#### [Chemical Engineering Science 190 \(2018\) 48–59](https://doi.org/10.1016/j.ces.2018.05.039)

# Chemical Engineering Science

journal homepage: [www.elsevier.com/locate/ces](http://www.elsevier.com/locate/ces)

## Experimental validation of online monitoring and optimization strategies applied to a biohydrogen production dark fermenter



CHEMICAL **ENGINEERING SCIENCE** 

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- A coupled robust observer is validated in an experimental bioreactor.
- The observer is used for implementing a heuristic optimization strategy.
- Optimization strategy and coupled bioreactor are thoroughly explained.
- The application maximizes the hydrogen production rate in a dark fermenter.



#### ARTICLE INFO

Article history:

Received 28 August 2017 Received in revised form 13 March 2018 Accepted 23 May 2018 Available online 26 May 2018

Keywords: Hydrogen production Anaerobic digestion Dark fermentation Online monitoring Online optimization Nonlinear observer

#### **ABSTRACT**

This work presents the proposal and experimental validation of an online optimization strategy for maximizing the hydrogen production rate (HPR) in an anaerobic bioreactor through the dark fermentation of glucose. The proposal comprises a heuristic optimization strategy, a coupled nonlinear observer, and a PI controller to track the desired maximum HPR. The optimization is achieved by solving online a nonlinear programming problem which considers the relation between the HPR and the organic loading rate (OLR), such that the latter is manipulated to maximize the former. Since the OLR requires online knowledge of the unmeasured influent substrate concentration, the nonlinear observer is used to estimate it using measurements of the hydrogen flow rate at the output of the bioreactor. This observer comprises the coupled operation of a robust Luenberger observer and a super-twisting observer. The proposal was designed and tested experimentally in a continuous stirred tank laboratory bioreactor fed with glucose, and the results are very promising.

1. Introduction

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The worldwide energy need has been increasing exponentially, the reserves of fossil fuels have been decreasing, and the combustion of fossil fuels has serious negative effects on the environment because of  $CO<sub>2</sub>$  emissions. Hydrogen is considered as a viable

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alternative fuel and the energy carrier of the future, with no  $CO<sub>2</sub>$ emissions. Besides, it has a high energy yield of 122 kJ g $^{-1}$ , which is 2.75 times greater than hydrocarbon fuels ([Kapdan and Kargi,](#page--1-0) [2006](#page--1-0)).

Most hydrogen used nowadays is produced from nonrenewable sources such as oil, natural gas and coal; about 50% is obtained using thermocatalytic techniques and gasification of natural gas [\(Logan, 2004\)](#page--1-0). Nevertheless, biological production of hydrogen (biohydrogen), using (micro) organisms, is an area of technology development that offers the potential production of usable hydrogen from a variety of renewable resources. Biological systems provide a wide range of approaches to generate hydrogen, and include direct biophotolysis, indirect biophotolysis, photofermentations, and dark fermentation [\(Das and Veziroglu, 2001;](#page--1-0) [Levin et al., 2004; Kapdan and Kargi, 2006\)](#page--1-0).

Dark fermentation, i.e. the fermentative conversion occurring when methanogenesis is prevented in anaerobic digestion processes, constitutes an attractive alternative to conventional physical/chemical methods for hydrogen production because it requires low investment and is well suited for decentralized energy production in regions where biomass or organic waste is available, thus avoiding the expenditure and energy cost of transport ([Trad](#page--1-0) [et al., 2016\)](#page--1-0). Besides, it has the advantage of high hydrogen production rates, simple operation and, regarding the substrate, it can be applied to organic solid wastes and to the organic compounds present in the wastewater ([Wang and Wan, 2009](#page--1-0)).

In fermentative hydrogen production the operational parameters have a crucial role on establishing the metabolic pathway of the microorganisms, which influence the process efficiency, the product gas quality and the required energy inputs. Many research works have focused on finding optimal operational parameters (e.g. pH, temperature, hydrogen partial pressures, or inoculum) to obtain the maximum hydrogen yield and/or hydrogen production rate (HPR). An operational parameter that affects the hydrogen production rate in a dark fermenter is the organic loading rate (OLR). According to [Ramírez et al. \(2015\), Hafez et al. \(2010\), and](#page--1-0) [Shen et al. \(2009\)](#page--1-0), an optimum OLR exists in which the HPR is maximum; however, this optimum OLR is close to the overloading one. Therefore, in order to maximize the HPR, the OLR, which depends on both the inflow rate and the influent substrate concentration, should be maintained at an optimal value, even despite possible external perturbations.

