MICROFLOW GENERATOR FOR FUEL CELL METHANOL HYDROGEN MICROREACTOR

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Key words: flow generator, methanol hydrogen microreactor, micropump, PID regulation, microchip, MPC control

Abstract: Design, fabrication and characterization of low-cost, energy efficient flow-generator for hydrogen production in microreactors is presented. Design guidelines of three approaches, based on micropump and pressure sensor are discussed in detail, followed by experimental setup description and system characterization. A comparative analysis of all three approaches was performed and most appropriate solution for methanol hydrogen microreactor is proposed. Due to adequate system energy efficiency and system stability, approach based on differential pressure sensing, supported by PID control regulation was found as the most appropriate, what was further confirmed with successfully running microreactor applications.

Dozirni sistem za mikroprocesor goriva

Kjučne besede: generator pretoka, metanol / vodik mikroreformer, mikročrpalka, PID regulacija, mikročip, MPC kontroler

Izvleček: V prispevku predstavljamo tri pristope za učinkovit mikrodozirni sistem, namenjen doziranju goriva pri gorivnih celicah. Poudarek je na načrtovanju in izdelavi prototipov v makro izvedbi, čemur sledi testiranje, ki je ključnega pomena za objektivni izbor najprimernejše metode za našo aplikacijo. Vse metode združujejo uporabo mikročrpalke, tlačnega senzorja in regulatorja. Dokazali smo, da je s temi elementi možno realizirati zanesljiv dozirni sistem in se izogniti uporabi dragih, velikih komercialnih merilnikov pretoka. Kot najbolj stabilna in učinkovita se izkaže metoda, ki vključuje merjenje diferencialnega tlaka na zaslonki.

1. Introduction

Precise dosing of substances is becoming an important part of many microfluidic devices, for example in the field of biotechnologies, etc. This is dictated by the advantageous fluidic behavior at small volumes and consequently ever increasing need for high throughput assays in pharmaceutical and chemical industry, as well as other combinatorial based studies, such as in biotechnologies.

The integration of conventional techniques onto microchip platforms would render a substantially faster, less labor-intensive and inexpensive approach, both in terms of operating cost and of low volume reagent usage. This allows their introduction into the clinical setting as powerful analytical tools, for example in disease detection and monitoring. Many molecular-biology techniques require the use of precision substance supply systems to catalyze successful and accurate biochemical reactions /1/.

Similar approaches as found in the pharmaceutical and chemical field /2, 3/ can be introduced also in application of microreactor hydrogen production for microfuel cells, where reliable and efficient fuel dosing is crucial for proper operation. Methanol steam reforming refers to the chemical reaction between methanol and water vapor for the production of hydrogen gas. This process is typically carried out in the presence of metal oxide catalysts at temperatures ranging from 220 °C to 300 °C /4/.

The main objective of present work was to assemble low-cost fuel supply system for hydrogen production microreactor with good efficiency. Providers of such microsystems are still scarce due to very specific requirements, depending on customer application.

In the field of measurements, there are quantities which are rather easy to measure, but also quantities where measurements on micro level represent difficulty, e.g. flow rate. Measurement and control of highly dynamic fluid flow at extremely low magnitudes are still a challenging task. To avoid expensive, usually bulky professional low-level fluid flowmeters, in our case pressure sensor was used for flow-generator realization.

Presented paper presents three different approaches to fulfill flow-generator design requirements, dictated by fuel-cell methanol hydrogen microreactor. A detailed insight into the design, optimization, fabrication, analysis and performance of the proposed approaches is presented as well as its applicability potentials.

2. Flow-generator design requirements

Flow-generator refers to apparatus which supplies defined amount of fluid into the system, independent from its dynamic hydraulic resistance and other conditions.

Classic flow-generator comprises pump controlled by appropriate controller and fluid flowmeter. Measured flow rate passing through the supplied system is fed back and compared with desired setpoint. To correct a difference between set point and measured flow rate, closed-loop controller is employed. Ideally, supplied flow rate must be independent of system hydraulic resistance. In fluid flow measuring, lots of various approaches can be applied /5/.

Methanol hydrogen microreactor is composed of several basic modules. The first module is evaporator. It consists of microchannels etched on the surface of silicon substrate. On the other side of silicon substrate a platinum heater /7/ is located. Microreactor evaporator is therefore thermally coupled with heater, thus enabling evaporation of input energent methanol and water mixture. The statistical nature of evaporating process causes stochastic changes in frequency and magnitude of micro droplet explosions. Therefore, the evaporator can be modeled as a highly dynamic hydraulic resistor. The hydraulic resistance is primarily dependent on the heater temperature, microchannel geometry, fuel mixture concentration ratio and foremost, on input fuel rate supply.

Therefore, our primary flow-generator design requirement is to achieve a constant output flow, independent from stochastic pressure impulses caused by evaporator micro droplet explosions.

Another important design requirement in flow meter design refers to output flow generation range. In order to provide efficient fuel evaporation, in our case a constant fuel supply in the interval from 0.1 ml/h up to 1 ml/min is required. This low level flow range is rather unusual but nowadays often met in microchannel applications.

Professional low level flow meters can be found on the market but were found inappropriate for flow-generator in microreactor applications due to their price and size. The majority of low level flow meters for microreactor application are based on thermal mass method, which requires expensive, electric power consuming custom designed electronic circuits. Therefore, the idea of microdosing system based on pressure sensor was adopted. Furthermore, data from pressure sensor can provide additional information about evaporation process quality. In addition, further investigation could be extended on defining and controlling the fuel amount supply rate, based on pressure sensor data processing.

3. Approaches

3.1. Method I – Model Predictive Control (MPC)

To provide constant fuel flow through microreactor, an unconventional approach was employed. The basic idea was equipping controlled system (micropump in line with evaporator) with internal mathematical model in order to control fluid flow rate by considering only measured stagnation pressure *p* before evaporator and applied micropump actuating voltage *U.* To achieve this goal, feedback loop of internal variable was established and fed back to the reference flow rate value through controller, finally resulting in proper micropump voltage supply *U* (Fig. 1).

