

A Novel Approach of Control Design of the pH in the Neutralization Reactor

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Abstract—This paper deals with the design of a control strategy which will effectively control the level of pH in a neutralization reactor in the whole range of pH. The process consists of a continuously stirred tank, where aqueous solutions of acetic acid and sodium hydroxide are mixed together. The main challenge of a successful control strategy for this process arises mainly from its non-linear behavior. The paper will show how to handle such non-linearity efficiently by introducing an augmented output and an optimization based control. Simulation results will be given to demonstrate the behavior of the proposed control strategy. Comparison between an optimal based controller and a simple PI controller is discussed.

I. INTRODUCTION

Maintaining a specific value of the pH level in chemical and technological processes is vital in order to satisfy quality requirements of final products. This is important in water treatment facilities where the value of pH greatly affects the quality of water purification [1]. It has been reported, that bad pH conditions result in a production of bacteria dangerous to human health. In [2] is described how the pH condition affects the coagulation process in the treatment of reservoir water which has then impact on the purity of the water. Biochemical experiments are another application where regulation of a specific value of pH is needed. Here, unfavorable pH conditions negatively affect entire experiments [3]. pH plays a vital role in medical research and medicine preparation since a vast majority of all drug preparation requires specific pH conditions [4].

There are several scientific papers dealing with pH control or neutralization control. Since the behavior of the neutralization process is highly non-linear, controller design via conventional means has been proven to be insufficient [5]. Therefore, rather than simple PID controllers, advanced control techniques are explored in connection with pH control. Fuzzy controllers [6], [7] are often used for control of a specific range of pH values. Adaptive controllers form the second very broad class of control strategies as these can compensate non-linearities in neutralization process behavior [8], [9]. Generalized Predictive Control based on the experimentally found linear model was proposed to control pH [10]. However, a significant drawback of this work is that non-linearities are not considered at all, hence the tuning of the controller was very conservative in order to avoid large overshoots. This resulted in a considerable rise time even if minor reference changes in pH were considered. In all these papers, the output variable to be controlled was directly pH itself. As mentioned, a drawback of this approach is a very

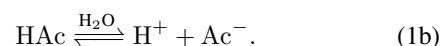
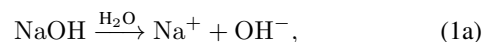
strong non-linear behavior. This problem can be remedied by introducing an augmented output, which more resembles linear behavior. Such augmented output was mentioned in [11], but limited discussion on the control performance was offered.

In this paper, we propose to use an augmented output variable as process variable instead of actual pH measurement. This variable is defined as the difference between the base and acid concentrations. We will show that the relation between input and new process variable is much less non-linear. A model based on this process variable is thus more suited for controller design than the standard approach with pH as the primary output. Furthermore, we achieved an effective control of the pH in the whole range of pH values, which in our case is, from 3 to 13, which compared to the mentioned strategies is a substantial improvement.

II. NEUTRALISATION PROCESS

Neutralisation process considered in this paper consists of a continuously stirred tank of volume $V = 1.5 \text{ dm}^3$. Into this tank are fed two streams of solutes. First stream is the solute of acetic acid (CH_3COOH), second stream is solute of sodium hydroxide (NaOH). By F_1 is denoted flow rate of solute of acetic acid with concentration c_1 . The flow rate of sodium hydroxide with concentration c_2 is denoted as F_2 . The control input to this process is the flow rate of sodium hydroxide. Flow rate of acetic acid is considered as disturbance. Illustrative figure of such chemical process is shown on Fig. 1. In order to simplify notation, acetic acid CH_3COOH is denoted as HAc and acetic anion CH_3COO^- is denoted as Ac^- .

Before the non-linear mathematical model is given, we must first introduce the dissociation reactions of used solutes in water



Reason for this is that from chemical equations (1) directly stems the definition of model states as well as determination of pH value in reaction vessel. Since sodium hydroxide is strong hydroxide, in water this substance dissociates completely (1a), contrary to the acetic acid. Acetic acid is considered as weak acid, thus it dissociates only partially. This means that in water solution there exists both HAc molecules as well as Ac^- molecules.

