Control Engineering Practice 77 (2018) 225–234

Contents lists available at ScienceDirect

Control Engineering Practice

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

Advanced process control and monitoring of a continuous flow micro-reactor

Furqan Tahir [a,*], Ewan Mercer [a], Ivan Lowdon [b], David Lovett [a]

[a] Perceptive Engineering Ltd, Vanguard House, Sci Tech Daresbury, Cheshire WA4 4AB, UK
[b] Centre for Process Innovation, Redcar, Cleveland, TS104 RF, UK

ARTICLE INFO

Keywords:
Continuous flow reactors
Model predictive control
PLS calibration model
PCA monitor
Fault detection

ABSTRACT

This paper presents a real-time advanced process control and monitoring scheme for a continuous flow micro-reactor producing an ether compound. A PLS calibration model is designed to predict the ether product yield using inline spectral data. This yield is then controlled to the desired setpoint by the proposed MPC scheme. Through real-time results, it is shown that the MPC controller is able to deliver accurate setpoint tracking even in the face of substantial plant-model mismatch caused by diluted catalyst. Furthermore, it is shown that the designed PCA monitor can effectively detect process/reaction faults, such as irregular reaction chemistry.

1. Introduction

Continuous flow reactors for the synthesis of organic compounds have received substantial attention from academia as well as the industry (Corning Inc, 2018; Fabry, Sugiono, & Rueping, 2016; Hessel, 2009; Jähnisch, Hessel, Löwe, & Baerns, 2004; Schwalbe, Autze, & Wille, 2002). This is due to their several advantages over the more traditional batch manufacturing approaches. For instance, batch reactors typically suffer from scale-dependent dynamics which invariably mean process/chemistry re-design when transitioning from lab scale to production scale. Flow reactors, on the other hand, overcome this limitation through the introduction of adjustable throughput and scale-out (instead of scale-up) of continuous flow units, i.e. replication of multiple units rather than dimensional changes (Jensen, 2017). Other benefits include possibility of precise spatial and temporal control for optimising product yield, often resulting in a reduction of energy usage and waste generation.

Continuous flow reactors allow for the manufacture of various different products through the adjustment of process operating conditions and chemical reagents. To achieve this, these reactors enable access to real-time measurements from various process sensors and in-line PAT (Process Analytical Technology) thereby facilitating process adjustment through micro pumps and regulating valves etc. This in turn allows for precise control and optimisation of the integrated process to eliminate the effects of raw material variability and maintain the prescribed product quality and yield. Furthermore, using the process/sensor data within a multivariate monitor can help to detect faulty operation of the reactor/reaction in real-time (Mercer, Mack, Tahir, & Lovett, 2018).

Despite the aforementioned benefits, flow reactors present a challenge from a control perspective. The process is of a multivariate nature, involves long time delays as well as various system constraints — for example pump speeds and heater temperature limits. This makes the implementation of classical control techniques, such as the PID algorithm, mostly impractical. On the other hand, advanced control techniques, such as model predictive control (MPC), are particularly well suited to this problem as they explicitly take account of the process constraints and multivariate interactions. However, advanced process control of continuous flow micro-reactors is still a relatively under-examined area. To this end, an MPC scheme along with a process monitor is proposed for the Corning Advanced Flow Reactor (Corning Inc, 2018) which is set up to produce ether product through O-Alkylaktion reaction of Phenol compounds (Carey & Sundberg, 2007; Zani & Colombo, 2012). A calibration model is first developed to predict, in real-time, the ether yield based on the in-line Mid-IR (infra-red) spectra. Then, an MPC scheme is designed and implemented to control the ether yield to the desired setpoint by manipulating process parameters such as pump speeds and reactor temperature. Furthermore, a process monitor based on PCA (principal component analysis) is developed to detect any process/reaction faults, such as poor product yield due to inadequate catalyst.

This paper is organised as follows. Section 2 describes the experimental setup, chemical reaction and software implementation. The proposed advanced control and monitoring scheme is developed in Section 3. The real-time results are presented in Section 4. The conclusion is given in Section 5. A list of acronyms is provided in the Appendix.

* Corresponding author.
E-mail addresses: ftahir@perceptiveapc.com, ftahir49@hotmail.com (F. Tahir).

https://doi.org/10.1016/j.conengprac.2018.06.003
Received 20 March 2018; Received in revised form 3 June 2018; Accepted 4 June 2018

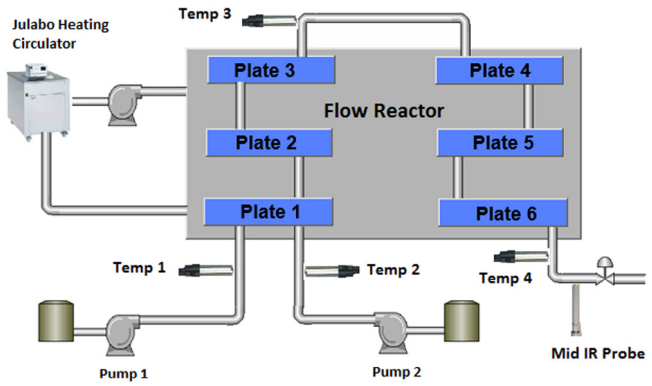

Fig. 1. Block diagram of the experimental setup.