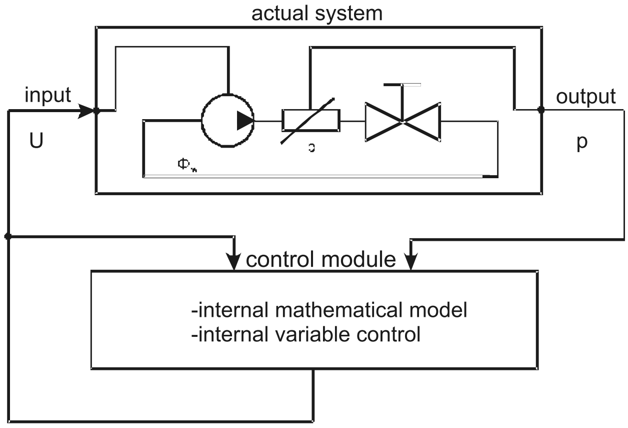


Fig. 1. Micro-flow generator with MPC approach.

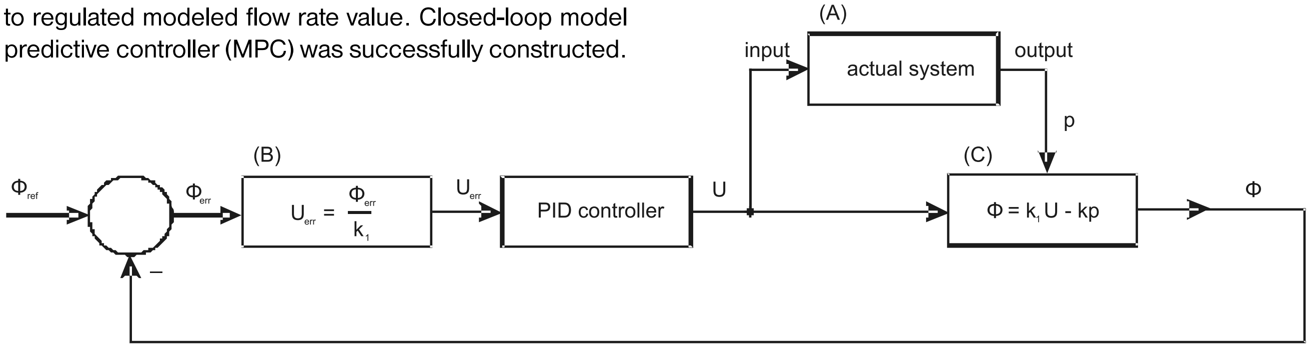
Model Predictive Control (MPC) approach represents an advanced method of process control that has been in use in the process industries since the 1980s /8/. MPC controllers rely on dynamic models of the processes, most often linear empirical models obtained by system identification. The models are used to predict the behavior of dependent variables (e.g. outputs) of the modeled dynamical system with respect to changes in the process independent variables (e.g. inputs). The MPC controller uses the models and current measurements to calculate future moves in the independent variables. This will result in operation that honors all independent and dependent variable constraints.

For the purpose of MPC method, a simple micropump mathematical model (Fig. 2, block C) was introduced.

Simulated flow Φ was obtained from actual micropump driving voltage *U* and stagnation pressure *p* measured before evaporator. In approximation, flow rate can be expressed as linear function of both two variables *U* and *p*. (Fig 2, block C). Coefficients k_1 and *k* in mathematical model were determined from previously measured micropump characteristics.

In the next step, modeled flow value is sent through negative feedback where it is subtracted from the reference value (set point) to create the error signal which is then amplified by the PID controller. Block B (Fig. 2) can be used to transform Φ_{err} signal into U_{err} signal, but due to its linear dependency it can also be achieved with modified PID parameters.

The controller then takes the error signal (difference) between the reference flow value and modeled flow value to change the input micropump driving voltage *U* for both, actual system (Fig. 2, block A) and mathematical model. If the mathematical model (Fig. 2, block C) is in good agreement with actual system, system flow rate Φ for measured stagnation pressure *p* at pump driving voltage *U* is similar to regulated modeled flow rate value. Closed-loop model predictive controller (MPC) was successfully constructed.



ϕ_{ref} .... desired flow (set point)
ϕ_{err} .... difference between calculated flow and desired set point
ϕ .... calculated flow through stagnation pressure p and micropump driving voltageU
U_{err} ... micropump driving voltage error
U ... micropump driving voltage

Fig. 2. MPC controler schematics.

In internal mathematical model (Fig. 2, block C), simulated flow rate *Φ* needs to be obtained from actual micropump driving voltage *U* and stagnation pressure *p* measured before evaporator.

As claimed by manufacturer and confirmed by measurements, almost linear characteristics can be expected from micropumps applied (Fig. 3).

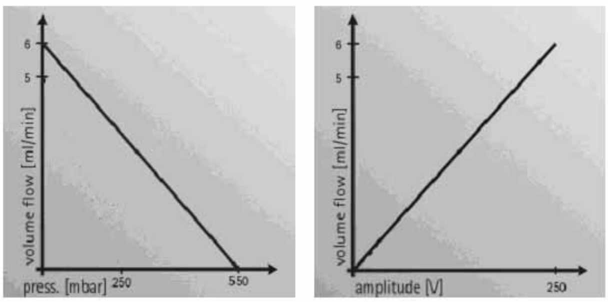


Fig. 3. Micropump characteristics Φ(p), Φ(U).

Therefore, as seen on Fig. 3, flow rate can be expressed as a linear function of both two variables *U* and *p*.

$$\Phi(U,\ p) = k_1 U - kp \qquad (1)$$

Presentation of mathematical model (1) for characteristics on Fig. 3 (water pumping @ 100Hz) was thus obtained by using *Mathematica* /9/ computer software. Results are shown on Fig. 4.

