A NEW AND ORIGINAL MICROWAVE CONTINUOUS REACTOR UNDER HIGH PRESSURE FOR FUTURE CHEMISTRY

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Abstract

A new and original high pressure reactor has been designed and developed for continuous flow chemistry under microwaves at industrial scale. The reactor originality is that the microwave applicator is the reactor itself. It allows then the use of metallic and thick walls for the reactor adapted to a use at high pressures and high temperatures. Wave propagation coupled to heat transfer in the full system was simulated using COMSOL Multiphysics® and the design was optimized in order to minimize wave reflections and maximize energy transfers in the reacting medium. This leads to extremely good energy yields. Experiments confirm that the microwave energy is fully absorbed by the reacting medium. The reactor allows continuous chemical reactions at a kg/h scale, under microwave heating, up to 70 bar and 200 °C. The double dehydration of hexylene glycol has been performed under various operating conditions and showed a good production of 2-Methyl-1,3-pentadiene and 4-Methyl-1,3-pentadiene demonstrating then the operability of this new reactor.

Keywords

Novel reactor, process intensification, microwave, high pressure, flow chemistry.

Introduction

Microwave chemistry has known a major development in the last decades, since its first use by Gedye et al. (1986) and Giguere et al. (1986). Chemists have taken advantage of the rapid and volumic microwave heating for accelerating reactions, reducing the process time and sometimes, improving product yield and selectivity. Numerous published works have been done in lab scale apparatus of few milliliters, mostly in batch mode, in multimode microwave cavity, without precisely controlling the temperature and the electric field distribution. But despite this fact, microwave flow chemistry has rapidly found its place in laboratories because it appears as the solution to scale-up and production at a larger scale. The first to propose a continuous microwave reactor for chemistry was Chemat et al. (1996). In the review of Benamara et al., (2015) on the existing continuous flow microwave reactors, the technological evolution is described until now from the microreactor scale to the industrial scale. It appears that the main challenge for industrialization is to cope with the penetration depth of the microwaves in the reacting medium. It is about a few centimeters for common solvents at ambient temperature and atmospheric pressure, when working at 2.45GHz. But at higher temperatures and elevated pressures, dielectric

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properties of the medium can be radically different and both microwave cavity and applicator have to be fully designed to allow the wave propagation for these new operating conditions.

Performing chemical reactions under microwaves at high temperature is obviously an interesting challenge. High pressure is the way to increase the operating temperature keeping the media in the liquid state and obtain faster kinetics. For these reactions, microwave technology allows a substantial power input (commercial generators of 6kW are available for a long time at 2.45 GHz, and more at lower frequencies) leading to fast and efficient heating, especially if a large reactor volume and a significant flowrate are used. Nevertheless, electromagnetic waves have to cross over the walls of the reactor in order to heat inside, and this necessity is a real barrier to the development of microwave reactors under pressure. Common materials like glass, quartz or Teflon, which are transparent to microwaves do not resist to high temperature and pressure. Other materials, like ceramics, have to be used but are relatively fragile. Definitely, waves have to cross over the thick walls and a substantial part of the energy can be lost by reflection and/or absorption.

The new reactor presented in this paper, has been designed to run chemical reactions under microwave up to 70 bar and 200°C, in liquid phase, with a production flowrate of about 1 kg/h. Very few reactors of this type exist to our knowledge. The closest working in the range of our operating conditions under microwaves has been developed by Morschhäuser et al. (2012). Compared to this latter, the originality and advantage of our new reactor is that the microwave applicator is the reactor itself. The reacting material is directly flowing inside the metallic guide where waves are propagating, limiting then energy losses. The guide can then be as thick as necessary for containing the pressure and pipe and sensor connection is simplified.

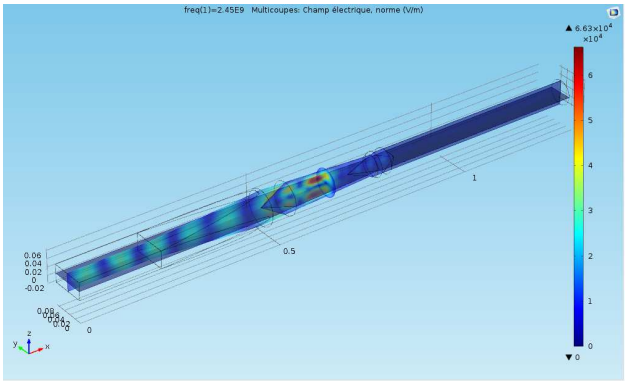

Figure 1. Schematic view of the electric field distribution in the system

In this paper, a first part will be dedicated to the reactor description and to the design work, accomplished to obtain the final configuration. Wave propagation (see Figure 1) in the two parts of the system will be described and temperature distribution shown under reacting conditions. In a second part, operating condition limits will be scanned and performance validation and energy efficiency will be presented. In a last part, results on the double dehydration of hexylene glycol (see Figure 2), continuously run in the reactor at various operating conditions will be given.

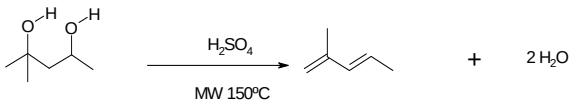

Figure 2. 2-Methyl-1,3-pentadiene synthesis from hexylene glycol

Conclusions

A new and original pressurized microwave reactor is now available for continuous chemistry at the kg/h scale. Chemical synthesis performed under various pressure and temperature conditions demonstrate its operability. The preliminary design work is of first importance and wave propagation has to be finely studied for allowing penetration in the reacting medium and optimal energy yields. This study represents a new step towards industrialization of microwave continuous flow reactors for future chemistry.

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