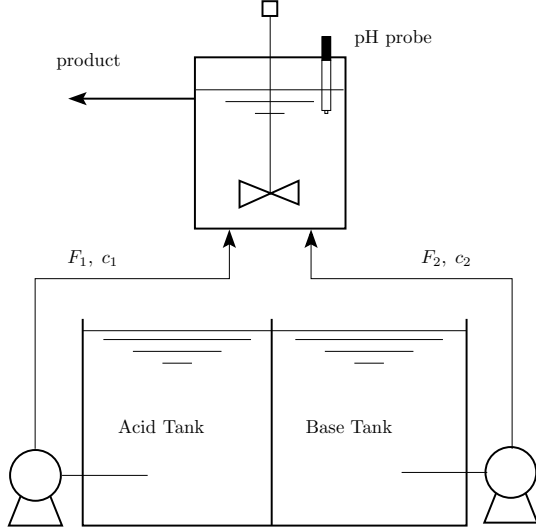


Fig. 1. Illustration of neutralisation reaction vessel

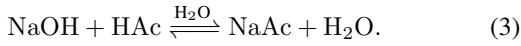
When material balance of entire system is combined with dissociation equations (1), the state space model can be formed

$$V \frac{dx_1}{dt} = F_1 c_1 - (F_1 + F_2) x_1, \quad (2a)$$

$$V \frac{dx_2}{dt} = F_2 c_2 - (F_1 + F_2) x_2. \quad (2b)$$

where x_1 is concentration of acetic acid. Specifically, based on (1b), $x_1 = [\text{HAc}] + [\text{Ac}^-]$. State x_2 is concentration of sodium cation, namely $x_2 = [\text{Na}^+]$.

In the reaction vessel takes place following chemical reaction



Since final product has no electrical charge as well as reactants, in non-linear model derivation must be also considered total electro-neutrality equation, which can be written as follows

$$[\text{Na}^+] + [\text{H}^+] = [\text{OH}^-] + [\text{Ac}^-]. \quad (4)$$

In order to calculate the value of $[\text{H}^+]$ from (4), state variables $x_{1,2}$ must be introduced to the electro-neutrality equation. It can be done using dissociation constants for water k_w , and for acetic acid k_a ,

$$k_w = [\text{OH}^-][\text{H}^+] \approx 10^{-14}, \quad (5a)$$

$$k_a = \frac{[\text{Ac}^-][\text{H}^+]}{[\text{HAc}]} \approx 10^{-5}. \quad (5b)$$

Let us now insert $[\text{HAc}] = x_1 - [\text{Ac}^-]$ into (5b). After fraction manipulations we obtain following expression

$$[\text{Ac}^-] = \frac{x_1 k_a}{k_a + [\text{H}^+]}. \quad (6)$$

By combining together (5a) and (6) and substituting equilibrium concentrations in (4) we obtain

$$x_2 + [\text{H}^+] = \frac{k_w}{[\text{H}^+]} + \frac{x_1 k_a}{k_a + [\text{H}^+]}. \quad (7)$$

After straight forward fraction manipulations (7) can be rewritten into algebraic cubic equation, given as

$$[\text{H}^+]^3 + (x_2 + k_a) [\text{H}^+]^2 + (x_2 k_a - x_1 k_a - k_w) [\text{H}^+] - k_w k_a = 0. \quad (8)$$

Complete non-linear model of the pH neutralisation process is then given by two differential equations (2) and one cubic algebraic equation (8). Finally value of pH is calculated using its definition as $\text{pH} = -\log_{10}([\text{H}^+])$.

