

Modelling for Control of Biodiesel Microreactors

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Abstract: The modelling of biodiesel production through transesterification in chemical reactors has largely been studied, but the control of this process in microreactors is not straightforward without a suitable process model. In this paper, a useful mathematical model for control is proposed. The model suitably represents the dynamics of biodiesel properties in a microreactor. By analysing the physical influences of convection, diffusion and chemical kinetics on biodiesel yield, an explicit relationship between feed flows and mixing ratios with biodiesel output concentration is captured for control. The model responses are analysed and compared against a variety of experimental results in existing literature at similar operating conditions. The proposed model shows appropriate responses and opens the possibilities of more accurate model-based control applications such as Model Predictive Control and Internal Model Control.

Keywords: Mathematical Modelling, Identification for Control, Process Control, Industrial Applications, Model-based Control, Microreactor Technology, Chemical Variables Control.

1. INTRODUCTION

1.1 Overview

Biodiesel being an alkyl ester is a useful natural alternative energy source with great environmental benefits. It is one of the end products of transesterification usually in chemical reactors. In the chemical engineering literature, there exist a variety of models which describe the transesterification of triglycerides and alcohol to biodiesel and glycerol. But for control, these available models are insufficient as they contain no explicit representation of a manipulated variable which can be used for control of biodiesel properties. Usually, this discrepancy lies in the intended use of these developed models in these research communities. In fact, this lack of a representative model contributes to the challenge of controlling process variables in industrial setups. Here, with good insights into the physical behaviour of the transesterification process in microreactors, a representative model is developed and tested. We also recommend the manipulated variables required for control.

1.2 Background

Transesterification is a three stage chemical reaction between an alcohol and a triglyceride (Richard et al., 2013; Han et al., 2011; Marchetti et al., 2007) usually in the presence of a catalyst such as potassium hydroxide and sodium hydroxide (Sun et al., 2010). These stages occur

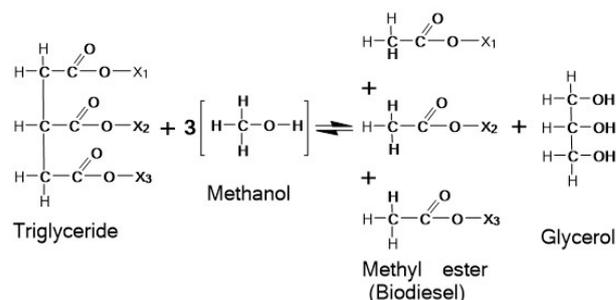


Fig. 1. Chemical equation showing complete transesterification of methanol and triglyceride (from oil)

almost simultaneously during which diglycerides, monoglycerides and glycerol are produced as intermediates in each stage (Richard et al., 2013). Also in parallel and at each stage is the formation of one molecule of an alkyl-ester which is collected as biodiesel (Hoque and Gee, 2012). Stoichiometrically, one molecule of triglyceride from oil and three molecules of alcohol are required to produce one molecule of biodiesel and one molecule of glycerol (see figure 1). This is a reversible reaction which could be favoured in either direction depending on the reacting conditions such as the quality and type of reactants used, the amount and type of catalyst, the reacting temperature, the reactant flow rates and mixing intensity among others (Sun et al., 2010; Richard et al., 2013). Also, it has been shown that an excess of methanol shifts the equilibrium of reaction towards the products, thus favouring the production of biodiesel (Marchetti et al., 2007; Bequette, 2002).

Transesterification generally takes place in chemical reactors. In commercial scales, batch or semi-batch reactors

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are usually used for this process (Costa and Naviera-Cotta, 2019; Han et al., 2011). But the reaction kinetics in such reactors are quite slow as summarised by Santana et al. (2019). In their work, they showed that minutes to hours are required to reach high yields of biodiesel in traditional batch reactors. Microreactors on the other hand, are continuous reactors and come with some advantages. In recent times, an evolving interest in micro scale reactors has opened the possibility of intensifying mass and heat transfer due to increased surface area to volume ratio (interfacial area) of reacting fluids (Richard et al., 2013; Han et al., 2011; McMullen, 2010). This is favoured by the micro size of the reactor geometry and translates to higher efficiency and throughput with shorter reaction times (Tiwari et al., 2018; Madhawan et al., 2017; Han et al., 2011; Santana et al., 2019; Sun et al., 2010). The advantages of microreactors over traditional batch reactors is enormous and have been identified by many other researchers such as Dai et al. (2014); Elkady et al. (2015); Santana et al. (2016) and Rahimi et al. (2014) among others.