In the last decade, optimization methods have been developed in order to online maximize the hydrogen production in dark fermentation bioreactors. For example, [Aceves-Lara et al. \(2010\)](#page--1-0) apply model predictive control (MPC) to optimize the hydrogen production in continuous anaerobic digesters using the influent flow rate as the main control variable. [Huang et al. \(2012\)](#page--1-0) apply fuzzy control-based real-time optimization of pH and temperature to achieve the best growing environment and hydrogen production rate, as well as to enhance hydrogen production in a dark fermentation reactor. [Ramírez et al. \(2015\)](#page--1-0) propose a heuristic optimization strategy to maximize the hydrogen production in a dark fermenter by considering as objective function the relation between the hydrogen production rate (HPR) and the organic loading rate (OLR). The influent flow rate was proposed as optimization variable, while the influent substrate concentration was maintained constant.

This article presents an improved version of the online optimization strategy proposed by [Ramírez et al. \(2015\)](#page--1-0). The nonlinear programming (NLP) optimization problem is reformulated by adding a constraint on the substrate consumption. Since in realistic situations the influent substrate does not remain constant, in this study concentrations varying between minimal and maximal values are considered. However, measuring online the influent substrate concentration is not practical. The observer proposed by [Torres Zúniga et al. \(2015\)](#page--1-0) is therefore used to estimate it and thus also the OLR. This OLR estimation allows us to solve the NLP problem and compute the maximum HPR. An anti-windup PI control law is then used to track this maximum HPR and to limit the hydraulic retention time of the process. At the same time, the biomass and substrate concentrations in the reactor are estimated. By considering the estimation of the concentrations of substrate in the influent and inside the reactor, the substrate consumption can also be estimated. This way, the effect of maximizing the process productivity on the substrate consumption can be analyzed and decisions on the reactor operation can be taken online via feedback control. On the other hand, the estimation of the substrate consumption and the biomass inside the reactor can help to online monitor the state of the dark fermenter. Thus, the purpose of this work is:

- to experimentally validate the online optimization strategy proposed by [Ramírez et al. \(2015\)](#page--1-0), but now with variable glucose concentrations in the influent, which are estimated using an enhanced version of the observer proposed by [Torres Zúniga](#page--1-0) [et al. \(2015\)](#page--1-0),
- to experimentally validate an observer-based online monitoring strategy to estimate the glucose consumption and the biomass produced.

The biohydrogen production process is depicted in [Fig. 1](#page--1-0). From a control engineering perspective, this process has two inputs: the substrate concentration ( $Glu_{in}$ ), considered as an uncontrolled input, and the influent flow rate  $(Q_{in})$ , considered as the controlled input. On the other hand, the total gas flow rate  $(Q_{gas})$  and the hydrogen fraction ( $\%H_2$ ) at the reactor output are measured online. Using both measurements, the hydrogen flow rate  $(q_{H2,gas})$  can be calculated and used as process output signal to control the process. A list of symbols and the nomenclature used is shown in [Table 1](#page--1-0).

#### 2. Materials and methods

#### 2.1. Inoculum and feed water composition

The inoculum was obtained from granular sludge of an anaerobic bioreactor used to treat the wastewater of a brewery plant. The sludge was subject to a heat pretreatment as described by [Buitrón and Carbajal \(2010\)](#page--1-0). The feed water was prepared using tap water and glucose as organic substrate, with several concentrations, as detailed later. For every gram of glucose in the synthetic wastewater, 104 mg NH<sub>4</sub>Cl and 50 mg  $K_2HPO_4$ were added, and for every liter the following amounts of mineral salts were added:  $0.4 \text{ mg } MnCl_2 \cdot 4H_2O$ ,  $20 \text{ mg } MgCl_2 \cdot 6H_2O$ ,<br> $20 \text{ mg } FeSO_4 \cdot 7H_2O$ ,  $2 \text{ mg } CoCl_2 \cdot 6H_2O$ ,  $2 \text{ mg } Na_2MoO_4 \cdot 2H_2O$ , 20 mg FeSO<sub>4</sub> · 7H<sub>2</sub>O, 2 mg CoCl<sub>2</sub> · 6H<sub>2</sub>O, 2 mg Na<sub>2</sub>MoO<sub>4</sub> · 2H<sub>2</sub>O, 2 mg H<sub>2</sub>O, 2 m 2 mg  $H_3BO_4$ , 2 mg NiCl<sub>2</sub>  $\cdot$  6H<sub>2</sub>O and 2 mg ZnCl<sub>2</sub>. The feed water was prepared daily and was refrigerated to minimize its biodegradation outside the reactor.

#### 2.2. Experimental setup, startup and reactor operation

The experiment was carried out in a continuous stirred tank reactor (CSTR) of 1.2 L with 0.9 L of reaction volume equipped with a bio-controller (Applikon Biotechnology), which kept the reactor operating at 35  $\degree$ C, with a stirring velocity of 100 rpm and pH regulated at 5.5 by the addition of NaOH. The culture was started in batch mode with 15 g  $L^{-1}$  of glucose and 4 g  $L^{-1}$  of volatile suspended solids (VSS). After 5 cycles of 12 h, the reactor was switched to continuous operation. The feed solution was fed into the reactor by a remotely controlled variable speed peristaltic pump (Masterflex).

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