III. PROCESS ANALYSIS

A. Non-linear Model Analysis

Before any controller design is considered, analysis of the process is necessary. Especially it is necessary to realize how much the non-linear behaviour reflects on the output. Since most of conventional controllers are based on linear control theory, linearisation of the neutralisation process is desirable. By performing step response (Fig. 3), we can see that the behaviour of pH is non-linear (Fig. 3(a)). There is actually huge discrepancy between response to positive and negative step change in control input. Naturally, control design based on linear control theory will not yield satisfactory results, when used to control the pH levels. To remedy this situation, we are proposing to use equation suggested in [12]. The equation has following form

$$z(\text{pH}, x_1) = \left(10^{\text{pH}-14} - 10^{-\text{pH}} \left(\frac{x_1}{k_a} + 1 \right) \right). \quad (9)$$

Using this equation we augment the system output, which will no longer be value of pH. When such augmented output is considered the response to same step changes resembles linear behaviour. This response is depicted in Fig. 3(b). Note, that all step responses are shown in deviation variables. Specifically we have considered following steady state values of system variables $F_2^s = 7.5 \text{ cm}^3 \text{ s}^{-1}$, for $\text{pH} = 12.4$ and for augmented output $z(12, 0.02) = 0.01$. Flow rate of HAc water solution was kept constant at $F_1^s = 5 \text{ cm}^3 \text{ s}^{-1}$. Acetic acid concentration c_1 was 0.05 mol dm^{-3} and sodium hydroxide concentration was set to $c_2 = 0.05 \text{ mol dm}^{-3}$.

The non-linear nature of the process can be seen in full on so called titration curve (Fig. 2). This figure shows evolution of pH level as a function of base flow rate F_2 . Along the titration curve is displayed dependence of augmented output $z(\text{pH}, x_1)$. Based on Fig. 2 can be concluded, that controlling augmented output $z(\cdot)$ using linear control theory will yield better control performance contrary to controlling directly pH level.

As it will be later shown, introducing augmented input significantly improves the overall control performance. Disadvantage of this approach is, that it requires the value of state x_1 . However, in controlling the real neutralisation process, we do not have access to this measurement. To remedy this situation, we are proposing to use constant value of instead of time varying x_1 . This constant value can be calculated from steady state condition of the model of the neutralisation model (2). Specifically

$$x_1^s = \frac{F_1^s c_1}{F_1^s + F_2^s}, \quad (10)$$

in which all parameters are known in advance. Such approach is supported also by simulation results.

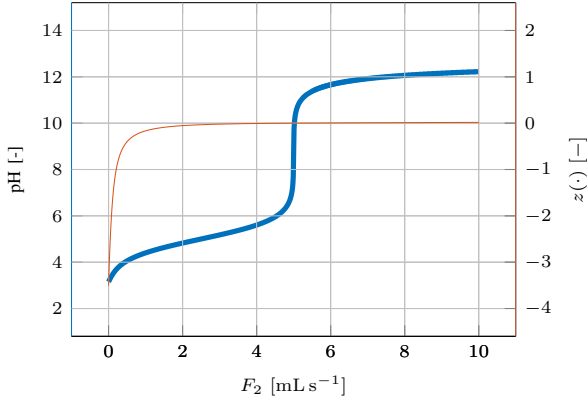


Fig. 2. The blue line represents value of pH as a function of F_2 (titration curve) in steady state. The orange line is the value of augmented output.

B. Linearisation

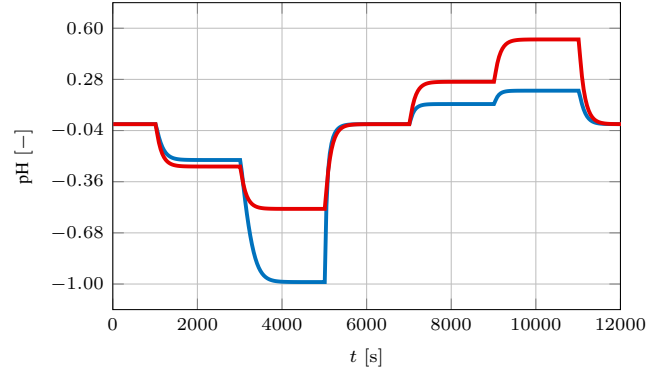
Most controller design approaches are based on linear models. This is the reason for obtaining the linear model for this process as well. There exists several ways how this can be achieved. First approach is to use first order Taylor expansion in order to linearize the analytical model presented in the previous section. Since the non-linear model is given by a set of differential equations as well as one algebraic equation, linearizing the model using this approach may prove troublesome. Second option is to obtain the linear model by system identification using experimental methods. In this work we use *n4sid* method [13].