Here *k* is regression line slope coefficient, drawn through measured points in micropump *Φ(p)* characteristics. *k* is assumed equal for any micropump operating voltage *U*.

k_1 is the slope coefficient of regression line, drawn through measured points in micropump *Φ(U)* characteristics. k_1 is assumed equal for any micropump output stagnation pressure *p*.

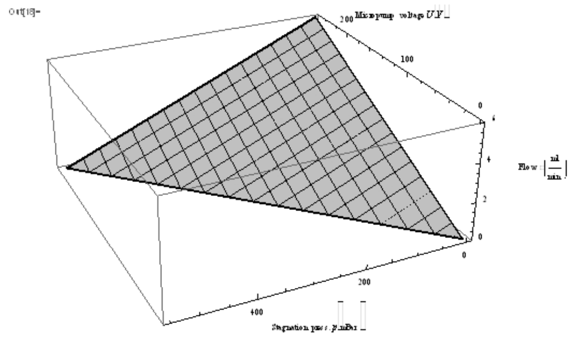


Fig. 4. Micropump mathematical model.

The same linear model is expected to be valid for any optional fluid pumped through the system. In this case, coefficients *k* and k_1 needs to be determined by flow rate *Φ* and stagnation pressure *p* measuring at optional micropump supplied voltage *U*. For parameter determination, a simple procedure was implemented: Micropump operating voltage *U* was set to maximum, and micropump output flow rate Φ_{max} was measured by using horizontal aligned pipette and time measurement:

$$k_1 = \frac{\Phi_{max}(U_{max})}{U_{max}} \qquad (2)$$

Pressure sensor was then attached to micropump output in order to measure maximum stagnation pressure p_{max}:

$$k = \frac{\Phi_{max}(U_{max})}{p_{max}} \qquad (3)$$

For the above mentioned method, sufficient agreement between model and real system as well as micropump stability was expected crucial for stable and accurate flow supply. It is difficult to estimate micropump stability and model linearity without adequate experiments as well as anomaly influence to overall system stability.

3.2. Method II – Electric current source analogy

The principle of second method originated from electric current source analogy. In electric circuit on Fig. 5, independent current source can be assembled with voltage source, followed by resistor *R* which resistance is always considerably greater then load resistance R_{load}. In this case, the current flowing trough load resistor R_{load} is independent of its resistance. Therefore, the quality of such current source depends on R to R_{load} ratio.

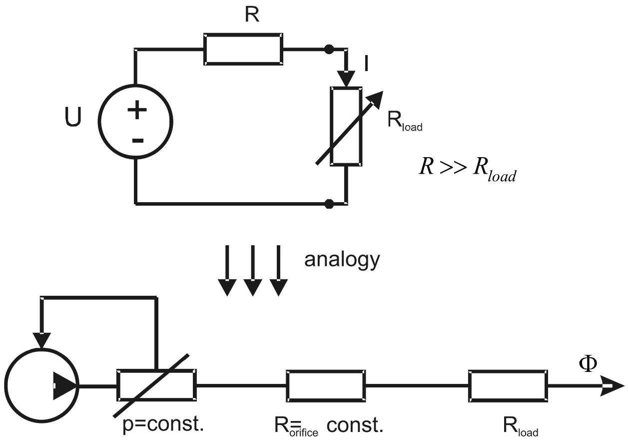


Fig. 5. Electric current source analogy.

In microfluidics, voltage source is in analogy to constant pressure fluid source while electric resistor R is in analogy with orifice nozzle. Fluid flow rate trough hydraulic load resistor R_{load} equals:

$$\Phi = \frac{p}{R_{orifice} + R_{load}} \quad (4)$$

In case when hydraulic orifice nozzle resistance $R_{orifice}$ is some orders of magnitude greater then the hydraulic load resistance R_{load}, system flow Φ can be given as follows:

$$R_{orifice} >> R_{load} \quad \Rightarrow \Phi \approx \frac{p}{R_{orifice}} \quad (5)$$

Therefore, fluid flow source at constant pressure can simply be realized with micropump as actuator, followed by pressure sensor measuring micropump output pressure. Pressure data are send trough negative feed back closed loop to set point comparator and its output is further led to PID controller, controlling micropump voltage supply. Reference pressure p_{ref} is determined as:

$$p_{ref} = R_{orifice} \cdot \Phi_{ref} \quad (6)$$

Closed loop control is established (Fig. 6).

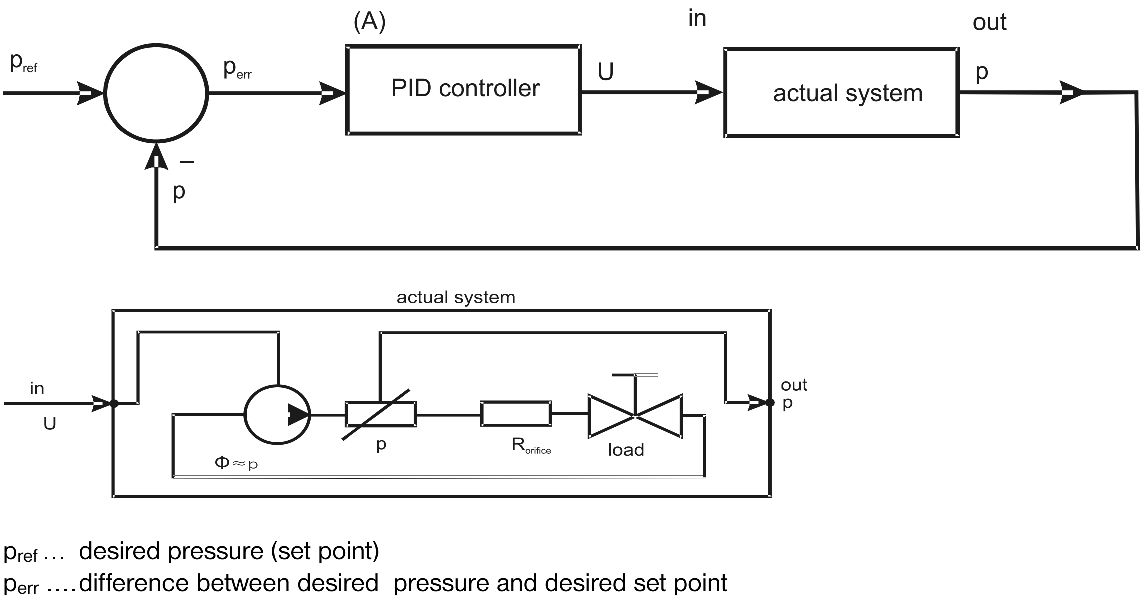
3.3. Method III – Venturi meter / orifice plate

