

## On-line calibration of spectroscopic sensors based on state observers

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**Abstract:** Spectroscopic sensors provide on-line information about process variables and they have been widely used for monitoring and control. These sensors measure the spectral responses at a large number of wavelengths correlated with the process variables of interest. However the spectral measurement can also be affected by external factors such as changes in temperature. In order to estimate the process variables from the acquired spectrum it is necessary the use multivariate calibration methods. Additive effects of external factors can be easily compensated by standard calibration methods, but multiplicative effects require complex off-line calibration procedures. This work, shows that this problem can be modeled by a non-linear state space equation. In addition, it also proposes an on-line calibration method based on a state observer for compensating multiplicative effects and at the same time estimating the desired process variable from the spectrum. The convergence of the observer requires a uniform observability condition to be satisfied. Simulation results obtained by using a spectral sensor for monitoring a mixing process under time-varying temperature show the main features and potential of the proposed approach. More complex spectral models for modeling the effect of temperature and other variables can be considered and included in the proposed framework.

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### 1. INTRODUCTION

The advances in optoelectronics have increase the availability of low cost spectral sensors with high resolution for a wide range of applications. Hyperspectral cameras and spectrometers are just a few examples of these type of sensors that have been applied at industrial level to monitor thermal and chemical processes. Spectral based sensors provide information about the radiation emitted, reflected or transmitted by a sample at a given wavelength. Typical spectral sensors are the Ultraviolet-Visible (UV-VIS) and Near-infrared (NIR) spectrometers. These sensors are routinely used in the industry for chemical analysis and process monitoring, (Bakeev., 2005); Chen et al. (2011). In order to estimate process variables from the spectral information, it is necessary to calibrate the sensor. There are many linear and non-linear calibration methods. See for instance the survey papers (Geladi, 2003), (Geladi et al., 2004), (Sekulic et al., 1993) and the book written my Marks and Workman (Mark and Workman Jr., 2007). All these calibration methods are off-line and require a set of data for calibration. The information provided by these sensors can be used for process control Chen et al. (2011). The use of model predictive control and Principal Component Analysis for extracting the information from the spectrum has been proposed in (Lin et al., 2009). In (Sbarbaro, 2014) two approaches for integrating spectral sensors into control structures are described. In order to

deal with nonlinearities, neural networks models has been also proposed in (Puebla, 1994).

Spectroscopic sensor signals can be affected by external factor such as temperature, viscosity and optical effects. These effects are in general nonlinear. In (Wulfert et al., 1998) different local and global Partial Least-Squares calibration strategies are analyzed with respect to changes in temperature. The results show that global models are considerably better but at expense of sensitivity. In addition, this work also points out that high sensitivity calibration models can be built if the temperature effect are explicitly modeled. Pre-processing methods, such as Extended Multiplicative Signal Correction, can be used prior to the use of linear calibration methods to eliminate the influence of these effect on the analyzed spectrum, (Martens and Stark, 1991). Recently additional process analytical approaches have been designed to cope with changes in temperature. Extended loading space standardization and systematic error prediction errors are two representative examples (Chen et al., 2004) (Chen et al., 2011). Most of these approaches are off-line and require informative calibration data sets.

This work proposes the use of observers for estimating the concentrations of components based on spectrum measurements, and at the same time taking into account the temperature variations of the sample. A mixing problem, similar to the one described in (Johansen and Sbarbaro,

2005), is considered to illustrate the application of the proposed approach. It is demonstrated that the calibration model of this problem can be described by a state-space representation with a nonlinear output map. Observer design for this structure has been widely studied. For instance, in (Glaria et al., 2012) the design of observers with nonlinear output maps satisfying some Liptchitz conditions have been analyzed and a design procedure based on LMIs is provided. In (Martino et al., 2004) the nonlinear output map is modeled as polynomial function, and by a simple transformation the system is represented as a time varying linear system. Thus, the observer gain is given in terms of a Riccati differential equation; which can be solved on-line.