In both identification procedure, we aim to identify a second order linear state space model in discrete time. Choice of the order of the system stems from the number of differential equations in (2). First, we performed the identification procedure on pair $\{F_2, \text{pH}\}$ (see Fig 3(a)). Secondly, we aimed to identify the response of the non-linear model when the augmented variable $z(\cdot)$ was applied (see Fig 3(b)). In case of the pH identification procedure, the goodness of the obtain model was evaluated as 47%, compared to the second procedure, where the goodness was as high as 85%. This directly shows, that introduction of the augmented output (9) increases the applicability of the linear-based control design strategies.

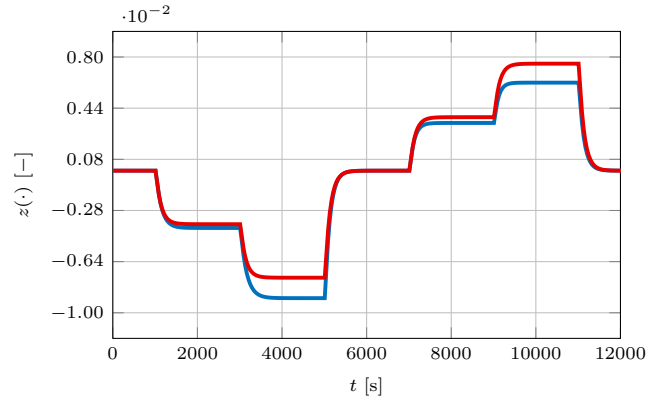
IV. CONTROLLER DESIGN

Main aim of this paper is to design a controller, which will be able to control the level of pH in the reaction vessel to a specific value. Since we are dealing with single-input single-output (SISO) systems, the first choice is to choose a PID controller [14]. PID controllers are the simplest controllers which are able to fulfill aforementioned requirements to some extent. Second, we will propose an optimal control strategy based in form of a model predictive controller [15].

Since the neutralization process is a strongly nonlinear process, a simple LTI-based control does not yield satisfactory



(a) pH identification.



(b) $z(\cdot)$ identification.

Fig. 3. Top figure shows the identification using direct pH measurements. Bottom figure, shows the identification via augmented output $z(\cdot)$. By red color is depicted the response of identified model, while the blue color represents the simulated response of nonlinear model.

results. Therefore, we chose to design a gain-scheduling controller in case of the PID control. Furthermore, a heuristic strategy is chosen also in case of the MPC-based controllers, when we switch between two controllers in certain pH intervals.

A. Gain Scheduling Control Design

In case of the gain scheduling design we consider the measurement of pH as the controlled variable. The closed-loop control scheme with multiple PID controllers is shown in the Fig. 4.

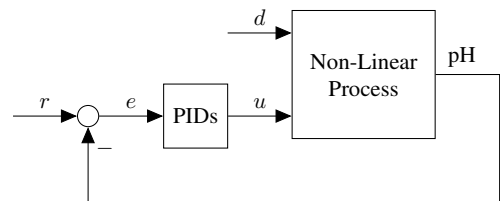


Fig. 4. Closed-loop scheme with PID controllers.

In our setup, we consider a PI controller in a form of

a transfer function

$$R(s) = P(\text{pH}) + \frac{P(\text{pH})}{T_i s}, \quad (11)$$

where the gain of the controller depends on the value of the pH. However, in implementation we consider a discrete time version of such a controller. A *Tustin* discretization method is considered with sampling period $T_s = 10$ s.