In modelling for control, the following steps (as outlined by Seborg et al. (2004)) are recommended: Determine modelling objectives, use and desired accuracy; develop process schematics; outline explicit and unambiguous modelling assumptions; determine representative differential equations; derive appropriate balance equations from physical and chemical properties; analyse equations for solvability and; classify process variables into input, disturbance and manipulated variables if required. In view of this, it is necessary to understand the various influencing factors of transesterification in microreactors.

1.3 Influencing Process Variables

Reaction temperature, feed flow rate, mixing ratio (molar ratio) of alcohol to oil, amount of catalyst and reactor geometry are some key factors that affect the production of biodiesel in microreactors.

Alkali-based catalysts such as sodium hydroxide (NaOH) and potassium hydroxide (KOH) are commonly used in transesterification (Madhawan et al., 2017) due to their relative low costs and high yields under standard operating conditions (Tiwari et al., 2018; Elkady et al., 2015). But they are suited for reactions involving reactants with low free fatty acid (FFA) contents (< 1 wt%) (Tiwari et al., 2018; Dai et al., 2014). Generally, increased amounts of catalysts increase biodiesel yield up to an optimum level after which alkaline catalysts react with FFA in a process called saponification. This inhibits biodiesel production (Guan et al., 2010).

The temperature dependence of transesterification is embedded in the Arrhenius equation given by (Rawlings and Ekerdt, 2002)

$$k = k_o \exp\left(-\frac{E_a}{RT}\right) \quad (1)$$

with k being the rate constant, k_o the pre-exponential factor, E_a the activation energy, R the universal gas constant and T the reaction temperature in Kelvin. This shows a direct proportional dependence of the reaction rate constant on the reaction temperature (given that the reaction temperature is below the boiling point of reacting

alcohol) (Madhawan et al., 2017). Increased temperature also enhances mixing of reactants and hence biodiesel production. This is shown in the experimental results of Santana et al. (2016).

For a fixed reactor volume, the feed flow rates of reactants vary inversely as the residence time. Therefore, an increase in feed flow reduces the residence time which generally results to a decrease in biodiesel yields. The rate of variation of biodiesel yields with feed flows also depends on the kind of reactor as shown in the works of Sun et al. (2010); Rahimi et al. (2014)

One of the most important factors that affect biodiesel production is the alcohol to oil mixing ratio (molar ratio) (Madhawan et al., 2017; Tiwari et al., 2018; Kusdiana and Saka, 2001). Its effect on the biodiesel production in microreactors varies with the operating condition. Generally, the production of biodiesel is favoured with an increase in mixing ratio up to an optimum value after which, further increase causes emulsification (Tiwari et al., 2018). Emulsification favours the reverse reaction and makes glycerol difficult to recover from the reactor products (Madhawan et al., 2017; Kusdiana and Saka, 2001).

Aside the chemical kinetics, convection and diffusion also affect biodiesel production in microreactors. Their effect is described by the convection-diffusion equation below.

$$\frac{\partial C_{ij}}{\partial t} + \bar{u} \cdot \nabla C_{ij} - \nabla \cdot (D_{xy} \nabla C_{ij}) \pm R_j = 0. \quad (2)$$

Here, C_{ij} is the concentration of component i in the j th phase, D_{xy} is the diffusion coefficient of component x in solvent y , \bar{u} is the fluid velocity and R_j represents the stoichiometric reaction rate of reactants in the j th phase. Convection describes mixing of reacting particles while diffusion describes mass transfer by diffusion across reacting phases. Richard et al. (2013) developed a model for transesterification in a microreactor with a well controlled laminar flow and negligible convection. Han et al. (2011) presented a numerical simulation of the convection-diffusion equation using finite element discretisation methods, but did not present an explicit model for control. Many others only focus on reaction kinetics without emphasis on convection or diffusion.

