MICROWAVE CHEMISTRY UNDER PRESSURE: THE FIRST TWENTY YEARS

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In 1986, organic synthesis and chemical manufacture were conducted often by wasteful methods and with equipment that had seen little change for at least half a century. Also, yield and cost typically were considered more important than safety issues and environmental effects of chemical activities. Hence, we began developing technologies and methodologies for safe, environmentally benign, synthetic organic chemistry. Microwave applications soon became key planks in the strategy [1]. A continuous microwave reactor (CMR) and microwave batch reactor (MBR) were the first microwave systems specifically designed for chemical processing with organic solvents [2]. With the CMR and MBR, times usually were decreased by two to three orders of magnitude compared with conventional reactions. The systems tolerated internal pressures of typically 2-5 MPa and allowed mixing, temperature measurement, post-reaction cooling, control of microwave input as well as sample addition and withdrawal during runs. Cleaner processes resulted through use of less or no catalyst and readily recyclable solvents or media. The microwave methodology often afforded higher yields than normal. Now, commercial systems based upon the rationale, the patents and/or know-how underpinning the MBR and CMR are distributed globally [3]. Gains in efficiency accrue through process intensification. The reactors enable more reactions per unit time and through robotics and computer control, extension of the chemist's working day, to 24 hours if desired. The operating range for solvents normally boiling below 100 °C is extended by up to 150 °C. Water at high-temperature becomes a *pseudo-organic* solvent and acid or base-catalysed reactions typically required less catalyst than normal and proceeded more rapidly [2]. Products sometimes were isolated from cooled reaction mixtures by adsorption onto hydrophobic resins rather than by solvent extraction. Thus, convenience, efficacy and low cost were combined with environmental advantages including negligible toxicity, safe handling and disposal, to make water-based organic synthesis a practical and productive new field. These developments have created a need for rapid translation of conditions for lengthy, traditional processes into faster and more convenient microwave protocols. Software has been developed for the researcher to determine desired reaction conditions involving any time, temperature and yield [3]. It employs an iterative, interactive approach that is reliable and applicable to diverse and unrelated reactions. Typically, the desired conditions are obtained within two iterations (*i.e.* two calculations and two experiments) and the success rate is high. Since 1986, microwave chemistry aided by dedicated pressurised reactors has created a paradigm shift in synthetic chemistry and in approaches to it. The field of Green Chemistry has emerged and the highest priorities for the chemical industry are safety and the environment.

Reviews

[1] Strauss, C. R., *Aust. J. Chem.*, 1999, **52**, 83.
[2] Strauss C. R. and Trainor R. W., *Aust. J. Chem.*, 1995, **48**, 1665.
[3] Roberts, B. A. and Strauss, C. R., *Acc. Chem. Res.*, 2005, **38**, 563.