B. MPC-based Control Design

The closed-loop realization with the MPC-based controller is more complex than the one in the gain scheduling case. First, the design model, based on which the MPC is constructed is a discrete time state space model. A consequence is, that we need the knowledge of the states at each sampling instant, hence we need to include an observer in the closed-loop scheme. The complete control scheme with MPC-based controller and an observer can be found in Fig. 5.

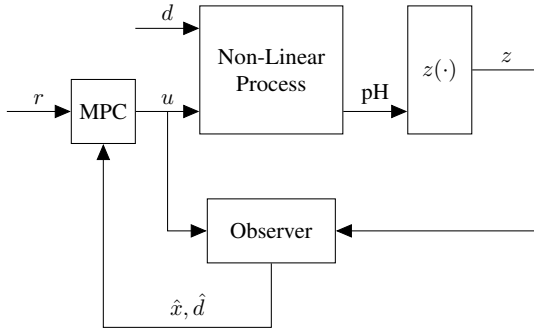


Fig. 5. Closed-loop realization with MPC, observer and with augmented output $z(\cdot)$.

The design model considered in the MPC construction is as follows

$$x(t + T_s) = Ax(t) + Bu(t), \quad (12a)$$

$$y(t) = Cx(t) + d(t), \quad (12b)$$

$$d(t + T_s) = d(t), \quad (12c)$$

where dynamics of disturbances $d(t)$ is considered constant. Disturbance modeling is included in the MPC design in order to achieve offset-free control [16].

Subsequently, the dynamics of the observer is given by:

$$\begin{bmatrix} \hat{x}(t + T_s) \\ \hat{d}(t + T_s) \end{bmatrix} = \begin{bmatrix} A & 0 \\ 0 & I \end{bmatrix} \begin{bmatrix} \hat{x}(t) \\ \hat{d}(t) \end{bmatrix} + \begin{bmatrix} B \\ 0 \end{bmatrix} u(t) + L \left(y(t) - C\hat{x}(t) - E\hat{d}(t) \right), \quad (13)$$

where state and disturbance estimates are denoted by $\hat{x}(t)$ and $\hat{d}(t)$, respectively. The variable L is the gain of the observer. To keep the notation consistent, we will refer to the measured output as $y(t)$. In the simulation, the measured output can be either the value of the pH or the value of the augmented variable $z(\cdot)$, depending on the choice of the controller. In both observer and MPC design, the structure remains the same, only tuning parameters changes.

$$\min_{u_0, \dots, u_{N-1}} \sum_{k=0}^{N-1} \left(Q_u \Delta u_k^2 + Q_y (y_k - y_{\text{ref}})^2 \right) \quad (14a)$$

$$\text{s.t. } x_{k+1} = Ax_k + Bu_k, \quad \forall k \in \mathbb{N}_0^{N-1}, \quad (14b)$$

$$y_k = Cx_k + Ed_0, \quad \forall k \in \mathbb{N}_0^{N-1}, \quad (14c)$$

$$\Delta u_k = u_k - u_{k-1}, \quad \forall k \in \mathbb{N}_0^{N-1}, \quad (14d)$$

$$u_{\min} \leq u_k \leq u_{\max}, \quad \forall k \in \mathbb{N}_0^{N-1}, \quad (14e)$$

$$y_{\min} \leq y_k \leq y_{\max}, \quad \forall k \in \mathbb{N}_0^{N-1}, \quad (14f)$$