To the best of our knowledge, the proposed approach is one of the few works addressing chemical analytical problems from a control engineering perspective.

This work is organized as follows: Section 2 presents the model describing the process dynamic and the spectroscopic sensors. Section 3 introduces an on-line calibration strategy based on the information provided by the spectral sensor. In section 4 the convergence of the observer is analyzed. Section 5 illustrates, by means of simulation examples, the main characteristics of the proposed approach. Finally, in section 6 some conclusions and future works are given.

## 2. PROCESS AND SENSOR MODELING

Let us consider a process represented by a linear model

$$\frac{dx_j}{dt} = \sum_{j=1}^n a_{ij}x_j(t) + \sum_{j=1}^m b_{ij}u_j(t), \quad m \leq n \quad (1)$$

$$w_i(t) = \sum_{j=1}^n c_{ij}x_j(t), \quad i = 1, \dots, m$$

where  $x_i$  are state variables and  $w_i$  are concentrations. The coefficients  $c_{ij}$ ,  $a_{ij}$  and  $b_{ij}$  define the dynamical behavior of the system. The manipulated variables  $u_1(t), \dots, u_m(t)$  can change the concentrations  $w_i$  to satisfy some control objectives.

According to the Beer's law, the absorbance spectra of the mixture can be expressed as linear combinations of the absorbance contributions of all the constituents. Thus, the relationship between the sensor output  $y(t, \lambda) \in R$  can be represented in terms of the following model:

$$y(t, \lambda) = z(t) \sum_{i=1}^m w_i(t) \phi_i(\lambda) + d(\lambda), \quad \lambda \in \Lambda, t \geq 0 \quad (2)$$

where the variable  $\lambda \in \Lambda$  represents the spectral coordinate as a wavelength value,  $\Lambda$  the wavelength interval,  $\phi_i(\lambda)$  is the absorbance of the  $i$ -th element,  $w_i(t)$  is the corresponding concentration,  $z(t)$  represents a multiplicative effect and  $d(\lambda)$  a baseline offset. This offset refers to the spectral measured values when there is no light. This value can be slightly different for each wavelength. However, in practice a single average value is used to compensate its effect. Since this effect is sensor specific, the offset compensation is performed previously to any process measurement.

In this work, a second order polynomial in terms of the temperature of the sample is considered; i.e.

$$z(t) = \alpha_1 + \alpha_2 T(t) + \alpha_3 T^2(t) \quad (3)$$

where  $\alpha_i$  are calibrating factors.

Equations (1) and (2) can be written in condensed form as

$$\frac{d\mathbf{x}}{dt} = \mathbf{A}\mathbf{x}(t) + \mathbf{B}\mathbf{u}(t) \quad (4)$$

$$\mathbf{w}(t) = \mathbf{C}\mathbf{x}(t)$$

$$y(t, \lambda) = \sum_{i=1}^3 \alpha_i T(t)^i \Phi(\lambda) \mathbf{w}(t) + d(\lambda)$$

where  $\mathbf{u} = [u_1, \dots, u_m]^T$ ,  $\mathbf{x} = [x_1, \dots, x_n]^T$ ,  $\mathbf{w} = [w_1, \dots, w_m]^T$  and  $\Phi(\lambda) = [\phi_1(\lambda) \dots \phi_m(\lambda)]$  is a vector of absorbance spectra. Matrices  $\mathbf{A} \in R^{n \times n}$ ,  $\mathbf{B} \in R^m$ , and  $\mathbf{C} \in R^{m \times n}$  define the dynamic characteristic of the system.

