressuring the chamber allows the boiling points of any of the solvents and the absorbing liquid to be raised.

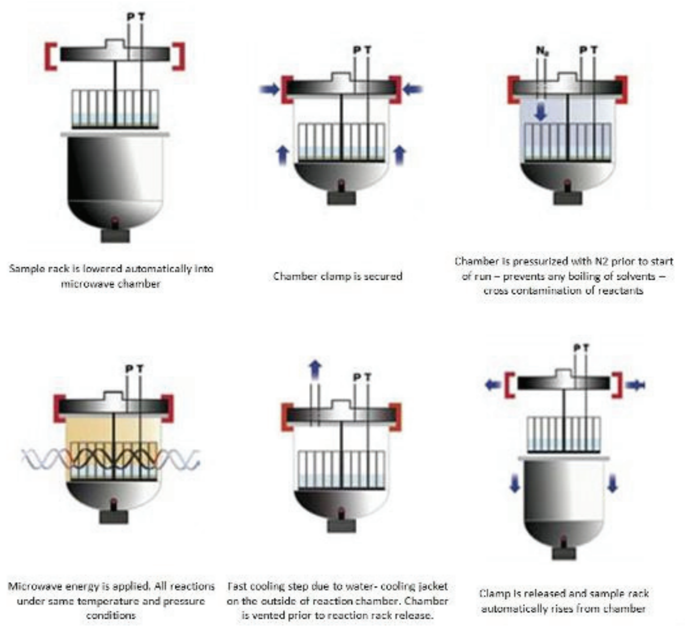
As the temperature and pressure rise, the reactions and chamber reaches an equilibrium – all of the reactions are all at the

MILESTONE

same temperature and pressure for a specific period of time. Unlike conventional systems, where having different solvents in each reaction would absorb power at different rates and reach different temperatures in an uncontrolled way.

KK - How does SynthWAVE work?

DG - As an expert in the field – I currently run a blog on microwave chemistry for synthesis – there are several areas of research that are discussed in an open forum and as a resource (totallymicrowave.wordpress.com). A recent post on the site



SRC operation.

describes the SynthWAVE being used in an academic lab.

SRC operation is an automated process (figure 1). During this process, a reaction rack is loaded with several reaction weights directly into glass vials. The glass vials are secured with Teflon caps which helps to minimize condensation entering the vials. Following this, the reaction rack is then secured onto the chamber top and mechanical stirrer. Both the chamber top and the mechanical stirrer are then lowered.

The reaction vials are exposed to a consistent "load" created in a microwave liquid pool and the chamber is pressurized with N2 (typical: 40 bar). This process prevents any boiling of solvents or cross-contamination of reactants. Microwave energy is then applied with all reactions running under the same temperature and pressure conditions. Termination of the heating cycle initiates a water cooling process that brings the chamber to an ambient temperature which releases pressure. The chamber is opened and the rack is removed as the final step in this process.

KK - What are the key features of SynthWAVE?

DG - Key Features of SynthWAVE include:

- Capability of screening and scoping reactions simultaneously
- High temperature (up to 300°C) and high pressure 199 bar with multiple gas valves to operate under modified conditions.
- The chamber has a cooling jacket on the outside where cooling can be controlled through the software interface (fast cooling for organic chemistry and controlled fast/slow for some materials applications.
- Multiple vessel options and stirring options to run small and larger scale reactions (the 1 L chamber offers the ability to take a reaction on small scale and transfer those conditions to a larger scale.
- Running parallel reactions using a Microwave-based Reactor.

KK - How many reactions can the user run with this microwave-based reactor?

DG – This reactor can run 22 independent reactions in a single operation.

KK - At what temperatures can you run these reactions?

DG - Technical specs (300°C) we generally recommend reactions up to 260°C and the chamber can be modified to run at 270°C for short reaction methods.

KK - What small-scale synthetic methods are easily transferrable to SynthWAVE?

DG - Anything that has been traditionally used in normal syn-

SRC Synthesis

Running parallel reactions using a Microwave-based Reactor.

KK - How does the SynthWAVE in combination with SRC technology compare to traditional microwave synthesis processes?

DG - The key difference is that reaction screening and multiple reactions can all be run in a single method. Traditional microwaves require these modifications to be made one method at a time. SRC allows chemists to modify catalysts, solvent and starting reagents during one operation, allowing a true screening process for research.

KK - How have recent advancements in microwave synthesis changed the approach to organic synthesis and material research?

DG - There have been several changes in the last few years. Chemists can now perform reactions on scale with larger commercial batch reactors. Advances in microwave reactors have changed how chemists are starting to use the technology – truly a way to obtain production scale in a small platform. Lastly, fundamental research using SRC technology offers a new way to screen and scope for optimal conditions utilizing microwave irradiation. On the materials side, microwave technology has moved into new frontiers. Inorganic chemists, polymer and nanomaterial researchers can make materials that have eluded them by conventional heating processes.

KK - What method development efforts are in place to advance microwave synthesis?

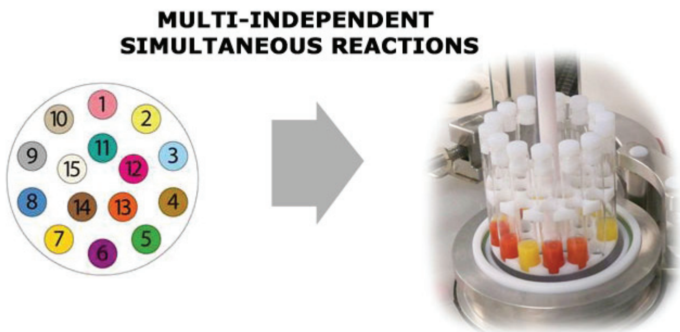
DG - With the growth in nanoparticle research and applications derived from these materials, this is where the development is currently taking place. Catalysts, semi-conductor materials, photo-catalytic materials to trap undesired products, carbon nanotube grafting to target medical therapy are some of the newer efforts in research.

On the reactor design side, much effort over the last 4-5 years has been in the effort of making larger reactors or modified microwave flow-reactors to improve the capability of making larger quantities of material.

KK - Where can we find further information on your products and services?

Further information can be found at the Milestone site.

About David Gunn



Multi-independent simultaneous reactions.

David Gunn received a B.S. in Chemistry (1990) from Loyola University of Chicago. He continued his studies while working as a research associate at Abbott Laboratories. After several years, David moved east and joined the medicinal chemistry department at Bayer Healthcare and The Institute for Diabetes Discovery (IDD).

As a principal investigator at Bayer and IDD he contributed to several clinical candidate selections (Sorafenib, Lidorestat and Pozanicline). He is currently an Applications Manager with Milestone Inc. and utilizes microwaves in a number of industries. His scientific papers can be found in Journal of Medicinal Chemistry, Current Topics in Medicinal Chemistry, Synthesis, among others.

Date Added: Oct 18, 2013

http://www.azom.com/article.aspx?ArticleID=10164

MILESTONE