Venturi meter determines the flow by measuring the differential pressure before and within a local channel constriction. This method is widely used to measure flow rate in the transmission of gas through pipelines. Constriction can be implemented as an orifice. Orifice plate is a plate with a through hole, placed in the flow. Both Venturi and orifice plate flow measuring were performed. The data were then fed back and compared with desired set point. To correct discrepancy between set point and measured flow rate, again classic closed-loop PID controller is employed as shown on Fig. 7.

4. Experimental setup

Micropumps available on the market, silicone tubes, metering valve Hoke type 1345G2B and PC with developed PID implementation software were used to assemble micro flow-generator prototypes.

Piezoelectric type micropump was chosen as flow-generator actuator device due to small size and weight, with good particle tolerance and temperature resistance. It combines two piezoactuators inside a single housing and has an in-



p_{ref} ... desired pressure (set point)
p_{err} ....difference between desired pressure and desired set point
p .... measured micropump output pressure
U .... micropump driving voltage

Fig. 6. Micro flow generator based on electric curent source analogy.

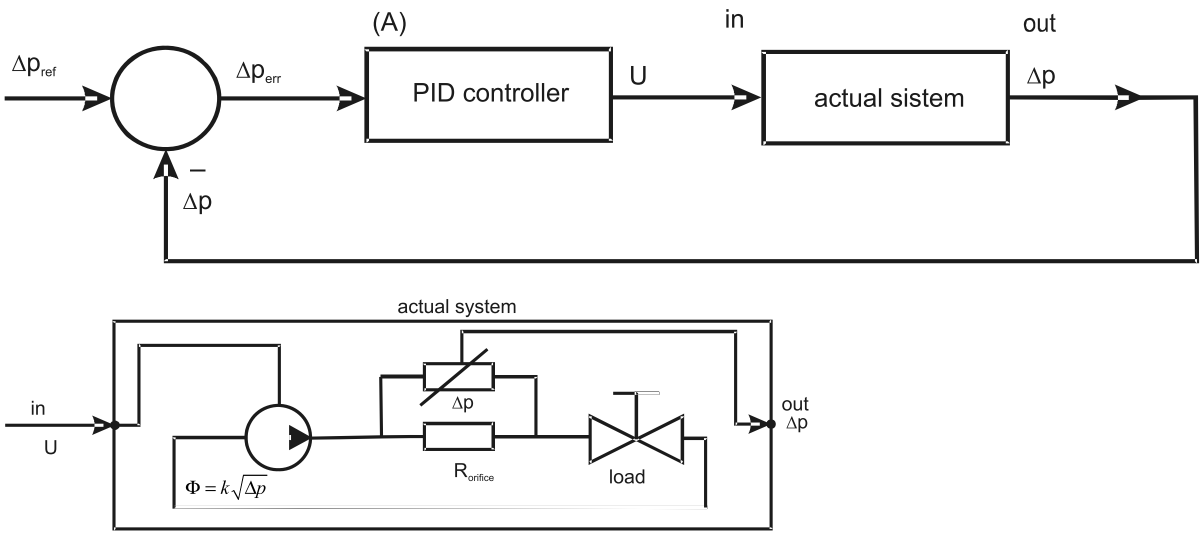


Fig. 7. Micro flow generator schematics. It is based on differential pressure measuring on venturi pipe or orifice plate.

creased priming capability. It provides high bubble tolerance, so that even gas-liquid-mixtures can be pumped without problems. Micropump controller with variable voltage amplitude and frequency was used for micropump driving.

During measurements, metering valve Hoke, type 1345G2B /10/ was used as evaporator simulator. Standard differential pressure sensor EST2233 /11/ was initially utilized, later replaced by temperature compensated EHT23000 /12/ type, additionally equipped with inlet and outlet acryl tubes, and in our lab assembled I^2C/USB converter (based on 24LFXI /14/ integrated circuit) as shown on Fig. 8.

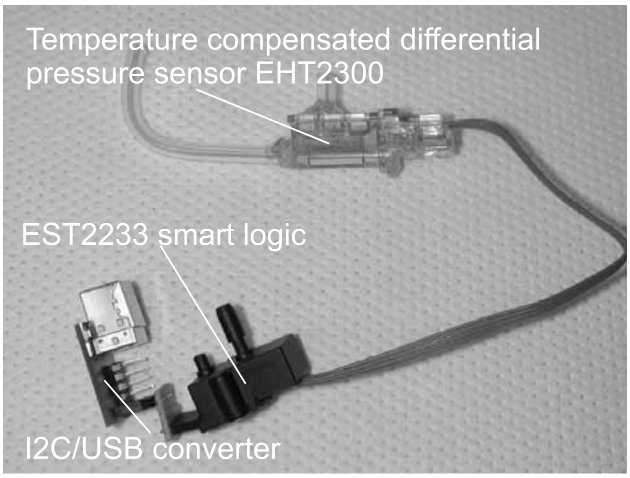


Fig. 8. Temperature compensated differential pressure sensor EHT2300 with EST2233 smart logic and I2C/USB converter.