*Remark 1.* More complex sensor models can also be considered in the same framework. For instance it can be assumed that each absorbance spectrum depends on the temperature; i.e.  $\phi_i(\lambda, T(t))$ . As suggested in Chen et al. (2011) the following parametrization can be used to model this dependency

$$\phi_i(\lambda, T(t)) = \phi_{i0}(\lambda) + \phi_{i1}(\lambda)T(t) + \phi_{i2}(\lambda)T^2(t) \quad (5)$$

In this model, each wavelength dependent function  $\phi_{ij}(\lambda)$ ,  $j = 0, 1, 2$  can be modeled by a parametric model, in terms of basis function, as follows:

$$\phi_{ij}(\lambda, T(t)) = \sum_{k=1}^{l_j} c_{jk} \psi_{jk}(\lambda) \quad (6)$$

where  $l_j$  is the number of basis function  $\psi_{jk}(\lambda)$  and  $c_{jk}$  represents a set of parameters to be identified. The basis functions can be predefined by taking into account mathematical considerations and the characteristic of the absorbance spectrum; see for instance Dubrovkin (2018), Kärnä et al. (2008).

## 3. ON-LINE CALIBRATION METHOD

Calibration methods provide estimates of the concentrations given the measured absorbance spectra. Thus the calibration problem addressed in this work can be defined as follows:

Given measurements of  $\mathbf{u}(t)$ ,  $y(t, \lambda)$  and  $T(t)$  estimate the concentrations  $\mathbf{w}(t)$  and the calibration factors  $\alpha_i$ ,  $i = 1, 2, 3$ .

In order to estimate the concentrations and the unknown parameters, the system equations are written in terms of a new variable  $\mathbf{q}_i(t) = \alpha_i \mathbf{x}(t)$ , as in Martino et al. (2004),

$$\frac{d\mathbf{q}_i}{dt} = \mathbf{A}\mathbf{q}_i(t) + \alpha_i \mathbf{B}\mathbf{u}(t) \quad (7)$$

$$y(t, \lambda) = \sum_{i=1}^3 T^{i-1}(t) \Phi(\lambda) (\mathbf{C}\mathbf{q}_i(t)) + d(\lambda)$$

The full system can then be described by three dynamical equations and an output map.

$$\begin{aligned}
\frac{d\mathbf{x}}{dt} &= \mathbf{A}\mathbf{x}(t) + \mathbf{B}\mathbf{u}(t) \\
\frac{d\alpha_i}{dt} &= 0, \quad i = 1, 2, 3 \\
\frac{d\mathbf{q}_i}{dt} &= \mathbf{A}\mathbf{q}_i(t) + \alpha_i\mathbf{B}\mathbf{u}(t), \quad i = 1, 2, 3 \\
y(t, \lambda) &= \sum_{i=1}^3 T^{i-1}(t)\Phi(\lambda)\mathbf{C}\mathbf{q}_i(t) + d(\lambda)
\end{aligned} \tag{8}$$

By defining  $\mathbf{p} = [\mathbf{x} \ \mathbf{q}_1 \ \mathbf{q}_2 \ \mathbf{q}_3 \ \alpha_1 \ \alpha_2 \ \alpha_3]^T$  the system equations (8) can be written as

$$\begin{aligned}
\frac{d\mathbf{p}}{dt} &= \mathbf{A}_p(t)\mathbf{p}(t) + \mathbf{B}_p\mathbf{u}(t) \\
y(t, \lambda) &= \mathbf{C}_p(t, \lambda)\mathbf{p}(t) + d(\lambda)
\end{aligned} \tag{9}$$

where

$$\mathbf{A}_p(t) = \begin{bmatrix} \mathbf{A} & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & \mathbf{A} & 0 & 0 & \mathbf{B}\mathbf{u}(t) & 0 & 0 \\ 0 & 0 & \mathbf{A} & 0 & 0 & \mathbf{B}\mathbf{u}(t) & 0 \\ 0 & 0 & 0 & \mathbf{A} & 0 & 0 & \mathbf{B}\mathbf{u}(t) \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 \end{bmatrix} \mathbf{B}_p = \begin{bmatrix} \mathbf{B} \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \end{bmatrix} \tag{10}$$

$$\mathbf{C}_p(t, \lambda) = [0 \ \Phi(\lambda)\mathbf{C} \ T(t)\Phi(\lambda)\mathbf{C} \ T^2(t)\Phi(\lambda)\mathbf{C} \ 0 \ 0 \ 0] \tag{11}$$

