

---

Chair of Reaction Engineering  
Prof. Dr. David W. Agar

---

# **Optimisation and Implementation of a non-invasive measurement technique to monitor liquid-liquid slug flow in micro-channels**

---

## **Master Thesis**

*submitted in the fulfilment of requirements for the degree of  
Master of Science in Chemical Engineering*

### **Author:**

Shoneil Oswal

### **Supervising professors:**

Prof. Dr. David W. Agar

Prof. Dr.-Ing. Sebastian Engell

### **Supervisor:**

M. Sc. Linda Arsenjuk

Dortmund, July 21, 2015



# Acknowledgements

test



# Contents

<b>1</b>	<b>Introduction</b>	<b>1</b>
<b>2</b>	<b>Implementaton of online measurement</b>	<b>3</b>
2.1	Initial Scenario . . . . .	3
2.2	Switch to LabVIEW . . . . .	4
2.3	Signal filtering . . . . .	4
2.4	Reproducible electrode . . . . .	4
<b>3</b>	<b>External influences on signal</b>	<b>7</b>
3.1	Introduction from Sascha's Work . . . . .	7
3.2	Input length . . . . .	7
3.3	Brushing against polymer . . . . .	7
3.4	Heating of capillary . . . . .	8
<b>4</b>	<b>Parameter estimation</b>	<b>9</b>
4.1	Variables of interest . . . . .	9
4.2	Statistical data . . . . .	9
4.3	Scaling . . . . .	9
4.4	Variably peaked signal front . . . . .	10

4.5	Earthing using metal knob . . . . .	10
4.6	Shortening of input channel length . . . . .	10
4.7	Statistics of delpeak . . . . .	10
4.8	Curve fitting . . . . .	10
<b>5</b>	<b>Implementation of control</b>	<b>11</b>
5.1	Finite State Automata . . . . .	11
5.2	Characteristic line of valve . . . . .	11
5.3	Curve fitting for characteristic line . . . . .	11
5.4	Classical tuning method for fine control . . . . .	12
5.5	Online implementation in LV . . . . .	12
5.6	Performace of overall control loop . . . . .	12
<b>6</b>	<b>Conclusion and Outlook</b>	<b>13</b>

## List of Figures





## List of Tables



# 1 Introduction

Micro process engineering is a revolutionary technology of conducting chemical and physical processes in small volumina, typically in channels with an inner diameter in the sub-millimeter range. It offers high potential for significant improvement of process efficiency over conventional continuous flow systems. Due to small dimensions exceeding upto a maximum of 1 mm, this technology allows enhanced heat and mass transfer. For example, the diffusion length in micro-channels is comparable to its width, thus facilitating mixing of components in time of the order of micro-seconds. Similarly, excellent heat transfer capabilities allow conducting highly exothermic under isothermal conditions. Such characteristics can be well exploited for process intensification.

A key research focus in micro process engineering is the behaviour of two-phase flow in micro-channels. Biphasic liquid-liquid slug flows can be operated with high performances because of their inherently special hydrodynamics and therefore, form the subject of this study. The special properties of multiphase flow systems as given by are attributed to three key differences from conventional macro flow systems.

ref Jo-  
vanovic  
et al.

Formation of slugs of the order of channel width result in high specific surface areas which, inturn, leads to intensified mass transfer as mentioned above. reported a two order increase in magnitude of mass transfer compared to conventional methods. Internal circulation and eddys

ref  
Kashid  
et al.

inside the slugs additionally increase the interfacial mass transfer.

Secondly, the interfacial tension is not negligible compared to the viscous forces. Consequently, it is possible to create and regulate multiphase flows in the form of slugs under a given set of system conditions. Thus, a consistent flow regime can be established as previously discussed by several authors. This is called the slug flow regime, which forms a core part of the considerations of this work.

Finally, the developed flow is strongly influenced by the roughness and the wetting properties of the wall material. These influences can be exploited for guiding the flow. has shown that differences in the hydrophilicity of the wall material can be used for phase separation via a side channel.

ref  
4,7,8

ref  
Scheiff  
et al. 9

Add  
paper  
info  
sent  
by  
Prof.  
Agar

## **2 Implementation of online measurement**

In order to implement a control structure, the signals from the electrode need to be read and processed online. This chapter deals with the initial obstacles faced in doing so. Firstly, the software interface was changed mainly to facilitate online signal processing and subsequently, control. Secondly, the read signals are digitally processed to increase the Signal to Noise(SNR) ratio. This aids in reading and extracting the signal characteristics, such as amplitude, slope and period, better. Finally, a electrode of fixed dimensions and thus, reproducible in nature was identified.

### **2.1 Initial Scenario**

Discuss the initial state of the project. what were the ideas? What problems were there? The new ideas...