Acrylic glass prototypes of Venturi pipe and orifices were fabricated, utilizing acrylic glass tubes in combination with thermal treatment technique, mechanical drilling and acrylic adhesive Rohm Acrifix 192. Further study will be focused on device miniaturization by implementing components on silicon substrate, employing wet etching technique and Pyrex glass covering. Passive hydraulic prototypes are shown on Fig. 9.

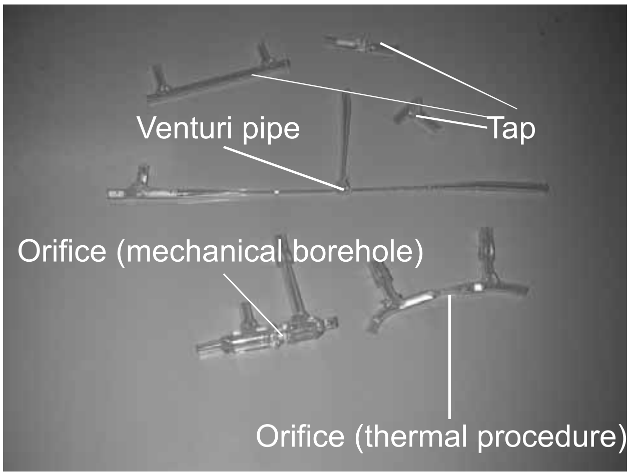


Fig. 9. Passive hydraulic prototypes.

Software implementations have the advantages that they are relatively cheap and flexible with respect to the implementation of the PID algorithm. For prototype version, *Visual Basic* /15/ program language on PC was initially used for discrete PID controller implementation to determine all required parameters and system test procedures. Once fully developed, the algorithm can be easily modified and compiled for any optional microcontroller board. The software development was obeyed to the following requirements: Real time pressure sensor data reading, micropump control ability, object oriented programming, simple control algorithm modifying capability, user friendly interface with option for essential parameters and reference editing, graphic output, FFT sensor signal processing and data saving.

Screenshot of developed user interface is shown in Fig. 10.

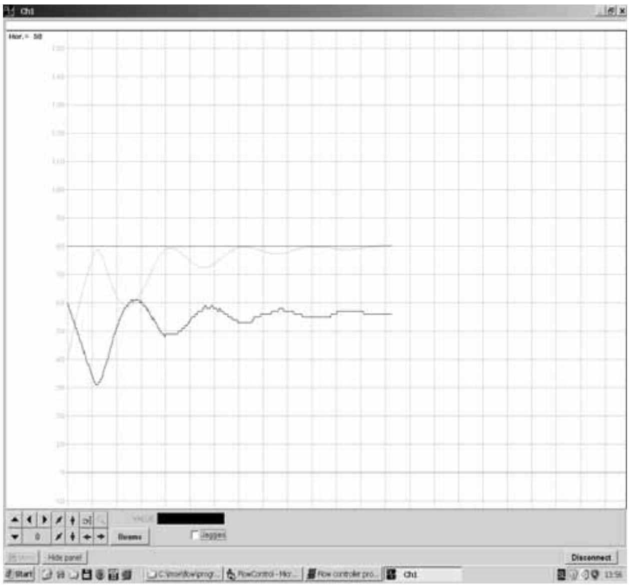


Fig. 10. User interface screenshot.

A Proportional–Integral–Derivative controller (PID) controller (Fig. 11) seemed to be suitable choice for fast and reliable control action and minimum system oscillation in our flow control application. However, it must be emphasized that the use of PID algorithm for control does not guarantee neither optimal control of the system nor system stability. The latter can be accomplished only through proper system architecture.

In short, PID represents a generic control loop feedback mechanism (controller) widely used in industrial control systems.

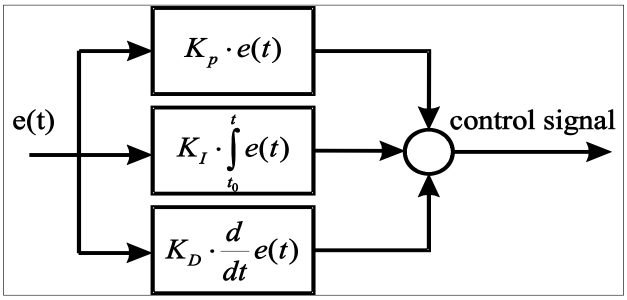


Fig. 11. PID controler schematics.

The PID parameters used in the calculation must be tuned according to the nature of the system. Several methods for tuning the PID loop exist /16/. In our case, however, precise manual tuning was found superior over conventional Zigler-Nicols method and was therefore used in described application.

Typical measurement setup for MPC approach is shown in Fig. 12. In characterization procedure, measuring valve 1345G2B in combination with measuring pipette was used.

Hydraulic orifice nozzle resistance $R_{orifice}$ in electric current analogy approach is realized with acrylic glass orifice as shown on Fig. 13. Two realizations of orifice plate and venture pipe are shown on Fig. 14, Fig. 15 and Fig. 16, respectively. After essential measurements, each flow-generator operation was finally confirmed by micro-evaporator as shown on Fig. 16.

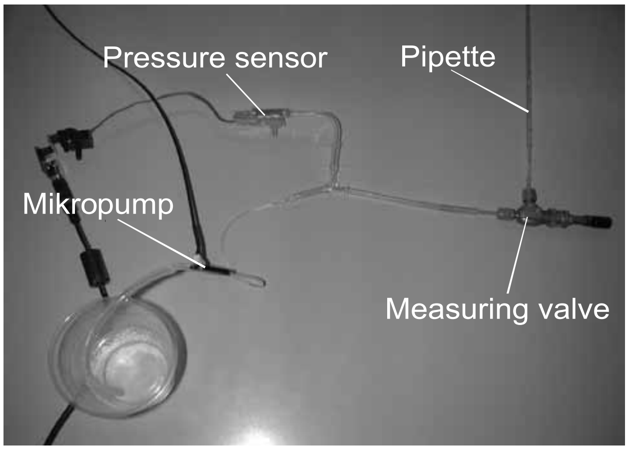


Fig. 12. Typical measurement setup with measuring pipette for flow determination.