2. MODELLING OF BIODIESEL PRODUCTION

2.1 Process Schematics

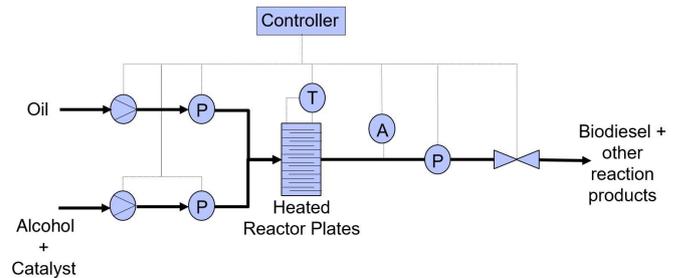


Fig. 2. Schematic representation of microreactor

The modelled process is assumed to have inlet pumps with precise flow controls, which pump alcohol and oil at

desired flow rates into the microreactor. The microreactor is fitted with tempering and reacting plates responsible for heating and housing reacting fluids respectively. From figure 2, P, T and A represent sensors for measuring pressure, temperature and amount of substances respectively, which is required for feedback control. The system is also equipped with an independent temperature control which makes isothermal reaction possible.

2.2 Assumptions and Hypothesis

The following assumptions are hugely based on the works of Richard et al. (2013) and are necessary to ensure accurate modelling.

- (1) The reaction is isothermal at an independently controlled temperature of 65 °C.
- (2) A bubble flow regime is assumed within the microreactor. The oil is dispersed in the stream of alcohol. Therefore the complete effects of convection and diffusion are accounted for.
- (3) The reaction is two-phased.
- (4) Pure methanol and oil are assumed. This is seldom the case in practice, thus for control, any variation from the pure states of reactants are considered disturbances.
- (5) No effects of FFA.
- (6) The reaction is homogeneous.
- (7) The fluids are in-compressible Newtonian liquids, therefore pressure is evenly distributed in the reactor. As such, total flow rate is constant throughout the reactor.
- (8) The reaction takes place in the methanol phase due to the solubility of catalyst in it.

2.3 Model Equations

Each element of the complete convection-diffusion equation is analysed with emphasis on its physical meaning. The stoichiometric and diffusion effects are completely captured in the model proposed by Richard et al. (2013). This is adapted in this paper. Convection, given by $\bar{u} \cdot \nabla C$ in equation 2 describes mass transfer by physical mixing in the reactor. This can be likened to the same mixing effects in traditional continuous stirred tank reactors (CSTR), but in a smaller scale. As such, this will be modelled as a CSTR as adapted from (Bequette, 2002) but modified in this paper. For each phase, this will be given by

$$\bar{u} \cdot \nabla C_i = \alpha \frac{Q_i}{V} (C_{fi} - C_i). \quad (3)$$

Here, C_{fi} and Q_i are the feed concentration and flow rate of component i respectively. C_i is the concentration of component i at time t , V is the volume of the reactor and α is a dimensionless constant unique to each reactor. The combined term $\alpha \frac{Q_i}{V}$ is equivalent to the dilution rate in a CSTR. With respect to assumption (6) and (7), the flow rate of each reacting component in each phase is uniform and equivalent to the combined flow rates of alcohol (Q_A) and oil (Q_O) at the reactor feed. i.e.

$$Q_i = Q = Q_O + Q_A. \quad (4)$$

From these assumptions and physical insights, the complete model equations which are representative of the

combined effects of convection, diffusion and the kinetics of transesterification are therefore given by the nine differential equations in equations 5.

$$\begin{aligned} \dot{x}_1 &= -\alpha \frac{Q}{V} x_1 - k_1 x_1 x_8 q + k_{-1} x_3 x_9 q + K_{tg} (x_2 - x_1) a \\ \dot{x}_2 &= \alpha \frac{Q}{V} (F_O - x_2) - K_{tg} (x_2 - x_1) a \\ \dot{x}_3 &= -\alpha \frac{Q}{V} x_3 + k_1 x_1 x_8 q - k_{-1} x_3 x_9 q - k_2 x_3 x_8 q \\ &\quad + k_{-2} x_5 x_9 q + K_{dg} (x_4 - x_3) a \\ \dot{x}_4 &= -\alpha \frac{Q}{V} x_4 - K_{dg} (x_4 - x_3) a \\ \dot{x}_5 &= -\alpha \frac{Q}{V} x_5 + k_2 x_3 x_8 q - k_{-2} x_5 x_9 q - k_3 x_5 x_8 q \\ &\quad + k_{-3} x_7 x_9 q + K_{mg} (x_6 - x_5) a \\ \dot{x}_6 &= -\alpha \frac{Q}{V} x_6 - K_{mg} (x_6 - x_5) a \\ \dot{x}_7 &= -\alpha \frac{Q}{V} x_7 + k_3 x_5 x_8 q - k_{-3} x_7 x_9 q \\ \dot{x}_8 &= \alpha \frac{Q}{V} (F_A - x_8) - k_1 x_1 x_8 q + k_{-1} x_3 x_9 q - k_2 x_3 x_8 q \\ &\quad + k_{-2} x_5 x_9 q - k_3 x_5 x_8 q + k_{-3} x_7 x_9 q \\ \dot{x}_9 &= -\alpha \frac{Q}{V} x_9 + k_1 x_1 x_8 q - k_{-1} x_3 x_9 q + k_2 x_3 x_8 q \\ &\quad - k_{-2} x_5 x_9 q + k_3 x_5 x_8 q - k_{-3} x_7 x_9 q \end{aligned} \quad (5)$$