Thus an observer can be built to estimate the extended state of the time-varying system (9). The proposed time-varying observer has the following standard structure:

$$\begin{aligned}
\frac{d\hat{\mathbf{p}}}{dt} &= \mathbf{A}_p(t)\hat{\mathbf{p}}(t) + \mathbf{B}_p\mathbf{u}(t) + \\
&\quad \mathbf{P}(t)\mathbf{C}_p(t, \lambda)(y(t, \lambda) - \hat{y}(t, \lambda)) \\
\hat{y}(t, \lambda) &= \mathbf{C}_p(t, \lambda)\hat{\mathbf{p}}(t) + d(\lambda) \\
\frac{d\mathbf{P}}{dt} &= (\mathbf{A}_p(t) - \mathbf{P}(t)\mathbf{C}_p(t, \lambda)^T\mathbf{C}_p(t, \lambda))\mathbf{P}(t) + \\
&\quad \mathbf{P}(t)(\mathbf{A}_p - \mathbf{P}(t)\mathbf{C}_p(t, \lambda)^T\mathbf{C}_p(t, \lambda))^T + \mathbf{Q}(t)
\end{aligned} \tag{12}$$

where  $\mathbf{Q}(t) \geq \gamma_0\mathbf{I}$  and  $\mathbf{P}(0)$  are symmetric positive definite matrices.

This observer will converge if sufficient conditions for persistency of excitation are satisfied. This condition depends on the structure of the system and the input signals; i.e.  $\mathbf{u}(t)$  and  $T(t)$ .

*Assumption 1.* The time-varying pair  $(\mathbf{A}_p(t) \ \mathbf{C}_p(t, \lambda))$  and the input variables  $\mathbf{u}(t)$  and  $T(t)$  are such that there exists positive constant  $\gamma_1$ ,  $\gamma_2$  and  $\delta$ , with  $\gamma_1 < \gamma_2$  such that

$$\gamma_1\mathbf{I} \leq \int_t^{t+\delta} e^{\mathbf{A}_p(\tau)}\mathbf{C}_p(\tau, \lambda)^T\mathbf{C}_p(\tau, \lambda)e^{\mathbf{A}_p(\tau)}d\tau \leq \gamma_2\mathbf{I} \tag{14}$$

for all  $t \geq 0$

*Remark 2.* This assumption also implies the uniform observability of the time-varying system (9) and therefore the boundness of  $\mathbf{P}(t)$  is also ensured; i.e. there exist positive constants  $\gamma_3$  and  $\gamma_4$  such that  $\gamma_3\mathbf{I} \leq \mathbf{P}(t) \leq \gamma_4\mathbf{I}$ , Bucy (1972).

#### 4. CONVERGENCE ANALYSIS

The convergence analysis is based on standard Lyapunov arguments and can be summarized in the following theorem.

*Theorem 1.* The observer defined by (12) and (13) is an asymptotic observer for system (9), if the pair  $(\mathbf{A}_p(t), \mathbf{C}(t, \lambda))$  and  $\mathbf{u}(t)$  and  $T(t)$  satisfy Assumption 1.