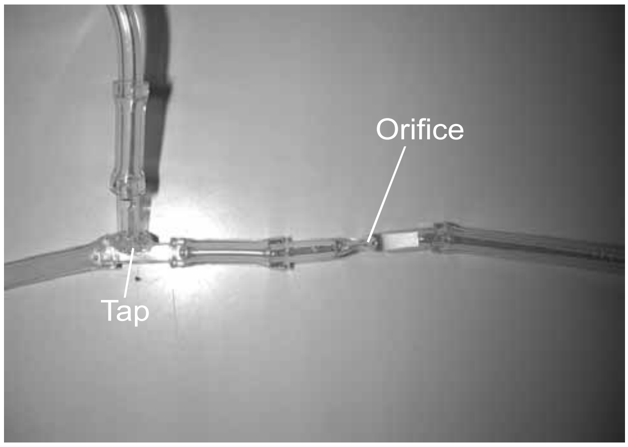


Fig. 13. Acrylic glass orifice. Intended for measurement setup for electric current analogy approach.

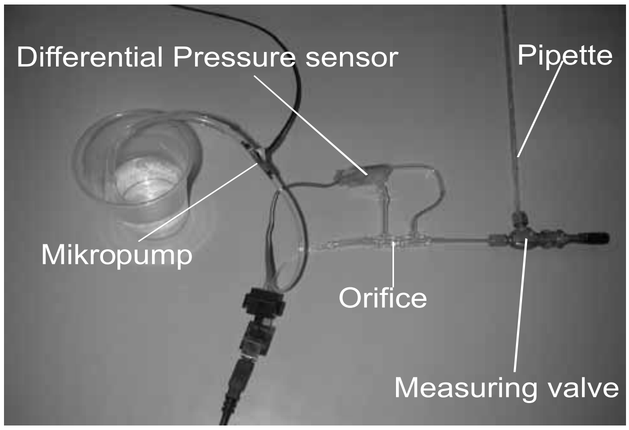


Fig. 14. Typical measurement setup for differential pressure measurement on orifice plate.

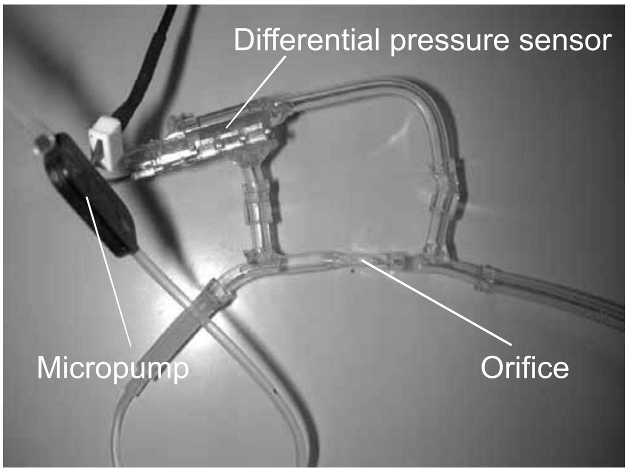


Fig. 15. Another prothotype of acrylic glass orifice.

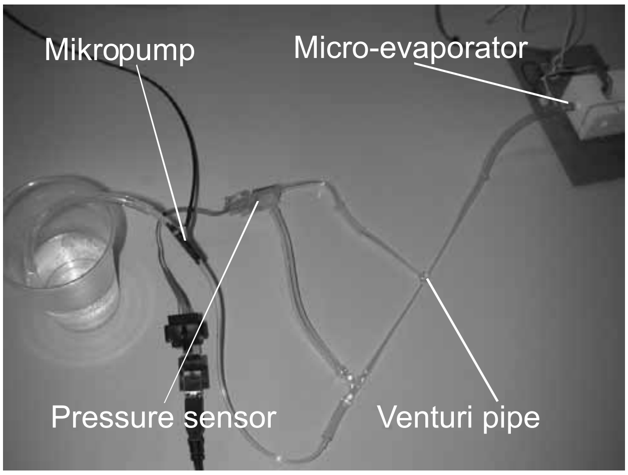


Fig. 16. Venturi pipe.

To obtain dependency of flow rate Φ vs. hydraulic load resistance R_{load}, measuring valve 1345G2B type was connected on micro flow-generator output. Later it was replaced by micro-evaporator to estimate its functioning in the real dynamic system. Flow-generator can only provide constant flow for a limited regulating range, which is defined primarily by a micropump head pressure.

Low level flow rate measuring was performed by placing horizontally 1 ml measuring pipette with scale resolution of 0.01ml and a stopwatch (volumetric method). This measurement technique was found appropriate for measuring flow rates as low as 0.1 ml/h.

5. Results and discussion

5.1. Method I – Model Predictive Control (MPC)

For the first proposed MPC method, verification of mathematical model and real system is crucial for stable and accurate flow supply. Therefore numerous micropump endurance tests were performed. Conducted tests enabled estimation of pump stability and repeatability (Fig. 17). All measured results apply to the DI water medium.

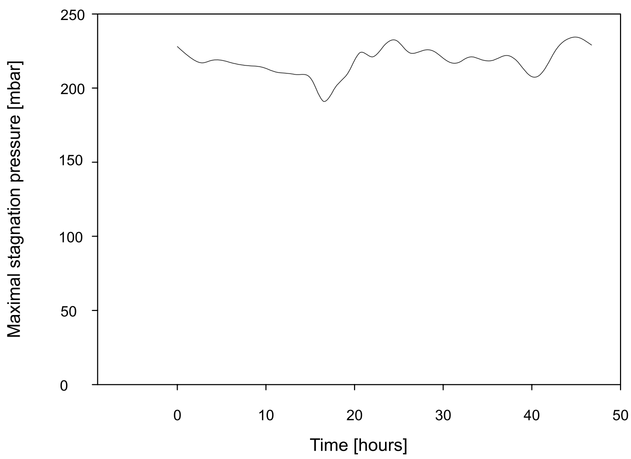


Fig. 17. Micropump two days stability test.

