Chemical Engineering Journal 211–212 (2012) 432–441

Contents lists available at SciVerse ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

Design, fabrication and characterization of microreactors for high temperature syntheses

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HIGHLIGHTS

- ▶ A new technological approach for microreactors fabrication is proposed.
- ▶ The microreactors integrate microfluidics and high temperature actuators.
- ▶ Their thermal and hydrodynamic performance was exhaustively characterized.
- ▶ Devices can be applied to the intensification of processes, e.g. quantum dots synthesis.

ARTICLE INFO

Article history:
Received 25 June 2012
Received in revised form 18 September 2012
Accepted 22 September 2012
Available online 4 October 2012

Keywords:
Process intensification
Microreactor
High temperature synthetic processes
Quantum dots
On-line syntheses
Thermal/microfluidic characterization

ABSTRACT

Microfluidic reactors offer many potential advantages in several research and industrial fields such as processes intensification, which includes a better reaction control (kinetics and thermodynamics), a high throughput and a safer operational environment (reduced manipulation of dangerous reagents and low sub-products generation). Nevertheless, scaling-down limitations appear concerning the materials used in the fabrication of microreactors for most of the liquid-phase reactions, since they usually require high temperatures (up to 300 °C), solvents and organic reagents. In this work, the development of a set of modular and monolithic microreactors based on the integration of microfluidics and a thermal platform (sensor/high-temperature heater) is proposed to perform high temperature reactions. The reliability and performance of both configurations were evaluated through an exhaustive characterization process regarding their thermal and microfluidic performance. Obtained results make the devices viable for their application in controlled and reproducible synthetic processes occurring at high temperatures such as the synthesis of quantum dots. The proposed microfluidic approach emerge as an engaging tool for processes intensification, since it provides better mass and temperature transfer than conventional methods with a reduction not only of the size and energy consumption, but also of by-products and reagents consumption.

1. Introduction

Conventional chemical syntheses commonly require the use of round-bottomed flasks, beakers, test tubes or even more complex equipments. Additionally, they are usually performed under moderate or high temperatures in organic media, involving the use of oil/sand baths or hot plates. In concrete, the temperature at which a reaction takes place is one of the most important parameters to be considered, since all reactions require a minimal energy to reach the activation threshold that enables to transform the reagents into a desired product. Increasing the temperature in a reaction, not only provides the necessary energy for the reaction to take place, but also favors kinetics [1]. Elimination reactions [2], nucleophilic substitutions [3], thermal cycloadditions [4], intramolecular rearrangements [5] and palladium catalyzed reactions [6] are some of the reactions that demand moderate temperatures. Other reactions involving thermal decompositions require higher temperatures, since in these processes it is necessary to break chemical bonds of reagents [7]. In fact, some nanomaterials syntheses, which nowadays are extensively studied due to their wide range of applications, require temperatures that might reach values up to 300 °C, for example quantum dots synthesis [8–10].

Chemical research is focused on the synthesis of new compounds, their application, and on process intensification. Hence, it is important to develop new equipment or techniques, which

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http://dx.doi.org/10.1016/j.cej.2012.09.101