*Proof 1.* The observation error is defined as  $\mathbf{e}(t) = \mathbf{p}(t) - \hat{\mathbf{p}}(t)$  and error dynamic:

$$\frac{d\mathbf{e}(t)}{dt} = (\mathbf{A}_p(t) - \mathbf{P}(t)\mathbf{C}_p(t, \lambda)^T\mathbf{C}_p(t, \lambda))\mathbf{e}(t) \tag{15}$$

Let  $V(t)$  be

$$V(t) = \mathbf{e}(t)^T\mathbf{P}(t)^{-1}\mathbf{e}(t) \tag{16}$$

and taking the time derivative of  $V(t)$  along the error dynamic trajectory (12)

$$\begin{aligned}
\frac{dV(t)}{dt} &= \frac{d\mathbf{e}(t)}{dt}^T\mathbf{P}(t)^{-1}\mathbf{e}(t) + \mathbf{e}(t)^T\mathbf{P}(t)^{-1}\frac{d\mathbf{e}(t)}{dt} + \\
&\quad \mathbf{e}(t)^T\frac{d\mathbf{P}(t)^{-1}}{dt}\mathbf{e}(t)
\end{aligned} \tag{17}$$

Considering (13) we obtain

$$\begin{aligned}
\frac{dV(t)}{dt} &= \mathbf{e}(t)^T[(\mathbf{A}_p(t) - \mathbf{P}(t)\mathbf{C}_p(t, \lambda)^T\mathbf{C}_p(t, \lambda))^T\mathbf{P}(t)^{-1} + \\
&\quad \mathbf{P}(t)^{-1}(\mathbf{A}_p(t) - \mathbf{P}(t)\mathbf{C}_p(t, \lambda)^T\mathbf{C}_p(t, \lambda)) + \\
&\quad \frac{d\mathbf{P}(t)^{-1}}{dt}]\mathbf{e}(t)
\end{aligned} \tag{18}$$

Using the derivative of the inverse of a matrix; i.e.

$$\frac{d\mathbf{P}(t)^{-1}}{dt} = -\mathbf{P}(t)^{-1}\frac{d\mathbf{P}(t)}{dt}\mathbf{P}(t)^{-1} \tag{19}$$

and replacing in (18)

$$\begin{aligned}
\frac{dV(t)}{dt} &= \mathbf{e}(t)^T\mathbf{P}(t)^{-1}[ \\
&\quad \mathbf{P}(t)(\mathbf{A}_p(t) - \mathbf{P}(t)\mathbf{C}_p(t, \lambda)^T\mathbf{C}_p(t, \lambda))^T + \\
&\quad (\mathbf{A}_p(t) - \mathbf{P}(t)\mathbf{C}_p(t, \lambda)^T\mathbf{C}_p(t, \lambda))\mathbf{P}(t) - \frac{d\mathbf{P}(t)}{dt} \\
&\quad ]\mathbf{P}(t)^{-1}\mathbf{e}(t)
\end{aligned} \tag{20}$$

by using (13) it follows:

$$\frac{dV(t)}{dt} = -\mathbf{e}(t)^T\mathbf{P}(t)^{-1}\mathbf{Q}(t)\mathbf{P}(t)^{-1}\mathbf{e}(t) \tag{21}$$

Considering the bounds on  $\mathbf{P}(t)$  and  $\mathbf{Q}(t)$

$$\frac{dV(t)}{dt} \leq -\frac{\gamma_0}{\gamma_4}V(t) \tag{22}$$

it follows

$$V(t) \leq -e^{-\frac{\gamma_0}{\gamma_4}t}V(0). \tag{23}$$

Using the upper bound of  $\mathbf{P}(t)$  we have that estimation error can be upper bounded by  $V(t)$ ; i.e.

$$\frac{1}{\gamma_4}\mathbf{e}(t)^T\mathbf{e}(t) \leq V(t). \tag{24}$$

Thus, equations (23) and (24) imply the observer asymptotic convergence.

#### 5. SIMULATION RESULTS

In this section, the mixing of two liquid components with different absorbance spectra, as shown in figure 1, illustrates the main ideas presented in this work. The

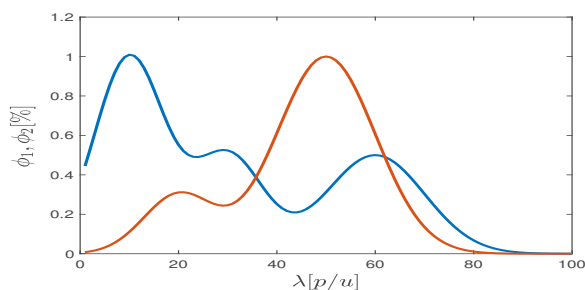


Fig. 1. Spectral response of each component.