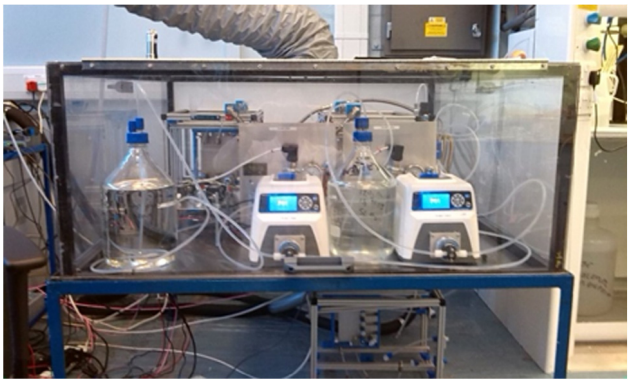

Fig. 2. Experimental setup.

2. Experimental setup

A block diagram of the overall experimental setup is shown in Fig. 1. Two valveless metering pumps (Fluid Metering Inc, 2018) respectively feed the aqueous and organic reagents into the flow reactor which consists of six plates — see Section 2.1 for details. The aqueous reagent consists of Potassium Hydroxide and 4-tetrabutylphenol in water, while the organic reagent consists of Dichloromethane (DCM) with Benzyl Bromide and tetrabutylammonium bromide (TBAB), which serves as a phase transfer catalyst. The TBAB plays a vital role as without it little or no reaction occurs due to the biphasic nature of the reaction and the immiscible reagents. The concentration of TBAB therefore directly effects the reaction rate and product yield. Further details around the precise reaction chemistry can be found in Zani and Colombo (2012).

A heating circulator was used to control the temperature of each reactor plate (Julabo GmbH, 2018). As shown in Fig. 1, four thermocouples were commissioned to measure the temperature of the inlet reagent streams (Temp 1 and 2), mid-reactor solution (Temp 3) and the outlet product (Temp 4), respectively. An inline mid-infrared (FT-IR) spectrometer probe (Bruker Corporation, 2018) was inserted at the reactor outlet to collect real-time spectra corresponding to the ether product. Fig. 1 also shows a pressure control valve, following the FT-IR probe, which is used to control the back-pressure within the plate reactor. A front-view of the experimental setup is shown in Fig. 2.

2.1. Corning advanced flow reactor

The organic synthesis of ether product was carried out in a continuous manner using the Corning Advanced Flow Micro-Reactor (Corning Inc, 2018). The total volume of the considered reactor – including the 6 plates and associated tubing – was approximately 213 ml. The reactor supports a maximum flow-rate of 70 ml/min, resulting in a residence times of 3 min and above.

Fig. 3. Overview of reactor plates.

An overview of the plate setup is shown in Fig. 3. The internal structure of each plate is designed to maximise the surface-to-volume ratio — see Corning Inc (2018) for further details. This in turn leads to faster reactions times due to efficient heat/mass transfer properties (Fukuyama, Rahman, Sato, & Ryu, 2008; Jähnisch et al., 2004). Furthermore, accurate control of critical process variables, such as temperature, pump speeds and residence time, can result in yield improvements. It is this problem of precise, real-time yield control and robustness against process/material uncertainties which is primarily addressed within the paper.

2.2. PharmaMV software

The proposed advanced control and monitoring scheme was designed and implemented, online, using PharmaMV software package from Perceptive Engineering Limited (Perceptive Engineering Limited, Daresbury, UK, 2018). Communication between the reactor unit, pumps, and temperature probes was set up using an OPC interface (OPC Foundation, 2018). Through this interface, PharmaMV collected all the process data, such as temperatures, and pressures, as well as wrote, in real-time, to various setpoints (SP) such as pump speeds and circulator temperature SP. The in-line FT-IR spectral data was also collected in PharmaMV to enable the designed PLS (partial least squares) calibration model to predict the ether yield. This, in turn, was used within the MPC scheme for real-time control as well as for process monitoring.

3. Design of the advanced process control and monitoring scheme

In this section, the design of the advanced process control and monitoring scheme is presented. As shown in Fig. 4, the proposed scheme consists of an MPC block controlling the real-time reactor ether yield prediction – coming from a PLS calibration model – to a desired setpoint. Furthermore, this yield prediction along with the reactor process data is used within the developed PCA monitor to detect any breakdown in correlations that is indicative of process/reaction faults.

Section 3.1 presents the PLS calibration model design. Then, the proposed MPC controller is described in Section 3.2. Finally, the design of PCA continuous condition monitor is presented in Section 3.3.

3.1. Calibration model design

A PLS-based calibration model was developed to predict the ether yield (wt/wt %) in real-time using the inline FT-IR spectra. In this section, a brief theoretical background to PLS algorithm is presented followed by the design of the calibration model.