$$u_{-1} = u(t - T_s), \quad (14g)$$

$$x_0 = \hat{x}(t), d_0 = \hat{d}(t). \quad (14h)$$

where $x_k \in \mathbb{R}^2$, $u_k \in \mathbb{R}$ and $y_k \in \mathbb{R}$ are states, control actions and system outputs at the prediction step k , respectively. Next, $N \in \mathcal{N}$ denotes prediction horizon, y_{ref} is the output reference, $\Delta u_k = u_k - u_{k-1}$ is increment of two consecutive control actions, Q_u , Q_y are positive definite weighting matrices and \mathbb{N}_a^b denotes a set of positive integers, i.e., $\mathbb{N}_a^b = \{a, a+1, a+2, \dots, b\}$. Moreover, the disturbance $\hat{d}(t) = d_0$ is assumed to be constant along the prediction horizon. The initial conditions (14h) for the optimal control problem in (14) are the estimates of the states and disturbance in accordance to the closed-scheme in Fig. 5.

V. RESULTS

Performances of proposed control strategies were demonstrated on a simulation based scenario involving a non-linear model of the neutralization process (2). In the first two subsections are described tuning setups of individual controllers, and in the final subsection simulation results are presented.

A. Gain Scheduling

Rules for the gain switching are summarized in following table I. The dependence of the gain P can be also viewed on the Fig. 6. The integration term T_i is fixed throughout the pH range to the value of 175. In the implementation we consider a discrete time version of the time-varying PI controller.

TABLE I
GAIN SCHEDULING

Interval No.	$P(\text{pH})$	interval for the pH
1	10.00	[0.00; 3.15]
2	$-2.59\text{pH} + 18.17$	[3.15; 7.00]
3	0.03	[7.00; 10.00]
4	$4.97\text{pH} - 49.67$	[10.00; 11.00]
5	5.00	[11.00; 14.00]

Values in the table I were found by trial and error. Simulation results with gain scheduling controller are visualized on the Fig. 7, where are compared with the performance of the optimal controller. The tunings of the MPC-based controller is described in the next sub-section.

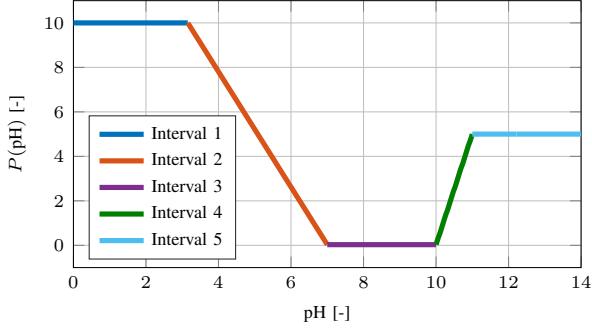


Fig. 6. Gain scheduling. Values for individual intervals can be found in table I

B. MPC-based Strategies Setups

Since nonlinearities in the process of the neutralization introduces great mismatch between the actual process and the design model even when the augmented variables in considered, also a switching MPC strategy is considered. Here, two intervals are utilized.

First considered interval is if $\text{pH} \in [3.15, 6.00]$, then an MPC where pH is measured variables is employed. The tuning of such an MPC was chosen as

$$N = 15, Q_u = 1, Q_y = 10^3, \quad (15)$$

with the design model identified as

$$A = \begin{bmatrix} 91.91 & 6.53 \\ 6.61 & 82.38 \end{bmatrix} \cdot 10^{-2}, B = \begin{bmatrix} 0.56 \\ 0.06 \end{bmatrix} \cdot 10^{-2}, \\ C = \begin{bmatrix} 3.25 \\ 1.33 \end{bmatrix}, E = 1, \quad (16)$$

The tuning of the MPC in this first interval is followed by a tuning of the observer. The Luenberger observer gain was found using pole-placement method, where the gain L was evaluated as

$$L = [1.43 \cdot 10^{-2} \quad 5.97 \cdot 10^{-3} \quad 9.34 \cdot 10^{-1}]^T. \quad (17)$$