For convenience, we have written $x(t)_j$ as x_j . In equation 5, for $i = 1, 2, 3, -1, -2$ and -3 , k_i represents the reaction rate constants. K_{tg} , K_{dg} and K_{mg} represent the mass transfer coefficients. q represents the catalyst concentration and a represents the interfacial area available for mass transfer. F_O and F_A represent feed concentrations of oil and alcohol respectively and all constants are well defined with appropriate units. The state descriptions are given in table 1.

Table 1. Model state descriptions

State	Description
x_1	Triglyceride concentration in alcohol phase
x_2	Triglyceride concentration in oil phase
x_3	Diglyceride concentration in alcohol phase
x_4	Diglyceride concentration in oil phase
x_5	Mono-glyceride concentration in alcohol phase
x_6	Mono-glyceride concentration in oil phase
x_7	Glycerol concentration
x_8	Alcohol concentration
x_9	Biodiesel concentration

The states are written for convenience as

$$x = [x_1 \ x_2 \ x_3 \ x_4 \ x_5 \ x_6 \ x_7 \ x_8 \ x_9]^T. \quad (6)$$

By performing steady state analysis, i.e. when $\dot{x} = 0$ and solving equation 5, we derive the expression

$$F_O + F_A = \sum_{i=1}^9 x_i. \quad (7)$$

This means that the sum of reacting component concentrations at any time is constant and equivalent to the total feed concentrations of alcohol and oil.

An expression for the interfacial area was derived by Richard et al. (2013) for an annular flow regime. But it

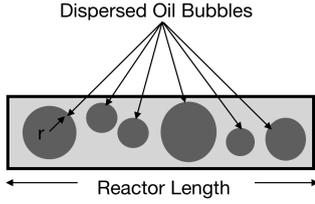


Fig. 3. Schematic representation of bubbly flow in a microreactor channel

is nontrivial to derive same for bubbly flow. Though it is practical to expect varying sizes of oil bubbles in the flow (see figure 3), we assume equal average radius r of bubbles in the flow for simplicity. Thus being spheres, the “interfacial area concentration” as coined by Kataoka et al. (2012) throughout the reactor length is given by

$$a = \frac{4\pi r^2 N}{V} = \frac{3\gamma}{r}. \quad (8)$$

Here, N is the number of bubbles in the reactor volume and γ is the void fraction of bubbles. We will liken this to the mixing ratio of alcohol to oil. For incompressible Newtonian fluids in a microreactor with cross-sectional radius R , an expression for the volumetric flow rates of reacting phases can be derived by integrating the velocity profiles in the microreactor given by equation 9 (Richard et al., 2013).

$$\begin{aligned} Q_O &= -\frac{\pi r^2}{4} \left(\frac{r^2}{2\eta_o} + \frac{R^2 - r^2}{\eta_o} \right) \partial_z P \\ Q_A &= -\frac{\pi}{8\eta_A} (R^2 - r^2)^2 \partial_z P \end{aligned} \quad (9)$$

By solving these equations, an expression for the oil radius in terms of feed flows is derived as

$$r^2 = R^2 - R^2 \left(\frac{\frac{Q_A \eta_A}{Q_O \eta_O}}{1 + \frac{Q_A \eta_A}{Q_O \eta_O}} \right)^{\frac{1}{2}} \quad (10)$$

with

$$\frac{Q_A}{Q_O} = M.R. \quad (11)$$

Here, P is the tube pressure in the flow direction and η_A and η_O are the viscosities of alcohol and oil respectively. $M.R.$ is the volumetric mixing ratio which is derived from the stoichiometric molar ratio using equation 12 below.

$$M.R. = \text{Molar Ratio} \times \frac{\text{Molar Volume}_{Alcohol}}{\text{Molar Volume}_{Oil}} \quad (12)$$

For instance, a 6 : 1 molar ratio¹ of ethanol to sunflower oil is equivalent to a mixing ratio of about 0.3672 : 1.