First order flow rate linear approximation in model was also taken into consideration by comparing measured and modeled data (Fig. 18).

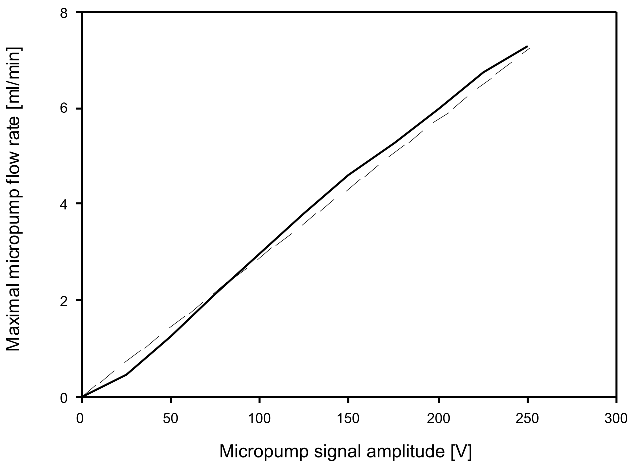


Fig. 18. Micropump linear inconsistency. Full line: Measured data. Dashed line: Linear regression.

Nonlinearity can be compensated by implementing appropriate compensation table. Unfortunately, micropump stability over time can not be compensated. Sufficient micropump stability is prerequisite in order to achieve stable MPC flow-generator system. In our case, endurance tests (Fig. 17) revealed micropump stability issue. Over longer periods (e.g. one week) obvious micropump performance degradation was detected.

Due to micropump longterm instability, the whole system was found unstable and therefore only functional for a short period, but still sufficient for evaluation. All measurements were taken in one hour, then the drift was detected and after 5 hours the system collapsed, which resulted as zero output flow rate. System drift is not linear function of time. At the beginning it is almost undetectable, but then it enhances until the regulation control collapses. System flow rate Φ vs. load hydraulic resistance R_{load} characteristic is presented on Fig. 19.

Measured stagnation pressure *p* vs. load hydraulic resistance R_{load} characteristic is presented on Fig. 20 and micropump signal amplitude response *U* on load hydraulic resistance R_{load} is shown on Fig. 21, respectively. In all characteristics (Fig. 19 – Fig. 25), the change of hydraulic load resistance R_{load} is here given by number of valve handle turns. Number of turns is proportional to decreased measuring valve hydraulic resistance R_{load} which can be calculated by using manufacturer valve characterization curve (number of valve handle turns vs. C*v* factor /18/) and a correction factor /18/ for different fluid types.

Short term operation was found functional. The main advantage of this system is foremost estimated energy efficiency, because this approach does not introduce any passive hydraulic elements. Flow rates down to 0.1 ml/min were achieved with this approach.

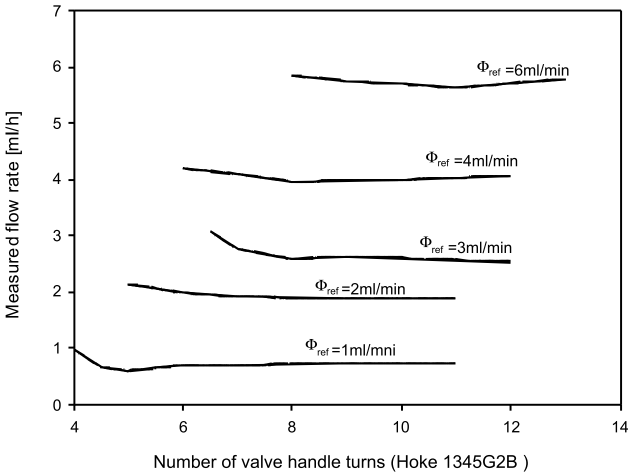


Fig. 19. MPC approach: System flow rate Φ vs. load hydraulic resistance R_{load} *characteristic.*

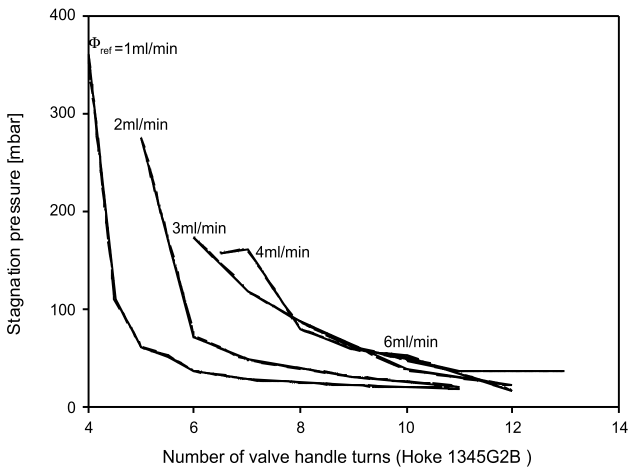


Fig. 20. MPC approach: Measured stagnation pressure p *vs. load hydraulic resistance* R_{load} *characteristic.*

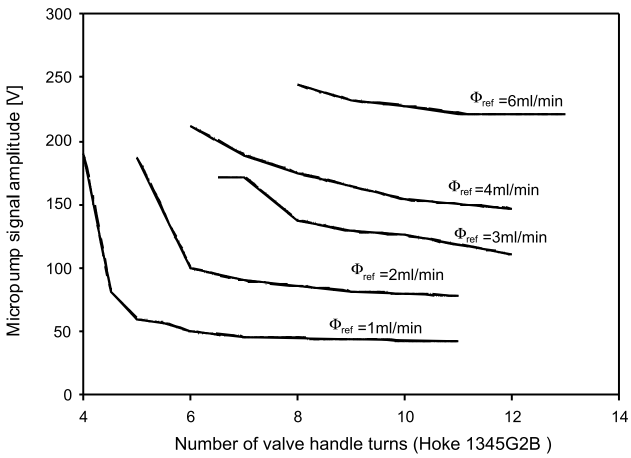


Fig. 21. MPC approach: Micropump signal amplitude response U *on load hydraulic resistance* R_{load}.