Second considered interval was chosen as $\text{pH} \in [6.00, 14.00]$. Here, we switch not only the tuning of the MPC, but also we use the augmented variable $z(\cdot)$ as a measured variable. Tuning factors for this MPC were chosen as follows

$$N = 15, Q_u = 1, Q_y = 10^{10}, \quad (18)$$

with the design model obtain in a form of

$$A = \begin{bmatrix} 93.07 & 3.42 \\ 0.56 & 87.20 \end{bmatrix} \cdot 10^{-2}, B = \begin{bmatrix} 3.15 \\ -0.02 \end{bmatrix} \cdot 10^{-2}, \\ C = \begin{bmatrix} 1.11 \\ 0.29 \end{bmatrix}, E = 1. \quad (19)$$

The observer gain L for the second interval was calculated as

$$L = [5.19 \cdot 10^{-4} \quad 2.05 \cdot 10^{-4} \quad 9.90 \cdot 10^{-1}]^T. \quad (20)$$

Moreover, in both MPC setups, we have considered the same constraints on input and output variable. Specifically, we have

used $u_{\min} = 0, u_{\max} = 10, y_{\min} = 0$ and $y_{\max} = 14$, which translates into $F_2 \in [0, 10] \text{cm}^3/\text{s}$ and $\text{pH} \in [0, 14]$.

The choice of the intervals for MPC switch stems from the analysis of the titration curve (Fig. 2). We can observe, that in the interval up to $\text{pH} = 6$, the pH titration curve changes very little, compared to the $z(\cdot)$ curve. On the other hand, the changes in the interval between $\text{pH} = 6$ and $\text{pH} = 14$ are rather drastic in case of the pH curve. Here, the $z(\cdot)$ curve exhibits close-to-linear behavior throughout concentration change.

C. Control Performance Comparison

The simulations setups involves a sequence of reference changes, visualized in Fig. 7. The main difference in the performance can be seen on the control action, shown in Fig. 7(b). The profile of the manipulated variable in the gain scheduling control is more violent that in the case of the optimal controller. Moreover, the MPC policy also exhibits better tracking property. To be more concrete, we have employed following control quality criterion:

$$\Psi = \frac{1}{N_{\text{sim}}} \sum_{i=0}^{N_{\text{sim}}} (y_{\text{ref}} - y_i)^2 + \frac{1}{N_{\text{sim}}} \frac{1}{\sum_{i=0}^{N_{\text{sim}}} (\Delta u)^2}, \quad (21)$$

which is composed of the terms. The first one denotes the Sum of Squared Errors (SSE) between the controlled variable and the desired reference. The second term denotes the control effort. Both parts are normalized with respect to the simulation period. Generally it holds that the control policy performs better if the value of the criterion Ψ is lower. By applying (21) to our simulation depicted in Fig. 7, we have obtained $\Psi = 0.2683$ for PID and $\Psi = 0.2348$ for MPC. Thus, the MPC strategy exhibits 12% better control performance compared to PID controller. We would like to note, that one can still increase control performance via more precise tuning. In our setup, we have devoted the same amount of time to tune both control policies.

VI. CONCLUSIONS

The main contribution of this paper was the design of an MPC-based controller which will be able to control the level of the pH . Furthermore, the resulting controller is able to effectively control the pH in the whole range of applicable values of the pH . Needless to say, the optimal controller provides other advantages like constraint handling.

Among other novelties of this paper, is the introduction of augmented output in the controller design. Such a new output allowed us to utilize linear control theory even if the simulated model exhibit strong non-linear behavior.

ACKNOWLEDGMENT

Authors gratefully acknowledge the contribution of the Scientific Grant Agency of the Slovak Republic under grant 1/0403/15 and the contribution of the Slovak Research and Development Agency under the project APVV-15-0007. The first author also acknowledge the support by postdoctoral funding per STU grant 1311 and Martin Klaučo is supported by an internal STU grant for Young Researchers.