From the assumptions mentioned earlier, and assuming a fixed catalyst concentration, the useful input variables u_1 and u_2 for control are the total flow Q and the volumetric mixing ratio $M.R.$ respectively. In this report, the biodiesel content (y), being the model output, is given by the expression²

$$y = \frac{x_9 \times 100\%}{3(x_1 + x_2) + 2(x_3 + x_4) + (x_5 + x_6) + x_9} \quad (13)$$

¹ The stoichiometric molar ratio represents the amount of alcohol in *mol* required for transesterification with 1 *mol* of oil

² Adapted from Richard et al. (2013) but assuming no contributions of FFA in the model

Finally, because of the reactor tube length, there is an embedded time delay in the process. At the reactor outlet, measurements can only be acquired after a given time, equivalent to the residence time (τ) in the reactor. The residence time, expressed as a function of feed flow is given by (Nemethné Sívagó and Benké, 2014)

$$\tau = \frac{V}{Q}. \quad (14)$$

We therefore represent the process inputs as

$$\bar{u} = \begin{bmatrix} \bar{u}_1 \\ \bar{u}_2 \end{bmatrix} = \begin{bmatrix} u_1(t - \tau) \\ u_2(t - \tau) \end{bmatrix} \quad (15)$$

and the complete model for control is represented by the nonlinear state space equations below

$$\begin{aligned} \dot{x} &= f(x, \bar{u}) \\ y &= h(x, \bar{u}) \end{aligned} \quad (16)$$

with $f(x, \bar{u})$ given by equation 5 and $h(x, \bar{u})$ given by equation 13.

3. RESULTS AND DISCUSSIONS

We adopt same operating conditions as Richard et al. (2013) for the transesterification of sunflower oil and ethanol at a simulated temperature of 65°C but assuming bubbly flow. As such, the model parameters summarised in table 2 are used. We simulate for a microreactor with cross sectional radius of 254 μm , length of 2 m, $\alpha = 1$ and $\gamma = M.R.$

Table 2. Simulation model parameters

Term	Description	Value	Unit
k_1	Reaction rate constant	1.20×10^{-1}	$\text{L}^2 \text{mol}^2 \text{s}^{-1}$
k_2	Reaction rate constant	8.65×10^{-11}	$\text{L}^2 \text{mol}^2 \text{s}^{-1}$
k_3	Reaction rate constant	1.50×10^{-1}	$\text{L}^2 \text{mol}^2 \text{s}^{-1}$
k_{-1}	Reaction rate constant	3.00×10^{-2}	$\text{L}^2 \text{mol}^2 \text{s}^{-1}$
k_{-2}	Reaction rate constant	5.00×10^{-2}	$\text{L}^2 \text{mol}^2 \text{s}^{-1}$
k_{-3}	Reaction rate constant	1.20×10^{-2}	$\text{L}^2 \text{mol}^2 \text{s}^{-1}$
K_{tg}	Mass transfer coefficient	1.50×10^{-6}	ms^{-1}
K_{dg}	Mass transfer coefficient	1.70×10^{-5}	ms^{-1}
K_{mg}	Mass transfer coefficient	1.50×10^{-6}	ms^{-1}

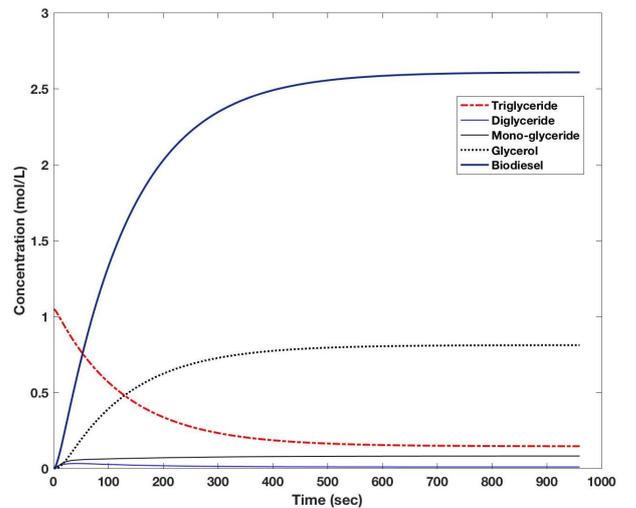


Fig. 4. Propagation of reactants in the transesterification of sunflower oil and ethanol at a molar ratio of 6:1