5.2. Method II – Electric current source analogy

The major drawback of the second method with electric current source analogy was estimated energetic insufficiency due to additional orifice in flow generator design (Fig. 5). To achieve proper operation, its hydraulic resistance $R_{orifice}$ must be few orders of magnitude greater then load hydraulic resistance R_{load}. But unfortunately, additional orifice causes major pressure drop which needs to be compensated by greater micropump effort. To achieve best system flow rate Φ vs. load hydraulic resistance R_{load} characteristic, orifice resistance $R_{orifice}$ is tuned according to load resistance R_{load}. Tuning is performed at desired system reference flow rate Φ at near maximal micropump supply voltage *U*. During operation, this results as fully driven micropump independently on system load hydraulic resistance R_{load}.

On the other hand, system operated fast, reliably and was practically insensitive of back pressure strokes from mi-

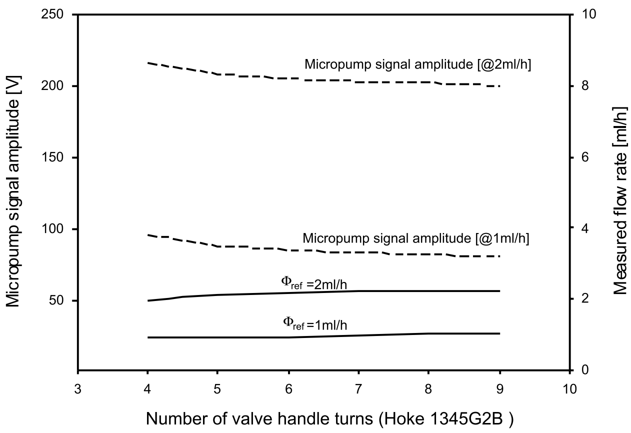


Fig. 22. Electric current source analogy approach: System flow rate Φ and micropump signal amplitude response U *vs. load hydraulic resistance* R_{load} *characteristic.*

croreactor. The lowest flow rates were achieved with this method. System flow rate Φ and micropump signal amplitude response U vs. load hydraulic resistance R_{load} characteristic is presented on Fig. 22.

5.3. Method III – Venturi meter / orifice plate

The third method was implemented with orifice plate. In flow rate measuring, orifice plate was found superior to Venturi pipe, due insufficient measured Venturi effect at extremely low flow rates. Its energy efficiency was estimated conditional on orifice plate hydraulic resistance. It was prone to microreactor dynamic back pressure strokes sensitivity which could be to a certain point corrected by software pressure sensor signal filtering. System flow rate Φ and micropump signal amplitude response U vs. load hydraulic resistance R_{load} characteristic is presented on Fig. 23.

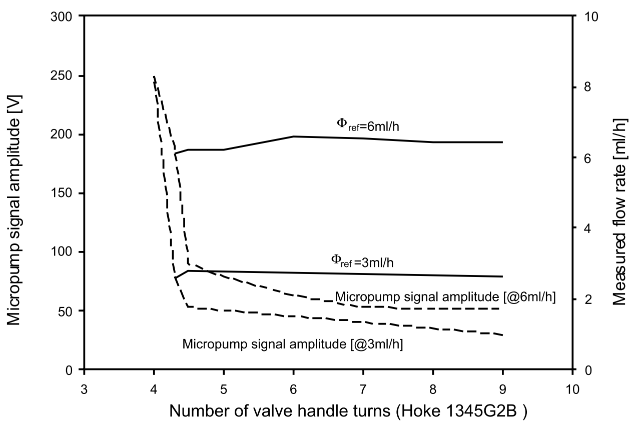


Fig. 23. Orifice plate approach: System flow rate Φ and micropump signal amplitude response U *vs. load hydraulic resistance* R_{load} *characteristic.*

6. Conclusions

In the presented paper, design guidelines of three approaches for micro-flow generator in hydrogen production microreactor were discussed in detail, followed by the description of experimental setup and characterization. To find the superior solution, basic properties of each method were estimated.

The main advantage of proposed MPC flow-generator method is its estimated energy efficiency. However, the main problem of this method lies in its dependency on long-term micopump stability. Based on our experimental work, used micropumps stability was found inadequate, therefore the whole system became unstable which on long term resulted in flow rate drift. Consequently, flow rate could even reach its zero or maximum value, and this cannot be predicted. To summarize, the assembled MPC system can for now only be efficient on shorter time periods, but as soon as it moves with time sufficiently from its starting stable point of operation, it can even collapse. Despite the fact that micropumps under test were performing rather unstable at long-term operation, the proposed MPC concept and realized flow-generator was successfully operated and tested, even for longer times. For very long operation times, a control flow calibration should be introduced. Due to the promising simplicity of this approach which employs no passive hydraulic elements and therefore estimated system energy efficiency, further investigations with better micropump actuators are going on.

Second, current source analogy approach, was found highly stable, fast and reliable, capable of generating extreme low level flow rates, but estimated as energy inefficient. Therefore, this approach should certainly be considered, when energy efficiency is not in question.

Finally, taking everything into account, differential pressure measuring on either side of orifice plate, supported with PID control regulation and micro pump actuator was found as the most appropriate, what was further confirmed also with successfully running microreactor applications.

Proposed flow-generator approaches are expected to enable a compact integrated hybrid implementation, comprised on a silicon substrate, anodic bonded with pyrex glass, including micropump, pressure sensor and microcontroller. Such integrated flow-generator is expected to be small enough for applications in advanced microreactors.

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