## 2.2 Switch to LabVIEW

Earlier, the voltage signals were read using a Digital Storage Oscilloscope(DSO) and its corresponding software program. The voltage data was recorded in MS-Excel. This data was then read into MATLAB and a spline regression was performed to extract the underlying signal. The major drawback of this process was that it is offline and thus, a continuous control on the liquid-liquid slug flow is not possible. Additionally, the software does not feature any means to digitally process or analyse the signal in any way. Conversely, a new software interface is required which can fulfill all the above mentioned limitations of the DSO and its allied software. LABVIEW facilitates not only reading the signals online, but also digitally process them, for example filtering, analyse and subsequently control desired variables. A NI-USB 6212 measuring card has been used as an interface between the plant and LABVIEW program. The voltage output from the amplifier is fed to the measuring card. This voltage is continuously read into LABVIEW using its in-built Data Acquisition System(DAQ) as a digital signal. The measuring card has a maximum sampling frequency of 400k Samples/s. is an example of a signal sampled at 1000 samples/s in LABVIEW.

## 2.3 Signal filtering

test

## 2.4 Reproducible electrode

Until now, the electrode being used was formed by dropping molten tin into a Polytetrafluoroethylene(PTFE) mould with the capillary in-situ. Molten tin engulfs the capillary in the form of a ring thus forming the electrode. Often, the molten tin solidified quickly before it could completely surround the capillary and form the ring. This method yields electrodes of slightly different dimensions each time. Thus, in general, to achieve higher reproducibility and eliminate any added influences, a search for an electrode with fixed dimensions was carried out.

Based on the phenomenon of triboelectroc effect and induced polarisation, the following characteristics of an ideal electrode are enumerated:

1. It is a very good electrical conductor and thus, can be polarised to a higher extent
2. It should wrapped around the capillary as closely as possible, again, to achieve higher polarisation

3. The lateral width of the electrode should be small enough such that voltage signals from consecutive slugs do not interfere each other

Washers, nuts or simply electrical wires such as that of copper are the possible alternatives that fit the above mentioned criteria. In earlier studies, use of a copper wire as an electrode was studied. . The wire was wound around the capillary as tight as possible and the voltage signals were recorded. Although, care had to be taken to avoid snapping the wire under the application of excess force. The results produced are shown in . The signal showed minor fluctuations which may be due to the fact that the copper wire cannot be wound perfectly tight.

Washers and nuts of different sizes were other alternatives of an electrode. Steel and brass washers as well as nuts of different sizes were tested as an electrode. The following figures show the signals corresponding to each of the electrode respectively.

It is evident from the above figures that the nuts produced better signals which can be digitally processed to extract its characteristics than do the washers. This is primarily because the washers do not fit around the capillary as do the nuts. Both, brass as well as steel nuts, produce voltage signals that can be used to extract signal characteristics. But, the brass nut is chosen owing to its higher electrical conductivity.

ref gat-  
bergadd  
figure  
from  
Gat-  
bergadd  
fig-  
ure of  
wash-  
ers  
and  
nuts





## **3 External influences on signal**

### **3.1 Introduction from Sascha's work**

Discuss the qualitative tests sascha did. Only the ones relevant to us... Say why length of ip channel was important.

### **3.2 Input length**

Repeat of experiment for verification. Experiment Details Findings, results and conclusion

### **3.3 Brushing against polymer**

Discuss this effect briefly. Conclusion can be that the slugs are not well charged; an understanding that can be exploited in future studies.

## 3.4 Heating of capillary

Discuss Experiment Exp details Findings and results (possibly Max's data here) and conclusion

## **4 Parameter estimation**

### **4.1 Variables of interest**

Discuss the possible variables that can be extracted. Which variables for which system parameter

### **4.2 Statistical data**

Present and discuss the statistical data of the a.m. parameters Explain the problem with amplitude and pulse duration

### **4.3 Scaling**

Why scaling is used and how was it implemented MAY help eliminate amplitude deviations

## **4.4 Variably peaked signal front**

Occurence much more often. Affecting slope calculations

## **4.5 Earthing using metal knob**

Amplitidue really low. Multiple peaks still there. No advantage. Determination of slope seems infeasible AS OF NOW. Estimate and control slug length.

## **4.6 Shortening of input channel length**

Too many unknown effects. Long length also not good for control. excessive dead time. Cut short length. Show statistics and reason for the choice made.

## **4.7 Statistics of delpeak**

Present model and validation data

## **4.8 Curve fitting**

Possibilities and alternative chosen. Why? Parameter Estimation complete. Show how you implemented it in LV.

## **5 Implementation of control**

General info Explain lack of plant model and why are we going to use FSA and then tuning rules.

### **5.1 Finite State Automata**

Figure and explain

### **5.2 Characteristic line of valve**

What is it? Advantages. Feed forward control. Dependency on vol flow rate. Combinatorial explosion. Solution: fixed flow rates Show results

### **5.3 Curve fitting for characteristic line**

Possibilities and choices made.

## **5.4 Classical tuning method for fine control**

## **5.5 Online implementation in LV**

## **5.6 Performance of overall control loop**

## **6 Conclusion and Outlook**

References

Appendix Calibration of pumps