The evolution of reactants and intermediate products to form ethyl-ester (biodiesel) is shown in figure 4. This is consistent with literature with an approximate 3 : 1 ratio of ethyl-ester to glycerol. It is also verified by similar experimental results of Richard et al. (2013); Han et al. (2011) and Marchetti et al. (2007).

The influence of the mixing ratio is simulated and shown in figures 5 and 6. These are consistent with literature and experimental results of Richard et al. (2013) and Kusdiana and Saka (2001), though the later investigated the transesterification of rapeseed oil and methanol at a much higher temperature.

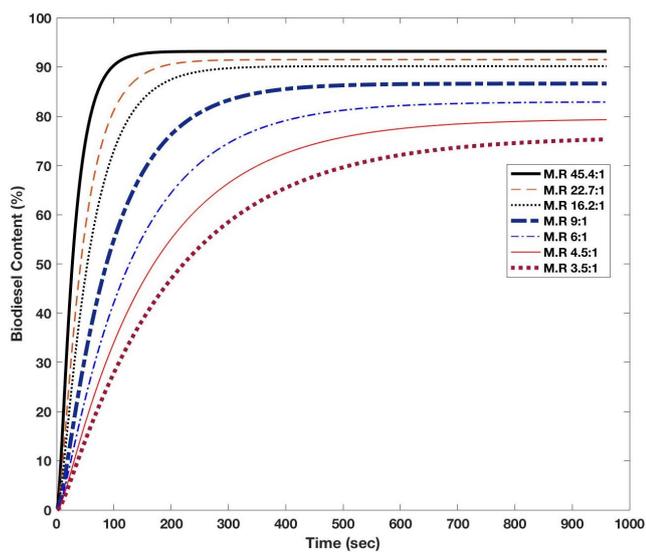


Fig. 5. Biodiesel yields at different molar ratios of methanol to oil

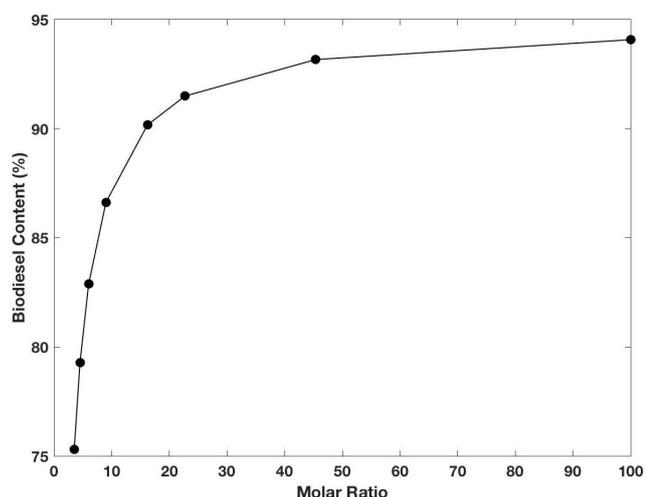


Fig. 6. Influence of molar ratio on biodiesel content

The model is able to capture “containment” in the reactor (Richard et al., 2013) which explains the phenomenon of faster initial kinetics for higher molar ratios as shown in figure 5. However, the effect of saponification at higher molar ratios is not captured by the model. This is seen in figure 6 where the biodiesel content continues to increase even at an impractical high molar ratio. It is therefore recommended to treat this variable as a constrained input

with the upper bound being a certain optimum value beyond which, saponification is bound to begin in the reaction.

The total flow rate on the other hand shows an expected effect on biodiesel content which is consistent with the experimental results of Marchetti et al. (2007) and Rahimi et al. (2014). An increase in flow rate reduces residence time, which reduces biodiesel content. This is shown in figure 7. For control, the flow is also constrained by the feed pumps which can only pump up to a certain flow rate. Therefore, this is treated as a constrained input.