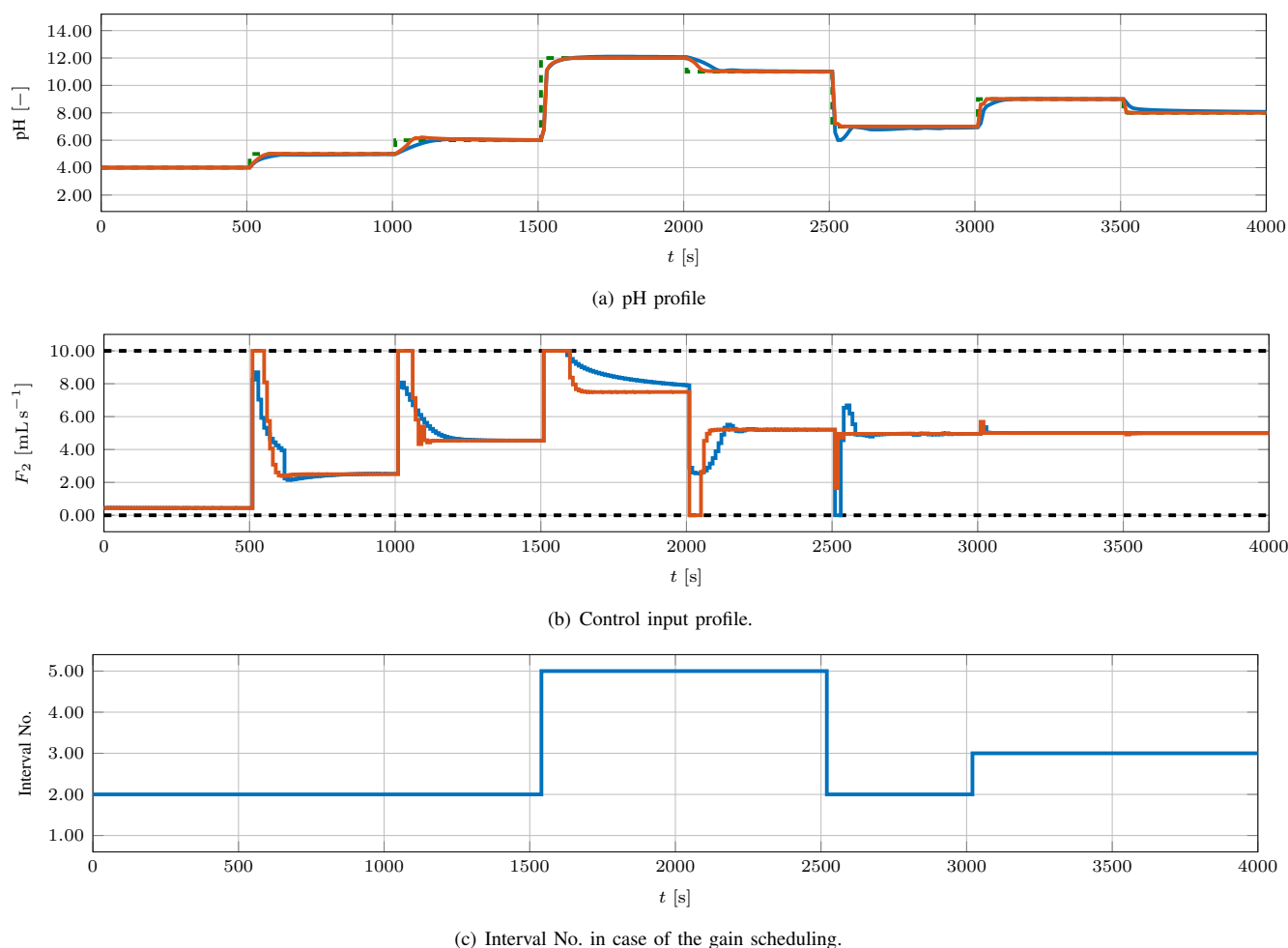


Fig. 7. The top figure shows the pH profile, middle figure shows the manipulated variable profile while the bottom figure shows the switching between interval for the gain scheduling controller. In all figures, the orange color depicts the performance of the MPC-based controllers, the blue color represents the performance of the gain scheduling.

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