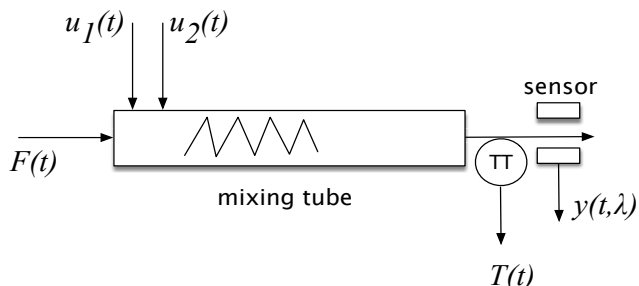


Fig. 2. Mixing process

mixing of these two components is carried out by the aid of a static mixing tube as depicted in figure 2. The resulting spectra is measured by a spectrophotometer at the output of the mixing stage, where  $F(t)$  represents the main stream,  $u_1(t)$  and  $u_2(t)$  are the flow rates of each component,  $T(t)$  is the mixture temperature and  $y(t, \lambda)$  is the measured spectra.

The sensor output is modeled as a linear combination of the absorbance spectra of the components plus the effect of temperature. The dynamics representing the transport and mixing processes are modeled by an over-damped linear system having four real poles. The parameters for the temperature function are  $\alpha_1 = 2$ ,  $\alpha_2 = 0.4$  and  $\alpha_3 = 0.1$ . We also consider that the offset; i.e.  $d(\lambda)$ , has been already compensated.

Step changes in the manipulated variables and a sinusoidal variation in temperature were considered. The time variations of these variables are shown in figure 3. The spectral response is depicted in figure 4, where the effect of changes in the manipulated variables and temperature are mixed.

The matrix  $\mathbf{Q}(t) = 10^{-4}\mathbf{I}$  was chosen to be a constant diagonal matrix,  $\mathbf{P}(0) = 10^3\mathbf{I}$  and the initial states of the observer were zero. The time evolution of the estimated concentrations and the parameters are shown in figure 5. As seen in the graphs, the time variation of the input variables and temperature ensures the convergence of estimated concentrations and parameters converge to the real. The exponential convergence of the state error is depicted in figure 6.

## 6. FINAL REMARKS

Spectroscopic sensors can provide non-contact measurements of key process variables through the spectral response of a given sample. However, the spectral measurements may be affected by both process and sensing condi-

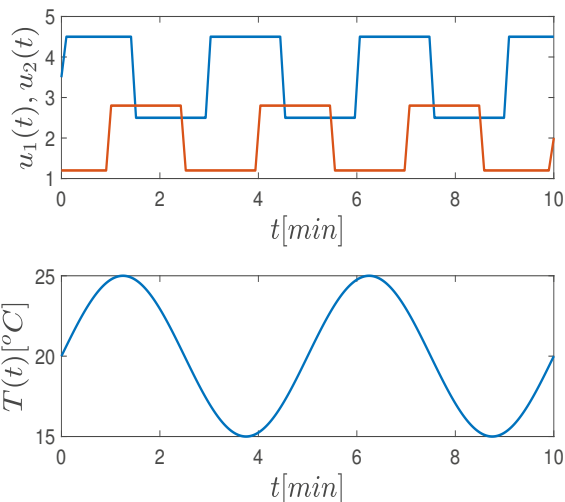


Fig. 3. Input variables and temperature.