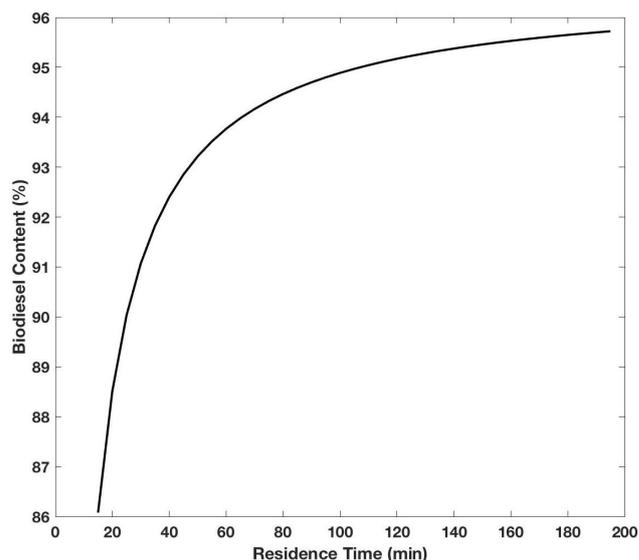


Fig. 7. Influence of residence time on biodiesel yield at a fixed molar ratio of 9:1 of methanol to oil

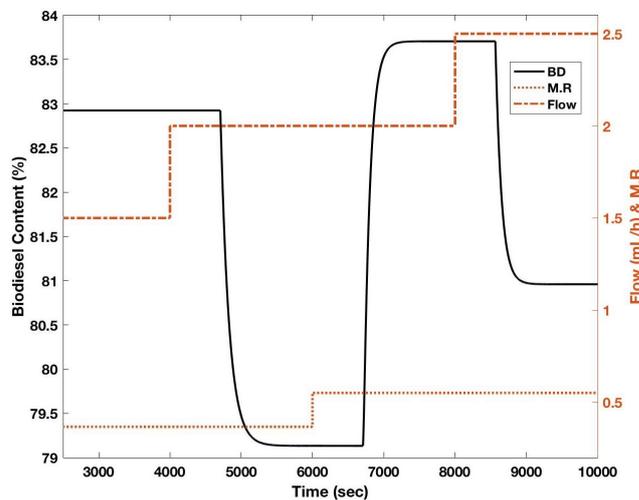


Fig. 8. Model response to step changes in total flow and mixing ratio. Biodiesel is abbreviated as BD in the figure.

A simulation of the model response to step changes in the mixing ratio and total flow is shown in figure 8. This is representative of a simple first order response with time delay, typical of chemical processes. At 4000 sec, a step change in the feed flow (u_1) is implemented from 1.5 mL/h to 2 mL/h. This causes a decrease in the biodiesel content after a delay of 706 sec, equivalent to the residence time at

1.5 mL/h. At the same flow rate, similar time delay is seen after a step change in the mixing ratio at 6000 sec, which shows an increase in biodiesel content. A step change in the flow rate to a higher value of 2.5 mL/h at 8000 sec shows a shorter time delay of about 565 sec, equivalent to a shorter residence time. This translates to a reduction in biodiesel content as expected. The model, being able to represent the behaviour of transesterification in a microreactor is thus a suitable model for control. With this model, well known control methods such as the Proportional Integral and Derivative (PID) control, Model Predictive Control (MPC) and Internal Model Control (IMC) can be applied depending on the desired control objective.

4. CONCLUSION AND FUTURE WORK

In this paper, a model for control is developed, which represents the response of biodiesel content in a microreactor to feed flow and mixing ratio of reactants. The model shows good responses consistent with experimental results of other known researchers. The model is able to capture containment in microreactors as well as the ideal effects of feed flows on biodiesel content, but it is unable to capture saponification at high molar ratios of alcohol to oil in the reaction. As such, it is recommended to treat the feed flow and mixing ratio inputs as constrained variables to account for feed pumps and saponification.

Being a novel attempt to model the complete behaviour of transesterification for control, a wide range of further research works are available. This includes well planned experiments to investigate the model performance across a wide range of flow regimes in the reactor. This will open the possibility of developing a universal model which would be representative of any microreactor given any operating condition and flow regime. In view of this, we have embarked on an experimental study of a microreactor test rig. Here we study the reactor properties in different residence time ranges using an empirical modelling approach. Specifically, System Identification techniques are being employed to determine an approximate model from input-output data.

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