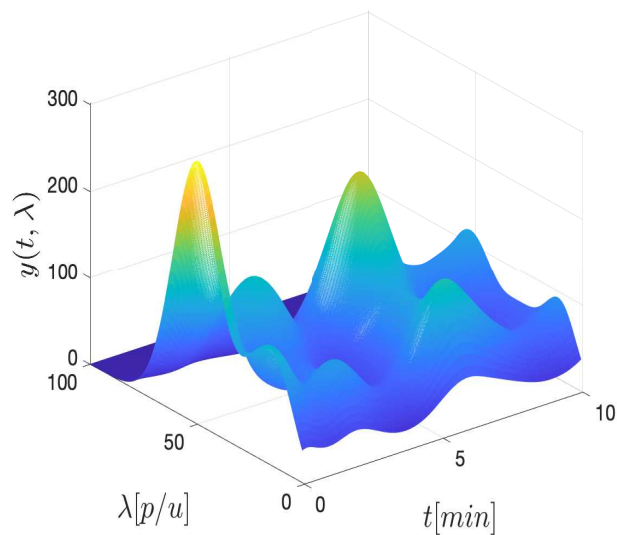


Fig. 4. Spectral response

tions. Linear and nonlinear off-line calibration techniques are used in practice to estimate the process variables. This work has addressed the problem of on-line spectral sensor calibration from a control system perspective. We have shown that the calibration problem can be casted as a state estimation problem associated to a time-varying representation. Conditions for ensuring the asymptotic convergence are based on the notion of uniform observability. This condition also represents a kind of persistent excitation condition depending on the input variables.

The on-line characteristic of the proposed approach opens the possibility of using this spectroscopic sensors where it is difficult to perform extensive testing for building calibration sets. In addition, the state space structure of the observer makes its integration with control structures straightforward.

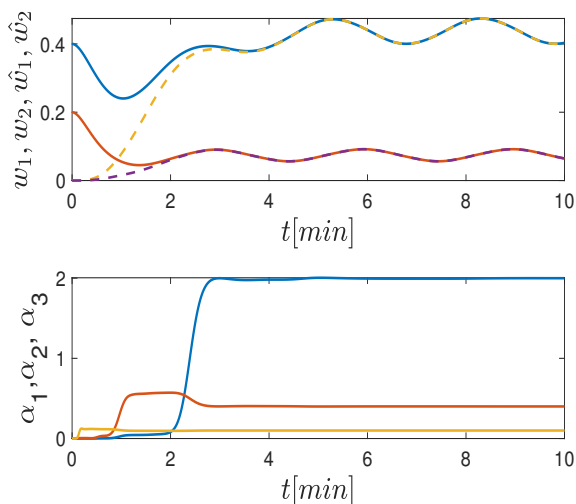


Fig. 5. Estimated concentrations and parameters (blue  $\alpha_1$ , red  $\alpha_2$ , yellow  $\alpha_3$ ).

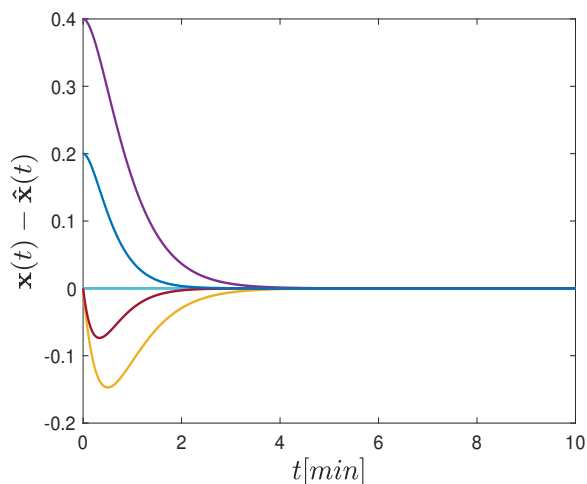


Fig. 6. State error evolution.

Simulation results considering the mixing of two components have shown its feasibility. Further work will consider more complex models for modeling the effect of temperature and other variables. In addition, a discrete-time version will be also implemented in a real-time mixing experiments, for testing the performance under real process conditions.

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